Milling and Extrusion Characteristics of New Zealand Corn. Development of a Hardness Test and an On-Line Extruder Viscometer

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Abstract

Ready to Eat (RTE) snack foods are commonly manufactured using single and twin screw extruders and corn grits as raw materials. Variations in product quality caused by grits from different hybrids and grain hardness have not been investigated. Furthermore, the relationship between rheological properties of the extrudate melt and the operating conditions in the extruder is not fully understood. Appropriate methods to determine corn grain hardness to characterise corn hybrids and the on-line viscosity of the extrudate melts have not yet been developed. These methods could provide sound and appropriate techniques to investigate the areas of milling and extrusion of corn based food products.

In this study, milling characteristics of 38 corn hybrids from the 92-93 season and 12 corn hybrids from the 94-95 season produced in New Zealand were studied. A modified Stenvert Hardness Test (SHT) using new parameters including milling energy and milling resistance time was developed. It was found that the modified SHT was simple and easy to use with low variability. The SHT milling energy can be used as an effective hardness index. It increased with grain bulk density and the ratio of hard to soft endosperm. All measured properties were highly dependent on the moisture content. For the same hybrid, SHT hardness increased and the grain bulk density decreased when the moisture content of the corn grains increased.

A roller-milling test was also developed to study the dry milling characteristics of these corn hybrids. During milling, the breaking force measured in the roller-milling test increased with grain hardness.

Analysis of particle size distributions in the ground samples after the modified Stenvert Hardness tests and the roller-milling tests showed that grit recovery rate increased with the grain hardness.

Grits produced from hybrids harvested in the 92-93 and the 94-95 seasons, along with other grits and starches commercially manufactured in New Zealand, were used for the extrusion experiments.

A new Slit-Die-Viscometer (SDV) was developed to measure the viscosity of extrudate melts on-line. Unlike many other viscometers used on-line, the operation of the new SDV did not interfere with the operating conditions of the extruder.

The rheological properties and the degree of starch gelatinisation were affected by the operating conditions of the extruder and the characteristics of the raw materials:

It was found that the melt viscosity decreased as moisture content increased. The apparent viscosity had a maximum value at barrel temperature of about 130°C, changed very little when screw speed increased at constant feed, and decreased slightly when the feed increased at constant screw speed.

The grits were less gelatinised at high moisture content. The degree of starch gelatinisation increased slightly with screw speed and linearly with barrel temperature between 90°C and 130°C. At barrel temperatures higher than 130°C, the extrudate was almost fully gelatinised.

Melts produced with starch of high amylopectin content had an overall lower viscosity with less shear thinning and a higher degree of starch gelatinisation than that produced with starch of high amylose content.

Grit size affected the rheological properties and the degree of starch gelatinisation. Melts produced from medium and coarse grits had a lower viscosity and a lower degree of starch gelatinisation than that produced with fine grits.

The effect of different hybrids of the same season on the rheological properties of the melt was negligible. However, the rheological properties were affected by the methods used to produce the grits. Grits from degermed grains had less oil and produced melts with lower viscosity and less shear thinning than grits from whole grains (higher oil content).

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Nomenclature

Abbreviations:

BCFM	broken corn and foreign material
BD	Grain bulk density
CIMMYT	International Centre for Maize and Wheat Improvement
Com	commercial corn inbred
COV	coefficients of variation
DSC	Differential Scanning Calorimetry
ED	European yellow dent corn inbred
EF	European flint corn inbred
EI	a radial expansion index
H/S	a hard to soft endosperm ratio for corn kernel
HD	Hungarian yellow dent corn inbred
HT	highland tropical corn inbred
LDPE	low density polyethylene
LEI	Longitudinal Expansion Index measured in the axial direction
MEF	Milling Evaluation Factor in dry milling
MC	moisture content
NIR	Near-Infrared Reflectance
PCA	principal component analysis
RT	resistance time in the modified Stenvert Hardness Test
RTD	the Residence Time Distribution in extrusion
RTE	Ready-to-Eat snack
SBT	Stein Breakage Tester
SDV	Slit-Die-Viscometer
SEI	Sectional Expansion Index measured in the radial direction
SHT	Stenvert Hardness Test
SME	Specific Mechanical Energy in extrusion, $W \cdot h/kg$
STD	standard deviation
US	United States Corn Belt Dent inbred
WBT	Wisconsin Breakage Tester

dwb   dry weight basis

wwb   wet weight basis

Symbols:

$\dot{\gamma}$ shear rate, 1/s

$\dot{\gamma}_{app}$ apparent shear rate, 1/s

τ shear stress, Pa

ϕ electric power factor

$(\mu_a)_d$ apparent viscosity of the melt at the die, Pa·s

η_{app} the apparent viscosity, Pa·s

ρ_d the density of the extrudate inside the die, kg/m^3

ρ_e the density of the expanded extrudate, kg/m^3

ρ_g The density of the grit, kg/m^3

ΔP pressure difference, Pa

$\partial P/\partial Z$ the pressure gradient along flow direction on slit, Pa/m

τ_w the shear stress at the slit wall, Pa

+a*   the red colour co-ordinate

A_1 The absorbance at 600nm of the gelatinised sample in a spectrophotometer

A_2 The absorbance at 600nm of the total soluble starch sample in a spectrophotometer

C_{ID} a hybrid dependent coefficients, joules (J)

Dp   effective particle size (diameter) of corn grits, mm

d variability range

E The SHT milling energy, J

E_{ID} a hybrid dependent coefficients, J

F_{hybrid} a hybrid dependent shift factor, kg/hl

G weight, kg

Gr the grit recover rate ranging from 0.699 to 1.47mm

H the height of the slit of the SDV, m

I electric current, A

I_0	the initial current of the mill motor at empty load at the defined speed, A
K	the power law consistency index, $Pa \cdot s^n$
L	the length between two neighbouring pressure transducers of the SDV
$L*a*b*$	the absolute chromaticity colour space for a Minolta Chroma Meter CR-200)
N	screw speed, *rpm*
n	the power law index.
P	pressure, Pa
P	power, W
Q	the extruder throughput, kg/h
Q	the volume flow rate in SDV or capillary viscometer, m^3/s
R	the radius of the capillary, m
t_{end}	end time, s
Tq	screw torque, N·m
Tq_0	the screw torque at empty load, N·m
t_{start}	the starting time, s
u	the flow viscosity, Pa·s
V	the volume, m^3
V	voltage, V
W	the width of the slit of the SDV, m

subscripts:

d	conditions at the die
e	conditions of the extrudate
m	mixture
g	grits
w	water
max	the upper limit
min	the lower limit
0	initial values

Chapter 1

Introduction

The use of extruders in snack food manufacturing started in the 1930s when corn curls were first extruded (Moore, 1994). Today with the development of new technologies, snack food extrusion is becoming more advanced and many new Ready-to-Eat (RTE) snack products are manufactured using single and twin- screw extruders. Snack food extrusion is a continuous process where mechanical and thermal energy is rapidly introduced to transform the powdery ingredients into a fluid usually known as melt. Extrusion is also a continuous pumping and shaping process where materials are simultaneously transported, mixed, sheared, expanded and shaped through a die opening under elevated temperatures and pressures.

A large portion of RTE snacks are made using corn grits as the key raw material (Frame, 1994). The production of snacks usually involves two steps: In the first step, the grain is degermed and then milled to produce grits. In the second step, the grits are cooked and formed in the extruder to produce the desired product. Product quality depends on the properties of the raw material and the processing conditions in the extruder. Difficulties in maintaining product qualities have been experienced in the production of corn snacks (personal communication with Bluebird Foods Ltd. NZ). This is due to the variability of the properties of raw material and the lack of understanding of the interaction among operating conditions of the extruder, raw material properties and product qualities.

In New Zealand, the cool temperate climate causes large variations in the quality of corn grain. In the North Island of New Zealand, the eastern areas like Hawkes Bay and Gisborne are warmer and drier than the rest of the North Island. In these regions drought causes stress in the plant and results in maize grains of poor quality. In the south of the North Island near the Manawatu region, windy, wet and cold weather causes problems during harvesting as grains contain high moisture contents. Many overseas hybrids, not specifically bred for New Zealand, are being marketed and grown in these regions. Traditionally corn growers were paid on a per tonnage basis and they usually focused on

high yield rather than quality. All these factors contributed to the inconsistency of corn grit production in the past. The food industry was forced to adapt and utilise different raw materials depending on what was available at that time.

Recently, a consumer requirement for high quality products and a more competitive market have caused the food industry to demand raw materials with better defined specifications. Grits derived from dry milling must have defined moisture, oil and protein content, particle size distribution and mechanical strength. Hard grains produce a higher proportion of grits of large size and less flour. Grain hardness is a characteristic of each hybrid and affects the properties of the grits. Seasonal changes, genetic variation, agronomic practice, drying conditions and harvesting process can all affect grain hardness, the grain endosperm physico-chemical properties and therefore the quality of grits. Thus, grain hardness becomes an important quality index for grain grading before the milling process. The term grain hardness, however, is not clearly defined when applied to corn grains and there are no universally accepted test methods to measure this characteristic.

During the extrusion process, corn grits are fed into the extruder at a constant rate where they are heated by thermal conduction through the extruder barrel and internal friction. For some applications, the barrel temperature may be as high as 180°C. The actual temperature of the extruded material is difficult to measure and is commonly assumed to be close to that of the barrel. During the extrusion process corn grits are pushed from the feed zone toward a melting section and the extruder die by the rotation of one or two screws within the extruder barrel. The resistance of the die and the decreasing volume originated by a decrease in the screw pitch generates a very high pressure. Shear forces produced when the material flow through the extruder channels also play an important role in the plasticisation and chemical modification of the grits occurring during the process. The high temperature, pressure and shear existing inside the extruder convert the grits into a viscoelastic dough. The conversion from a powdery raw material to a viscoelastic mass is associated with modifications that include starch gelatinization and breakage, protein denaturation and the formation of amylose-lipid complexes. Under high pressure, the viscoelastic dough (also known as melt) flows through the die opening. As the melt exits the die, the sudden decrease in pressure results in a rapid flashing-off of part of the water contained in the melt as superheated steam. The

evaporation process and the subsequent cooling of the product create a porous structure. When the product cools the melt hardens and sets forming a light, crispy expanded product. Temperature plays a very important role in the expansion process. If the exit temperature is lower than 100°C, a dense and little expanded product is obtained. This dense product can be further expanded by frying to obtain the final product. These products are known as the third generation extruded snacks.

The quality of the final expanded product is closely related to the rheological properties of the melt. Melt viscosity is strongly affected by the properties of raw materials including grit size, moisture and oil content, starch type and the thermo-mechanical treatment history. At present variation in melt viscosity can not be detected until a large amount of product with poor quality is manufactured. At this point it is already too late to take any corrective action and avoid product wastage. It was hypothesised that if the melt viscosity, die pressure and temperature were closely monitored, and a relationship between product quality, melt viscosity and raw material characteristics were known, it would be possible to have a controlled operation and avoid wastage. Such knowledge is certain to be of value to the snack food industry. It is possible that some of this knowledge has been already developed in-house by large companies but it has not been published.

Extruder-fed slit die viscometers (SDV) have been extensively used in plastic extrusion to measure on line melt viscosity. The successful use of the on-line SDV in plastic extrusion encouraged researchers to apply this to the extrusion of food products. However, the extrusion of bio-materials is different from the extrusion of plastics because bio-materials are more prone to undergo physico-chemical changes during the extrusion process. Thus, the use of an on-line viscometer in extrusion of bio-materials becomes less straight forward as it produces interference between the rheological measurements and the extruder operation.

To significantly reduce product loss and to enhance the ability to produce high quality products we must understand the relationship between raw materials, operating conditions, melt viscosity and the properties of end products. Methods for determining key properties of corn grains in post-harvest handling and rheological properties of the melt in extrusion must be developed.

It is reasonable to expect that the quality of the final snack food product will improve if the entire process including crop selection and production, harvesting, milling, and extrusion is controlled and monitored with a view to producing a stable end product. Unfortunately knowledge concerning dry milling and extrusion of New Zealand corn varieties is not known and little information is available worldwide.

This project had two general objectives:
♦ to develop a hardness test to investigate the grain properties and milling characteristics of different New Zealand hybrids
♦ to develop an on-line viscometer to investigate the effects of raw materials and extruder operating conditions on melt viscosity during extrusion.

Specific objectives of this research project were:
1) to study the milling characteristics and grain quality of different New Zealand corn hybrids by:
   • developing methodologies to assess grain quality including grain hardness;
   • studying the dry-milling characteristics including grit recovery rate;
   • studying the relationship between grain hardness and grit production;
2) to develop a new on-line viscometer to obtain a proper estimation of the rheological properties of melts by:
   • designing a new on-line Slit-Die-Viscometer (SDV);
   • developing an appropriate data acquisition system including software for the new SDV and extruder;
   • evaluating the new SDV and assessing its application in extrusion of food products;
3) to study the effects of extruder operating parameters and grits derived from different corn hybrids on the rheological properties of the melt. With emphasis on:
   • investigating the effects of the extrusion operating parameters on the rheological properties of the melt;
   • investigating physico-chemical changes to the raw materials, notably the degree of starch and how it is affected by extrusion conditions;
   • studying the effects of corn grit size and hybrid on the melt viscosity;

- studying the effect of starch composition, using starches of different amylose/amylopectin ratio, on the viscosity of the melt and the degree of starch gelatinisation of the extruded product;

4) to investigate if the properties of the raw corn grain and the rheological properties of the extruded melt are correlated.

Chapter 2

Literature Review (Milling)

2.1 Introduction

Corn grit is the basic raw material for the production of many extruded snacks and breakfast cereals. It is produced by dry milling of grains from which the pericarp and germ have been removed previously in a degermer. The quality of the grit varies widely and depends on the hybrid grown, cultural and climatic conditions, harvesting techniques, drying and subsequent handling and storage (Watson, 1987a). In the dry-milling industry, hard grains increase grit recovery and soft grains produce a higher proportion of flour (Tran *et al.,* 1981).

Grain quality is a multi-factor property and its definition can vary for different end users. Quality can be measured by a variety of tests, from simple visual appearance to more complicated laboratory milling tests. Quality factors include bulk density, the amount of broken corn and foreign material (BCFM) and damaged kernels, moisture content, hardness, breakage susceptibility and disease level (Watson, 1987a).

In this chapter, the structure of the corn kernel, the function of moisture in grain quality, and the physical properties of the corn grain are reviewed. The methods for determination of gain properties are discussed. The dry milling performance of corn grain and the effect of hybrid on quality are also reviewed.

2.2 Structure of Corn Kernel.

Corn grains have a heterogeneous structure and in rheological terms can be classified as viscoelastic solids. Their mechanical and rheological properties change with time, temperature, moisture content, chemical composition and microstructure within the kernel.

A mature corn kernel consists of three major regions (Figure 2.1), the germ comprising the embryo and the scutellum, the endosperm, and a protective seed coat comprised of the aleurone layer and pericarp (Watson, 1987b). The seed coat and pericarp accounts for 5-6% of the kernel dry weight and are composed of dead cells that are cellulosic tubes. The germ makes up 10 to 12 % of the kernel dry weight and is high in fat (31 to 35% of its dry weight) and protein (17 to 19% of its dry weight). The germ contains about 80% of the total fat in the grain. The endosperm constitutes 80-85% of the kernel dry weight and its composition is 86-89% starch by weight. Depending on the structure and amount of protein matrix surrounding the starch granules, two types of endosperm are usually present in corn kernels, hard or horny and soft or floury. In the hard endosperm, a thick protein matrix remains intact on drying and binds the starch granules tightly together forming a strong structure with a translucent glassy appearance. The soft endosperm has a thinner protein matrix than the hard endosperm. During drying, the endosperm collapses, tearing the thin protein matrix, resulting in loosely bound starch granules (Watson, 1987b). The endosperm often contains voids and is structurally weak (Duvick, 1961). The outer region of the corn kernel tends to be comprised of hard endosperm while the inner region tends to be comprised of soft endosperm. In popcorns and flint type corns almost all the endosperm is of the hard type, while in the most floury varieties almost no hard endosperm is present. Varieties used by the food industry contain a mixture of both types. Generally a higher proportion of hard endosperm is preferred for milling as corn grit comprises a very large amount of hard endosperm fragments.
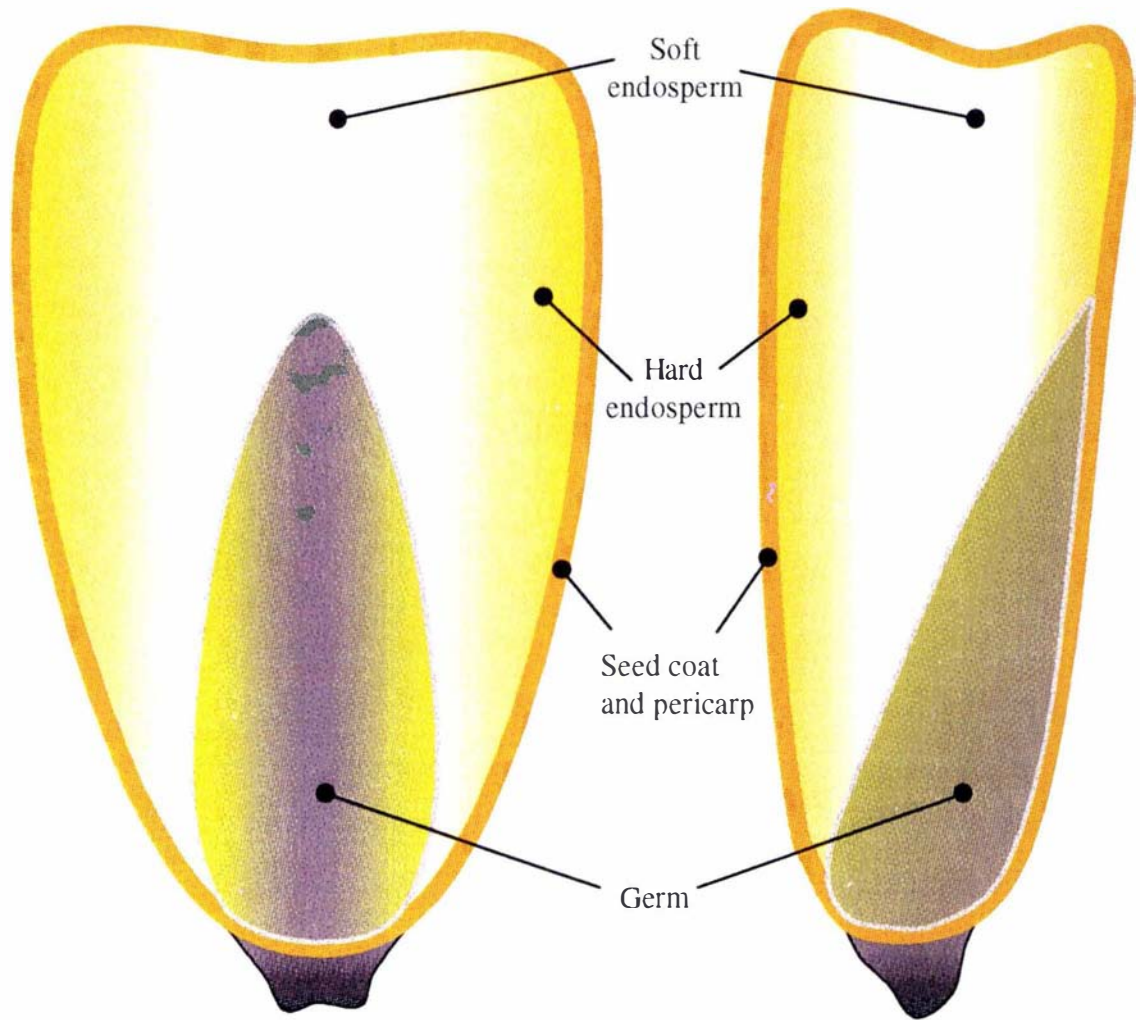
Figure 2.1 A schematic sectional plot of a yellow dent corn kernel.

2.3 Moisture Content

Water is a natural component of corn kernels and has a significant influence on corn grain quality, processing and storage. At high moisture contents (>24%), the kernels have a soft texture that is easily cut and punctured by harvesting and handling machinery. Before harvest, corncobs are naturally dried on the plant until the optimum moisture content of 23-24% for combine harvesting. However, this moisture content is not optimum for storage and the harvested corn must be further dried to prevent mould, bacterial, and sprout deterioration.

Corn quality changes with moisture content (Shelef and Mohsenin, 1969; Chung and Converse, 1971; Hall, 1972; Hall and Hill, 1974; Brusewitz, 1975; Nelson, 1980; Watson, 1987a; Dorsey-Redding et al., 1990). The rate at which moisture is removed from the kernel affects quality, for instance, stress crack formation is due to the rapid removal of water. Thus, drying temperature has a larger effect on breakage susceptibility than genotype selection (Paulsen et al., 1983; Moes and Vyn, 1988). The moisture content of corn also affects its bulk density value. Bulk density increases as moisture content decreases (Watson, 1987a). Using data from ten hybrids Dorsey-Redding et al. (1990) derived empirical moisture-correction equations for the Stenvert Hardness Test, Water Absorption Index, and kernel density. Kernel density decreased linearly as moisture content increased. The slope of the density/moisture relationship was the same for those ten hybrids (Dorsey-Redding et al., 1990). Shelef and Mohsenin (1969) used an Instron universal testing machine to apply uniaxial compression to individual kernels. It was found that the hard endosperm was the major contributor to the mechanical properties of shelled corn, which decreased with increase in moisture content. Brusewitz (1975) found that the bulk density of the rewetted corn grain decreased with increase of moisture content up to 30% (wwb). Nelson (1980) showed that bulk and kernel densities of corn are linearly and highly correlated in the range of 10 to 30 % moisture, both decreasing with increasing moisture content.

2.4 Physical Properties of Corn Grain

Watson (1987a) described corn grain hardness as an inherited characteristic that is modified by cultural conditions and post-harvest handling conditions. Hardness is

difficult to measure because of the complexities of kernel structure. It has been demonstrated that corn hardness is related to the ratio of hard to soft endosperm, pericarp thickness and cell structure (Watson, 1987a).

Despite the importance of corn hardness in industrial applications and the large number of studies that have been published on this subject, there is no generally accepted standard for evaluation of hardness and the milling characteristics of corn. Although no quality criterion is, as yet, universally recognised, classification of grain using properties such as milling characteristics and physico-chemical properties have been reported. The methods reported in the literature are summarised below and discussed later in this section:

1) Grain density or bulk density (Pomeranz *et al.*, 1984, 1985, 1986a, 1986b; Pomeranz and Czuchajowska, 1987; Kirleis and Stroshine, 1990; Mestres *et al.*, 1991; Pratt *et al.*, 1995);

2) Ratio of hard to soft endosperm (Kirleis and Stroshine, 1990; Mestres *et al.*, 1991);

3) Dynamic hardness of corn kernel measured by an impacting pendulum (Jindal and Mohsenin, 1978);

4) Resistance to compression and stiffness of individual corn grain in a compression test (Tran *et al.*, 1981);

5) Pearling test (Tran *et al.*, 1981);

6) Abrasive milling test (Lawton and Faubion, 1989);

7) Grinding test by using a disc grinding mill (Tran *et al.*, 1981);

8) Breakage susceptibility (Pomeranz *et al.*, 1986a; Watson and Herum, 1986; Kirleis and Stroshine, 1990);

9) Near-Infrared Reflectance (NIR) of grounded samples (Pomeranz *et al.*, 1984, 1985, 1986a, 1986b; Pomeranz and Czuchajowska, 1987) ;

10) Particle size analysis from grinding test (Pomeranz *et al.*, 1984, 1986b; Pomeranz and Czuchajowska, 1987; Mestres *et al.*, 1991);

11) Milling Evaluation Factor (MEF) (Kirleis and Stroshine, 1990);

12) Stenvert Hardness Test (Pomeranz *et al.*, 1985, 1986a, 1986b; Pomeranz and Czuchajowska, 1987; Kirleis and Stroshine, 1990).

A brief description of the more relevant tests is given below.

2.4.1 Bulk Density and True Density.

There are two definitions to describe corn grain density: true density and bulk density.

True density, also called kernel density, is usually measured by weighing a given volume of a liquid of known density such as water or ethanol displaced by a known weight of corn grains. True density can also be estimated by a floater test (Manoharkumar *et al.*, 1978; Paulsen *et al.*, 1983; Paulsen and Hill, 1985; Pratt *et al.*, 1995). A predetermined weight of randomly selected whole kernels is placed in a carbon tetrachloride-kerosene solution with a specific gravity of 1.275. The weight of kernels that float are measured, and kernel density (relative hardness) is read from a chart relating kernel moisture content to percent floaters.

Bulk density, also called test weight, is measured by weighing a known volume of the grain. Bulk density is the most widely utilised parameter to describe the density of corn.

The structure of the corn kernel plays an important role in its density. The density of a corn kernel is the contribution of the density of all its components such as starch, protein, oil, water and air filling internal voids. Differences in chemical composition only account for a small proportion of the variation in density. The amount of void space within kernels and the ratio of hard to soft endosperm are the major contribution to variation in grain density (Watson, 1987b). Thus, true density or bulk density is used as an indicator of the ratio of hard to soft endosperm and as a hardness index (Manoharkumar *et al.*, 1978; Paulsen *et al.*, 1983; Pomeranz *et al.*, 1984, 1985, 1986a; Pomeranz and Czuchajowska, 1987; Peplinski *et al.*, 1989; Dorsey-Redding *et al.*, 1990; Kirleis and Stroshine, 1990; Mestres *et al.*, 1991; Pratt *et al.*, 1995). From a practical point of view grain density measurements are relatively easy and accurate but they are not necessarily a good representation of the grain mechanical hardness and milling characteristics. Hardacre (1997) showed that grain hardness estimated from the Stenvert hardness can vary independently of bulk density.

2.4.2 Ratio of Hard to Soft Endosperm.

In dent corns, the soft endosperm is located in the centre of the kernel and is usually surrounded by the hard endosperm and partially by the germ. The hard endosperm has a

cylindrical shape and is rigid in structure. The ratio of hard to soft endosperm of the cross-section area (Hamilton, *et al.,* 1951; Kirleis *et al.,* 1984; Mestres *et al.,* 1991) and the longitudinal-section area of the kernel (Kirleis and Stroshine, 1990) indicates the proportions of hard and soft endosperm of the corn grain and is used as an index of grain hardness, although it does not necessarily measure endosperm hardness. This method, however, is very time consuming and subject to a large variability (Watson, 1987a). The ratio of hard to soft endosperm, also known as vitreousness, is correlated with true kernel density (Mestres *et al.,* 1991). Generally hard grains have a higher bulk density and a higher value of hard to soft endosperm ratio than soft grains.

2.4.3 Dynamic Impact Test.

Jindal and Mohsenin (1978) reported dynamic hardness of corn grain under dynamic conditions using a pendulum impact tester. The angular displacements of the pendulum arm before and after the impact were used to calculate the impact energy and rebound energy. The peak deceleration and the duration of impact were monitored using a storage oscilloscope. The kernel was lightly sanded and the germ side of the sanded grain was cemented to the machined surface of a heavy cast iron block. The impact test was made on the horny endosperm side of the corn kernel. Dynamic hardness was expressed as the ratio of the energy absorbed to the volume of indentation and was found to be a function of moisture content and decreased with increasing moisture content. However, variation among tests was high. A truly accurate measurement of the energy absorbed by the specimen was difficult to obtain (Jindal and Mohsenin, 1978). The technique was time consuming and impractical for a large number of samples as only one kernel could be tested at a time.

2.4.4 Compression Test.

An Instron Universal Testing Machine was used by Tran *et al.* (1981) to perform compression on corn grains from one yellow dent hybrid. The grains had an original moisture content of 14% before being re-wetted to 26%. Individual corn kernels were ground flat at the tip cap to give a compression height of 9mm. Compression force was applied along the longitudinal axis of the grain until failure occurred. The resistance of the grain to compression was expressed as the maximum force or fracture force. Grain

stiffness was calculated from the ratio between this force and the deformation. Results showed that both indices decreased as the moisture content increased.

2.4.5 Pearling Test, Grinding Test and Abrasive Milling Test.

A Strong-Scott barley pearler with a strain gauge attached was used by Tran *et al.* (1981) to determine corn grain resistance to abrasion. The sample used was a yellow dent and was re-wetted from 14% to 26%. The barley pearler was run at a speed of 1725rpm and the torque was recorded during pearling. Twenty-five grams of grain were placed in the machine with the grit stone running for 60s. Three parameters were used to define the grain hardness. The material ground from the surface of the grain, expressed as a percentage of the initial sample weight after 5s of pearling, was defined as the pearling index. The maximum torque resulting from the resistance of grain to pearling was defined as the pearling resistance. The pearling energy was calculated from the torque-time curve and the rotational speed for a 60s running period.

With increased moisture content, the pearling index decreased and the pearling resistance increased due to the presence of a tougher bran layer around the kernel. Pearling index and pearling resistance measured the toughness of the outside layers of the kernel. The pearling energy increased non-linearly in a moisture range of 14 to 18%. It reached a peak value at 18%, then decreased as the moisture content of the grain increased from 18 to 25%.

Tran *et al.* (1981) also used a disc grinding mill to determine gain hardness for the same sample discussed above. The disc grinding mill had two corrugated metal discs rotating at 36 rpm with a clearance of 3mm. In the milling test 100g of corn was milled. Similar to the pearling test, three variables including grinding resistance, grinding index and grinding energy were determined. The maximum torque was defined as the grinding resistance. The percentage of particles remaining on a 1.7mm aperture sieve after sifting was defined as the grinding index. Grinding energy was determined as previously discussed for the pearling test. Grinding energy and grinding resistance increased with increasing moisture content but grinding index decreased. The grinding index varied linearly and inversely with grinding energy.

Tran *et al.* (1981) also evaluated the differentiating ability of these hardness tests by comparing the ratio between the variability range (d) and the standard deviation (STD). The variability range (d) was defined by the range of measured parameters on the softest and the hardest grain. The greater the value of d, the wider is the difference between samples. With small values of the standard deviation a test is more sensitive and there is more confidence in the measurements. The d/STD ratios revealed that the compression test had the poorest ability to differentiate between samples as it had the lowest d/STD values. Grinding index, grinding energy, breakage index and pearling resistance all had good differentiating power. The quotient of grinding index and grinding energy had an even better differentiating ability than grinding index or grinding energy alone. This would be indicating that a multivariable analysis and combination of more than one variable could provide as a more useful hardness index. Thus, it suggests that techniques such as principal component analysis could be suitable to analyse the hardness of corn.

Lawton and Faubion (1989) used a tangential abrasive dehulling device to measure kernel hardness of sorghum, wheat and corn samples. It was found that the percentage of kernel weight loss during milling followed a first-order decay model with milling time and the rate constant from this model was defined as the kernel hardness. Moisture content had a large effect on grain hardness of all samples. Significant differences in kernel hardness were found between popcorn, dent corn and white corn.

All the abrasive tests, however, describe only surface properties of the grain. For a representative estimate of grain hardness, the hardness of the entire kernel should be measured.

2.4.6 Near-Infrared Reflectance (NIR).

NIR was used to determine corn grain hardness by Pomeranz *et al.* (1984; 1985; 1986a; 1986b) and Pomeranz and Czuchajowska (1987). Corn grain was milled and NIR absorption at 1680 nm was measured with a Technicon Infralyzer. It was observed that the particle size distribution of the ground sample varied with grain hardness and this was correlated with NIR absorption at 1680nm. Greater NIR absorption was found in samples from hard corn.

2.4.7 Stenvert Hardness Test

The Stenvert Hardness Test (SHT) was proposed as a useful approach for the measurement of kernel hardness (Pomeranz *et al.,* 1985, 1986a, 1986b; Pomeranz and Czuchajowska, 1987; Kirleis and Stroshine, 1990). In this test 20g of corn kernels of a given moisture content are milled in a micro hammer mill. Three parameters are chosen to define a hardness index: resistance time (the time required to obtain 17ml of meal), the height of the meal in the collection tube and the ratio of coarse to fine particles in the resulting meal. Pomeranz *et al.* (1985) found that for three pairs of isogenic maize lines (dent and flint) and three yellow dent hybrids at 12% moisture, resistance time was highly correlated with true kernel density and the proportion of coarse particles in the meal. Soft grains had smaller resistance times, higher column heights, and lower ratios of coarse to fine particles than hard grains. For these hybrids, the various SHT hardness parameters, true grain density and NIR absorption at 1680nm were highly correlated. These authors concluded that SHT and the measured parameters were useful for determining corn hardness.

2.4.8 Breakage Susceptibility.

Breakage susceptibility is defined as the potential for kernel fragmentation or breakage when subjected to impact forces during handling and transport (AACC, 1983). Breakage susceptibility is highly related to stress cracks of grain formed during drying at elevated temperature and can be measured by various impact devices (Watson and Herum, 1986). The commonly used methods are the Stein Breakage Tester (SBT) and the Wisconsin Breakage Tester (WBT).

The Stein Breakage Tester impacts the grain with a rapidly rotating impeller fitted in a cup. The Wisconsin Breakage Tester uses a unique impeller having four channels cut in a metal disk through which the kernels are accelerated toward the impact zone. Breakage index is defined as the ratio of unbroken grain after the test and the initial weight of grains.

Stress cracking is an internal type of damage (fissures) and is caused by stresses inside the kernel, resulting primarily from differential zones of moisture content created during drying. Temperature gradients also contribute to stress inside the kernel but this is of

less significance because temperature equilibrium is reached more quickly than moisture equilibrium (Watson and Herum, 1986). The presence of stress cracks can be visualised when kernels are examined through a source of light.

The degree of multiple stress cracking in corn kernels was found to be highly correlated with breakage susceptibility (Moes and Vyn, 1988). Kirleis and Stroshine (1990) found that stress cracking was directly related to hardness with the hardest hybrid showing the most severe cracking. At low moisture levels a high correlation between corn hardness and breakage susceptibility was found by Pomeranz *et al.* (1986a). Moes and Vyn (1988) also showed that breakage susceptibility is highly related to hardness and moisture content. Kirleis and Stroshine (1990) concluded that Stein breakage susceptibility was primarily influenced by hardness, whereas the Wisconsin breakage susceptibility was correlated with stress cracking.

Although bulk density is correlated with grain hardness, surprisingly it was found not related to breakage susceptibility (Moes and Vyn, 1988). Paulsen *et al.* (1983) confirmed that the bulk density varied significantly among hybrids, but it was not correlated with breakage susceptibility for corn dried at low temperature.

2.5 Dry-Milling Performance and Particle Size Distribution in Milled Corn.

The performance of the grain during dry milling is very important in the estimation of quality for many processes and has attracted the attentions of various researchers.

In particular, corn hardness has a large effect on milling performance. Dry milling quality was estimated from a Milling Evaluation Factor (MEF) by Kirleis and Stroshine (1990). It was defined as an index reflecting grit and total endosperm yields. A specified short-flow dry-milling procedure using a horizontal drum degermer was used to determine MEF. It was calculated as the sum of fractions of grits remaining on sieves of sizes of 3½, 5, and 7 *W* on a Smico laboratory test sifter. MEF varied significantly among three corn hybrids of varying hardness. The hard hybrid gave the highest MEF

value and the soft the lowest. Furthermore, MEF was highly correlated with bulk density, kernel density and Stenvert hardness measured as resistance time. The single factor that best predicted milling quality was kernel density and the two factors that best predicted milling quality were kernel and bulk density. Other factors, in order of importance, were Stenvert hardness, Stein breakage, and Wisconsin breakage.

Grain hardness was found to be correlated to dry milling performance parameters such as grit recovery (Manoharkumar *et al.*, 1978). Pomeranz *et al.* (1985 and 1986b) also reported that grain hardness is correlated with particle size distribution of the ground material and hard grains can give higher ratio of coarse to fine particles than soft grains. High grit yields were obtained from corn with high bulk density and kernel hardness in a commercial scale dry milling process (Pomeranz and Czuchajowska, 1987). Furthermore Gupta *et al.* (1989) found that the endosperm texture influenced the distribution of products during milling, as soft-endosperm corn yielded more flour whereas hard-endosperm corn produced more grits. These results were confirmed in a commercial full scale milling study carried out by Paulsen and Hill (1985) in where an increase in the yield of large flaking grits was obtained by selecting hybrids with low breakage susceptibility and high bulk density.

Higher drying temperature increases the percentage of stress-cracked kernels and breakage susceptibility resulting in poor milling quality. Yield of grits from dry milling decreased as drying-temperature increased from 25°C to 60°C (Peplinski *et al.*, 1989; Stroshine *et al.*, 1986).

Mestres *et al.* (1991) summarised the dry-milling properties of 18 corn landraces from Africa using a range of techniques and concluded that vitreousness, which is defined as the ratio of the cross-sectional area of hard to soft endosperm, did not correlate with dry milling properties including semolina recovery. Kernel protein content was not highly correlated with vitreousness. Corn that was high in protein and low in ash gave high semolina recovery.

2.6 Hybrids.

The genotype and crop management determine the properties of the endosperm. Stroshine *et al.* (1986) showed that grain quality in terms of bulk density, thousand-kernel-weight, breakage susceptibility, and dry-milling performance varied among hybrids. These properties also varied with growing season even for the same hybrid. The quality of the endosperm for specific uses can be improved through screening, selection and breeding.

Peplinski *et al.* (1989) studied physical, chemical and dry-milling characteristics of different corn hybrids. Chemical composition varied between hybrids but was essentially unaffected by the drying temperature. Physical properties, including bulk density, kernel weight, floater test or hardness, stress cracking and kernel size, also differed among hybrids. The yield of grits and flour components during roller milling varied greatly with hybrid type and drying air temperature. The greatest effect was due to the hybrid. It was concluded that genotype is very important in determining the properties of the grain and can be used by the dry-miller, feed producer, and other end users to purchase grain with the desired chemical and physical properties. However, the environment in which the crop is grown will modify the basic properties of the hybrid. For some hybrids the effect of environment is very large indeed and may result in grain of unacceptable quality.

2.7 Discussion and Summary

From this literature review it is clear that the term quality is not clearly defined when applied to corn grains. However, it can be regarded as a particular property or a group of properties which reflect the value of the grain to the end user. It is surprising that for some of the properties that dramatically affect corn processing such as grain hardness, breakage susceptibility, and milling performance, there is no an unique definition or universally accepted test method.

Tests on single kernels are time consuming, tedious and have large statistical variation in their results. Although they can reveal the mechanical strength of individual corn

grains, they should not be classed as milling tests and have low value in determining the milling quality of the grain.

Bulk density can be easily measured and is commonly used as a quality index. Equipment for measuring bulk density is present in many companies and is much cheaper and easier to construct than the SHT. It is therefore important in determining grain quality in industrial practice. It is related with the structural characteristics of corn grains, although the ratio of hard to soft ratio endosperm gives more details in kernel structure. Unfortunately both bulk density and ratio of hard to soft endosperm do not always represent grain hardness and milling characteristics. Furthermore, bulk density has limitations in comparing corn hybrids (Hardacre, 1997).

Among the tests discussed above, the pearling and abrasive tests measure only surface properties of the kernel. The Stenvert Hardness Test seems to be the best method to determine corn grain hardness. It is rapid, simple, and reliable. It uses replicated 20g samples, in which many kernels are evaluated at a time. It is a milling process and its results are related to the dry milling quality. Tran *et al.* (1981) suggested that the grinding energy could be a useful hardness index, therefore, it would be logical to add this parameter to the measured SHT variables to further enhance the ability of this method to differentiate among grain types. It is necessary to evaluate the parameters obtained from the SHT systematically to estimate grain hardness rapidly and reliably. It is also important to determine if the SHT is correlated with the ratio of hard to soft endosperm and bulk density for a wide range of hybrid varieties, and where possible, performance during processing.

This literature review shows that corn quality varies significantly among genotypes. However, there is little information on the use of micro hammer mills, roller mills, or any other techniques for a systematic study of the endosperm properties and milling characteristics of a wide range of corn hybrids.

Chapter 3

Milling Experiments: Materials and Methods

In this chapter, materials used and methods of determining physical properties, milling characteristics and quality of corn grain are described along with the analytical techniques used.

3.1 Materials

3.1.1 Hybrids of the 92-93 Season

Thirty-five corn hybrids were machine harvested from a research farm in the Manawatu region (New Zealand) in June 1993. To this set, samples of DeKalb brand PX74 and Pioneer brand P3162 produced in the Gisborne region (New Zealand) were added along with a sample of Pioneer brand Dea produced in a southern New Zealand site. Immediately following harvest, the grain was slowly dried to about 14% moisture and stored in a cool room at 7°C and 30%RH until required. During storage, the grain equilibrated with the air in the cool room resulting in a mean moisture content (MC) of 10.5% (range 9.6 to 12.4%) when tested. Before the testing, all samples were placed in heavy paper bags and equilibrated to room temperature (25°C).

The corn hybrids used are listed in Table 3.1. They represented germplasm from diverse sources. The highland tropical (HT) source originates from the CIMMYT (International Centre for Maize and Wheat Improvement) maize breeding program and in this study refers to lines containing both highland tropical and corn belt dent germplasm (Eagles and Hardacre 1985). This material generally confers medium hardness properties to the endosperm. The lines of US origins used can be of soft or medium hard endosperm, while the European dents (ED) and Hungarian dents (HD) are of medium hardness. The European flints (EF) have very hard endosperm. The commercial hybrids are of unknown origin although it is suspected that those labelled soft share similar parentage.

3.1.2 Hybrids of the 94-95 Season

Twelve hybrids were machine harvested from the same research farm in the Manawatu region, Table 3.2. The moisture content of the grains after harvest was 20 to 25% on a wet weight basis (wwb). Grains were air dried to 19% moisture at 45°C and then sealed in plastic bags in a cool room at 7°C for a week to equilibrate to about 19% moisture content. Moisture content and bulk density of each sample were measured and SHT tests were conducted.

In order to investigate the effect of moisture content on grain hardness and test weight, grain samples of each hybrid were equilibrated to moisture contents ranging from 10.5 to 19.4%. Before the SHT test, all samples were equilibrated to room temperature (25°C) in small plastic bags for four to six hours. Ground meal from each SHT test was collected and dried in the cool room at 7°C and 30%RH for one month. Samples were then sieved to determine particle size distribution. After measuring moisture content, bulk density, and SHT parameters, samples of all hybrids were re-packed in heavy paper bags and retained at room temperature (25°C) to dry to the next desired moisture content.

Roller milling tests were conducted for the twelve samples at a grain moisture content of about 12%. Grits obtained from the roller milling tests were used for later extrusion experiments.

Table 3.1 Hybrids names, moisture contents, and bulk densities for samples from the 92-93 season.

#	Entry names	Origin	Moisture content(MC) (%)	Bulk Density (kg/hl)
1	PF2×NZ56	EF×HT	10.25	76.7
2	A676×NZ56	US×HT	10.20	75.7
3	PF2×NZ56	EF×HT	10.65	76.7
4	E1873×NZ3	EF×HT	10.30	75.2
5	PF1×AS3-94	EF×US	10.55	79.7
6	PF1×NZS1-141	EF×HT	10.70	77.9
7	MBS847×NZS3-59	US×HT	10.15	70.2
8	P3787	US Com Medium	10.90	73.5
9	PF1×BS22-39a	EF×HT	10.10	79.2
10	WGI207×BS22-22	US×US(NZ)	11.18	75.3
11	WGI207×M378-80	US×HT	10.50	73.4
12	WGI207×M396-14	US×HT	10.85	74.7
13	WGI29×PF1	US×EF	9.65	74.2
14	A665×M396-14	US×HT	11.15	74.4
15	P3394	US Com Medium	10.35	69.1
16	P3514	US Com Hard	10.55	70.8
17	P3515	US Com Hard	10.15	69.6
18	P3751a	US Com Medium	10.40	69.2
19	A82-8×NZ84	US×HT	10.10	73.1
20	PF1×BS22-78	EF×HT	10.95	80.0
21	PF2×NZ40	EF×HT	10.55	77.5
22	D1275×NZ84	ED×HT	10.90	75.3
23	PF1×NZS3-32	EF×HT	10.50	76.5
24	A82-8×MBS847	US×US	10.40	70.4
25	Furio	European Com Dent	10.25	72.2
26	D1260×BS22-22	ED×HT	10.40	72.4
27	PF1×BS22-39b	EF×HT	10.38	78.5
28	Hmv5313×PF1	HD×EF	9.85	77.9
29	N190×NZ2	US×HT	10.90	75.6
30	D1275×M378-80	ED×HT	11.25	77.3
31	P3162	US Com Hard	11.20	78.2
32	P3585	US Com Medium	9.60	69.0
33	P3751b	US Com Medium	10.45	71.2
34	P3901	US Com Medium	10.00	70.2
35	P3902	US Com Med-Hard	11.05	74.5
36	PAC42	US Com Medium	10.35	70.9
37	PX74	US Com Soft	10.60	68.6
38	Dea	European Com Flint	12.40	76.2

Note: 1) Samples No. 31 and 37 were produced in Gisborne region of New Zealand, sample No. 38 was produced in a southern New Zealand site, all other samples were produced in Manawatu region of New Zealand. 2) In the column of Origin, **EF** stands for European flints, **HD** for Hungarian dents, **ED** for European dents, **HT** for highland tropical sources , **US** for United States Corn Belt Dent, **Com** for Commercial. All the US commercial hybrids are dent types while the European Commercial hybrids are either flint or dent types.

Table 3.2 Hybrids names for samples harvested in the 94-95 season.

Sample #	Hybrid names
940901	P3751
940902	FURIO
940903	NZS3-523-1-1-1 × T1
940904	NZ84 × Mo17Ht
940905	Hmv-124-2 × T1
940906	A665 × H99
940907	NZ45 × T1
940908	M396-14-1-1-1-1-1 × A665
940909	157-R-3-9-1 ×A82-8
940910	N192 × Mo17Ht
940911	NZ84 × A82-8
940912	NZ84 × E1386

3.2 Analytical and Test Methods

3.2.1 Physical Properties of Grain

Oil Content

Oil content of corn grains and grits was measured by a modified extraction method using petroleum ether. Six grams of whole grain were ground into flour using a Cyclotech 1092 Sample Mill fitted with a 0.25mm screen. Four grams of flour were mixed with 20ml of petroleum ether in a test tube. The mixture was held at room temperature for one hour and agitated at 5-minute intervals. The oil in the flour is soluble in petroleum ether and therefore was extracted. The oil-petroleum ether solution filtered from the mixture using filter paper was placed into a water bath at 58°C where

the petroleum ether was evaporated. The remaining corn oil was weighed and the oil content calculated as percentage of the sample weight.

Protein Content

Protein content of grain or grits was measured by a LECO analyser (Leco CNS2000) which determined the proportion of nitrogen in the sample. This test was conducted at the Grain Foods Research Unit, the Institute for Crop & Food Research, Lincoln, Christchurch, New Zealand.

The sample being measured was burnt in a furnace at high temperature in pure (99.999%) oxygen. Any elemental carbon, sulphur and nitrogen were converted to their oxides. The gases obtained from combustion were dehumidified by passing them through a water absorbent Anhydrone and then passed through infrared cells to measure the contents of carbon and sulphur. Thereafter, the NO_X in the sample gas were reduced to N_2 by passing over a catalyst heater. The converted gases passed through an absorbent Lecosorb to remove CO_2 and Anhydrone to remove water. The resulting dry N_2 was passed through one side of a thermal conductivity cell of the LECO analyser where it was compared to filtered, pure Helium. The resulting voltage output was fed to the computer where it was processed, displayed and stored as the nitrogen content of the sample.

Moisture Content

Moisture content and bulk density (test weight) of stored and dried corn samples were determined using a grain analysis meter (Dickey-John GAC2000, Dickey-John Corporation, P.O. Box 10, Auburn, Illinois 62615, USA). The GAC2000 estimates grain moisture content using measurements of dielectric properties of grain. The bulk density was estimated from the weight of the sample in a pre-specified volume. Bulk density was recorded as kg/hectolitre and moisture content as weight percentage in a wet weight basis. The moisture content measured with the GAC2000 was calibrated against the standard oven drying technique and was found that the maximum difference was 0.5%.

Hard to Soft Endosperm Ratio

The hard to soft endosperm ratio of corn kernels was estimated by sectioning the kernels and measuring the areas of hard and soft endosperm presented at the cut surface. Dried kernels were sectioned just above the top of the embryo region using a pair of secateurs. The hard to soft endosperm ratio was calculated by measuring the average width and depth of the cut surface and the soft endosperm region using a pair of vernier calipers (Digimatic Caliper CD-8", Mitutoy Corporation, Japan). From the approximate areas measured, the ratio of hard to soft endosperm was calculated. Ten kernels for each sample were used to obtain the measurements.

3.2.2 Milling Tests

The Modified Stenvert Hardness Test

The Stenvert Hardness Test described in Chapter 2 was modified to have a better ability to measure milling characteristics of grains.

A 20g sample of grain of known moisture content was ground using a hammer mill fitted with a 2mm aperture particle screen. The mill was equipped with a computerised data acquisition system to log the instantaneous electric power consumption during the milling test (Figure 3.1). From the data the transient peak energy, total milling time and milling energy were determined. In addition, resistance time, the time taken to mill 17ml of meal and the meal height in the collection tube at the completion of milling the 20g grain sample was recorded. Details of the test are described below.
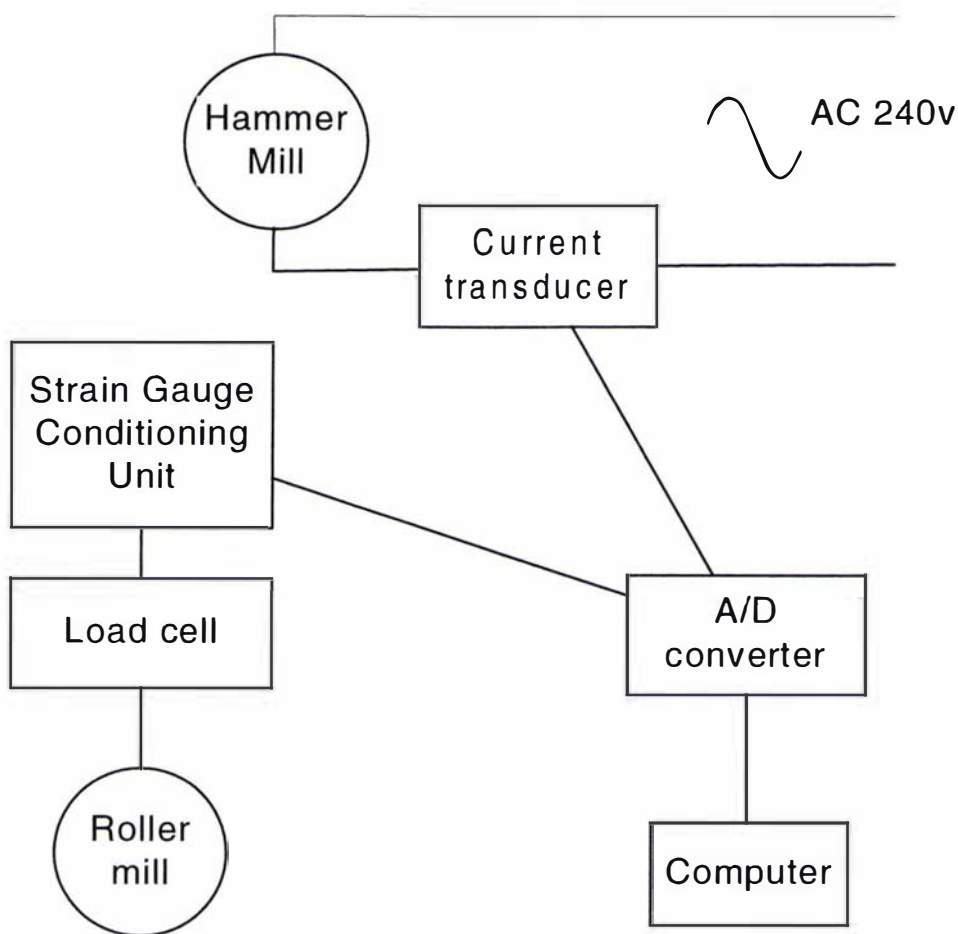
Figure 3.1 Schematic of the SHT and roller milling data acquisition system

A current transducer was connected to the mill AC motor and the motor current was monitored and measured. It was assumed that the power factor (*cosφ*) of the motor was close to 1 and remained constant under various load. The power drawn by the mill was determined as $P = V \times I$, where P is power (W), V is voltage (V), I is current (A). The fluctuation of electricity in New Zealand is very low thus the voltage was assumed constant (240V). The current transducer measured the current drawn by the motor and output a DC signal to one channel of the A/D converter (Remote Measurement System ADC-1, 2633 Eastlake Ave., Seattle, Washington). This signal was closely monitored by the program.

During the test the sample was dropped into the grinding chamber, resulting in an increase of both the torque applied to the motor and the current. This change in current triggered the data logging procedure, activated the on-line data processor and a timer for measuring resistance time. The power consumption of the mill climbed rapidly to a peak

value as a result of the sudden load. As the milling process progressed, more grits and flour were swept out through the particle screen and the power consumption decreased. After 20-40 seconds of milling, the grinding chamber was empty and the motor current decreased to its unloaded level.

Typical plots of power consumption during the milling of hard and soft corn samples are shown in Figure 3.2. Differences in power consumption during milling (area under the plots) are evident. There is no difference in the peak power consumption. The duration of the test was dependent on the time required to eliminate the grains from the milling chamber. It is evident from Figure 3.2 that the softer kernels of the hybrid PX74 were more rapidly and easily reduced to a size that could pass through the mill screen when compared with harder kernels such as those of the hybrid P3162.
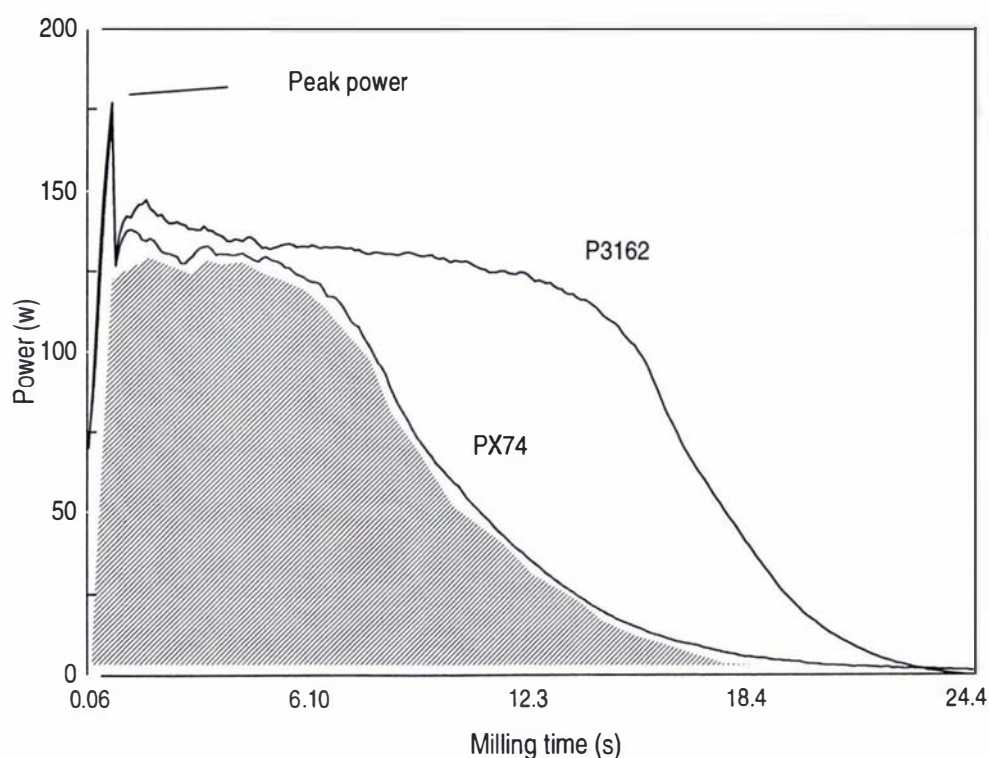
Figure 3.2 True transient power changes of SHT milling of two typical corn hybrids.

The current of the motor was then automatically logged onto a data file together with the calculated motor power. Data acquisition continued until power consumption

decreased to within 0.3 watts of the initial unloaded condition. The milling energy (E) was calculated by integration of the power between the starting (t_{start}) and end time (t_{end}):

$$E = \int_{t_{start}}^{t_{end}} V \cdot [I(t) - I_0] \cdot dt \qquad (3.1)$$

I_0 is the initial current of the mill motor at empty load before the test at the defined speed. The peak power was defined as the maximum value of $V \cdot [I(t)-I_0]$ in the measurement interval. Milling duration of the test was determined by the difference $t_{end}-t_{start}$. Resistance time was recorded as the time taken to mill 17ml of meal. A plastic test tube with a calibrated 17ml mark was fitted to the mill exit. Once the 17ml mark on the tube was reached, any key of the computer keyboard was pressed and the timer was stopped. The time taken was recorded as resistance time. The meal height in the collection tube at the completion of milling was also recorded manually. After the data were logged and processed, plots of the current test and three previous tests were plotted on the computer screen. Milling energy and resistance time were also displayed. If variation occurred within the duplicates of the same test, a warning was displayed on screen to remind that more tests on this particular sample were needed. All data except the transient power curves were analysed statistically using three to five replicates depending on the variable measured.

Two hammer mills with different speed settings were used in this study. A Glen Creston micro hammer mill N°5 without a tachometer (mill A) was used in an early stage of this project before a new purpose-built mill was available. The mill speed was set to 7,500 rpm at empty but the speed slowed substantially under load. This speed was considerably higher than recommended, but as the unit used was not fitted with a tachometer and as none was available at the time, a full speed setting was preferred. The mill was employed to test corn grain hardness of 38 hybrids from the 92-93 season. All later tests were conducted using a new Glen Creston micro hammer mill N°5 (mill B) equipped with a tachometer (Type 14-690, Glen Creston Ltd, Stanmore, England). The speed setting for the new mill was set to 3600 rpm unloaded.

The tachometer of mill B had an output of 0-5V DC signal which was connected to the second channel of the A/D converter. The speed of the mill was also closely monitored.

Only when the desired speed of 3600 rpm ±0.5% was reached and the speed was stable, did the program measure the initial motor power at empty load, then issue "ready" signals which allowed the test to begin. The behaviour of the speed was also plotted on the computer screen once data were processed.

Prior to collecting data, the mill was switched on and allowed to warm up by running without load for approximately 45 minutes. A set of 5 dummy grain samples was milled, followed by the set of test samples for which transient power of the mill was logged.

The SHT measures differences in the sample resistance to milling which could be associated with parameters such as the proportion of soft and hard endosperm and the bulk density. As the milling produces grits and flours, in theory, this method could result in a useful estimate of the yield of grits produced during milling.

Roller Milling

An existing roller mill was modified to investigate the properties of corn grain from different hybrids during dry-milling. The mill has an adjustable feeder and two fluted rollers running at different speeds thus grains can be crushed and torn into pieces (Figure 3.3). The gap between the rollers could be adjusted to produce grits of different sizes.

There are two pre-load springs at each side of one roller, which can be used to adjust the pre-load force. Between these two springs and the roller base, a load cell was fitted to measure the force applied to crush the grains (Figure 3.3). The load cell signal was regulated by a strain gauge conditioning unit whose output was connected into the third channel of the A/D converter as described in the previous section (Figure 3.1). A data acquisition program was employed to collect the milling force data. During a run, the data logging was turn on and off manually by pressing a key on the computer keyboard. The forces applied to the load cell were recorded onto data files. A simple statistics routine was incorporated to determine an average force for each experiment.

The roller mill was originally designed for handling barley. The flutes on the roller were small and suitable for smaller grains but too smooth for large grains like corn. The small flutes could not catch corn grains and the grains slid on the surface. Therefore, the grains remained in the mill and eventually a blockage occurred. A two-stage milling procedure was developed to prevent the corn grain from choking up the mill during operation.

In the first milling stage, the grains were milled using the roller mill with a 'soft' set up. The gap between the two rollers was set to 3mm and the pre-load force was adjusted to 22kgf. As shown in Figure 3.3, the load cell was used to measure the pre-load force during adjustment. In this set-up, the rollers could easily be pushed apart during milling. Therefore, this resulted in a large amount of unbroken grains and large grits. The unbroken grains were separated by sieving and re-milled. This sieving and re-milling procedure was repeated until almost all grains had been milled.

In the second milling stage, the ground material produced in the first milling stage was fed into the roller mill using a more rigid set-up. The gap between the two rollers was set to 1.4mm and the pre-load force was adjusted to 42kgf. The increase in pre-load force made the rollers difficult to push apart, therefore, smaller size grits were produced.

After the pre-load force and milling gap were adjusted to a suitable level, the roller mill was switched on and allowed to warm up by running for 30 minutes, then 5kg of dummy grains were milled. The samples were put through the roller mill, and the forces applied by the rollers were recorded by the computer.

Details of the computerised data acquisition system developed for this study, which was used for both the modified SHT milling test and roller milling test, are given in **Appendix A**.
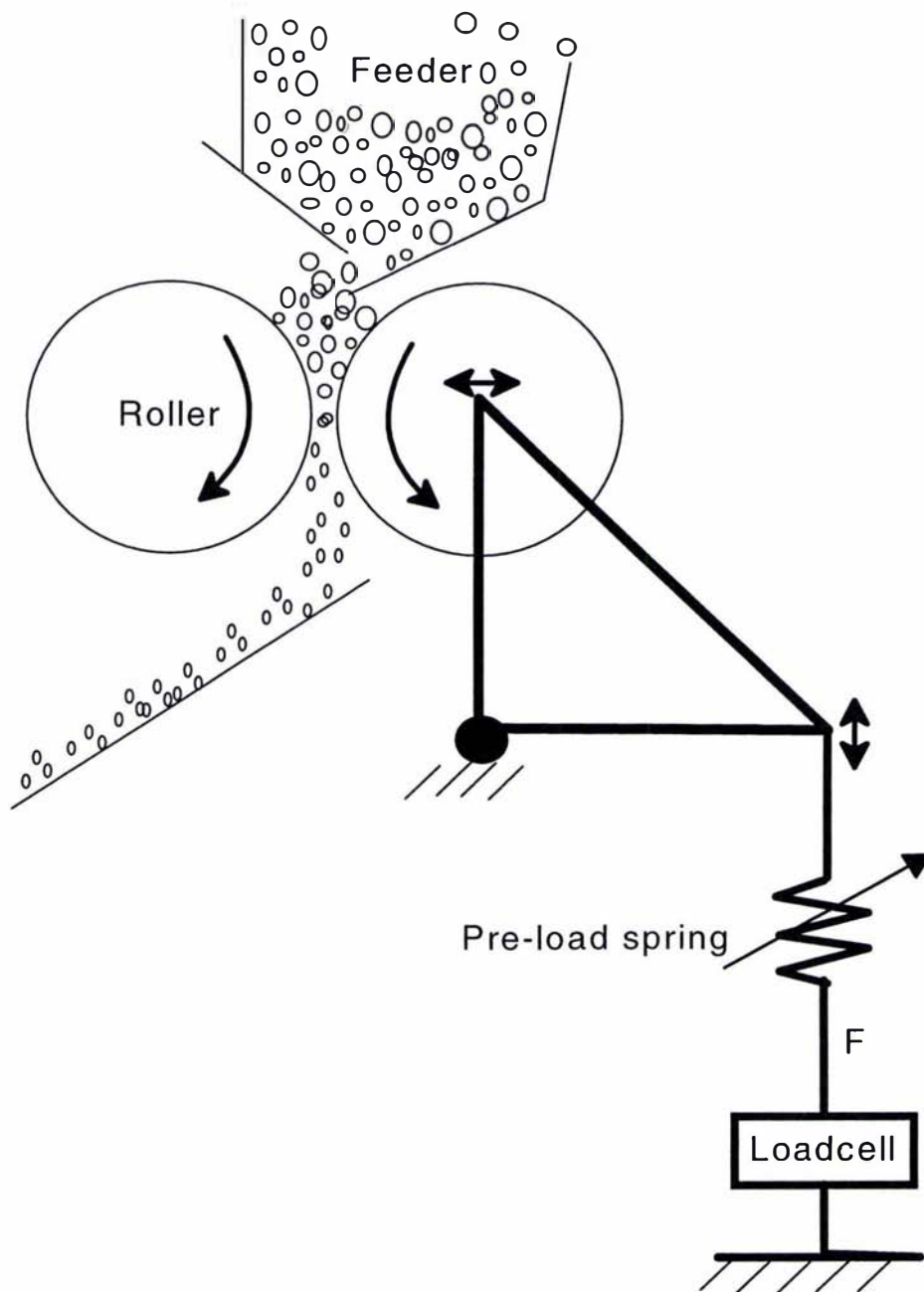
Figure 3.3 Schematic diagram of the roller mill and its load cell. Only one pre-load spring is shown.

3.2.3 Sieving Tests

Samples collected from the SHT test were sieved by using a set of standard sieves of sizes: 1.700mm, 1.400mm, 1.180mm, 1.000mm, 0.850mm, 0.710mm, 0.600mm, 0.500mm, 0.425mm and 0.355mm. Samples were shaken in the sieves for 5 minutes using a shaker (Model RX-86-1, W.S. Tyler, 8570 Tyler Blvd., Mentor, OH 44060, USA). Fractions remaining on each of the sieves were weighed and expressed as percentage of the original sample weight.

The procedure for analysing particle size distribution of samples from roller milling was similar to that described for the SHT samples. However, the grits were larger than those obtained from the SHT test, thus, a set of sieves of large sizes were used. The sieve sizes were 3.350mm, 2.400mm, 2.000mm, 1.700mm, 1.400mm, 1.180mm, 1.000mm, 0.850mm, 0.710mm, 0.600mm, 0.500mm, 0.425mm and 0.355mm.

3.2.4 ANOVA and Multivariate Analysis

Analysis of variance using SAS (SAS 1988) was applied to the data to generate F values and coefficients of variation (CV's) that enable the determination of differences among the hybrids.

Multivariate analyses of variance using principal component analysis (PCA) (SAS 1988) and cluster analysis (SAS 1988) were also applied to these data.

PCA transforms the SHT data and the physical properties, which are correlated, into a new set of uncorrelated variables called principal components. These new variables are linear combinations of the original variables and are derived in decreasing importance so that the first principal component accounts for as much as possible of the variation in the original data. If the first few components account for most of the variation in the original data, it is assumed that the effective dimensionality of the problem is less than the number of the original correlated variables. The original correlated variables represent effectively the same properties of the grain and, therefore, the PCA can help to understand the data better and to help to reduce the dimensionality of the problem.

Thus, for future analysis of the SHT data, one to two variables can be chosen as the key variables to determine the grain hardness.

The objective of the cluster analysis, using SHT data and grain properties, was to find out if the hybrids used in this research could be separated into groups. It is generally assumed that the properties of hybrids are related to their parents or even the remote origin of the inbreds. The groups generated from the cluster analysis could reveal if this assumption is true.

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Chapter 4

Results and Discussion: Hardness Measurements and Milling Characteristics of Corn Grain

</div>

4.1 Introduction

The modified Stenvert Hardness Test (SHT) was developed as a rapid, simple, and reliable test to determine corn grain hardness. This chapter presents results of measurements using this new developed technique to determine the hardness of different corn hybrids from the 92-93 and the 94-95 seasons. The effect of moisture content on bulk density and SHT hardness is determined and discussed. The milling characteristics of these corn hybrids using a roller mill test are also presented.

4.2 Results and Discussion

4.2.1 Hardness Tests for Corn Samples Harvested in the 92-93 Season

4.2.1.1 SHT and Properties of Different Hybrids

Grain bulk density (BD), hard to soft ratio (H/S) and five Modified Stenvert Hardness Test (SHT) parameters were measured for 38 hybrids from the 92-93 season (Table 4.1). Average coefficients of variation (CV's) within hybrids were less than 8.4% for most variables. However, the CV for the H/S ratio was 18.7% suggesting that for this variable, the differences among hybrids and the variation in measurements within each sample are hard to resolve.

From the F values presented in Table 4.1 and their probabilities at the 5% level it is evident that significant differences existed among the hybrids. F values were lower for sample height, peak power and milling time (MT) as were CV's, suggesting that the overall variability for these variables was low and that they may be less useful in separating the hybrids for milling characteristics. As moisture content and bulk density

measurements were un-replicated due to the small sample sizes available, statistical tests on these variables could not be conducted. However, past experience with these measurements has proven that errors are inherently very low (CV <4%) and therefore differences in bulk density greater than about 3kg/hl are probably significant. The reason for differences in moisture content is unknown as all samples were equilibrated under the same conditions. However, it is thought that differences in the chemical structure of the endosperm may be involved. Although moisture content has a significant effect on hardness measurements (Tran *et al* 1981) the differences found (Table 4.1) are probably too small to have a measurable effect.

Table 4.1 Experimental data for hybrids of the 92-93 season, ranked by bulk density.

Entry names	Moisture Content, (% wwb)	Bulk Density (kg/hl)	Peak Power (PeakP) (w)	Milling Time (s)	Total Energy (J)	Sample Height(H) (mm)	Resistance Time(RT) (s)	Hard/Soft Endosperm Ratio (H/S)
PF1×BS22-78	11.0	80.0	169	28.9	2391	98.0	8.9	5.0
PF1×AS3-94	10.6	79.7	178	27.0	2408	96.7	10.6	3.6
PF1×BS22-39a	10.1	79.2	180	26.4	2223	94.7	7.9	4.5
PF1×BS22-39b	10.4	78.5	175	24.3	2029	97.3	8.3	5.0
P3162	11.2	78.2	172	26.4	2373	100.0	8.5	7.4
PF1×NZS1-141	10.7	77.9	171	28.1	2253	100.0	9.3	4.1
Hmv5313×PF1	9.9	77.9	156	30.4	2802	100.0	9.8	3.9
PF2×NZ40	10.6	77.5	176	29.3	2397	95.7	11.6	4.3
D1275×M378-80	11.3	77.3	179	25.7	1936	100.0	7.9	1.8
PF2×NZ56	10.3	76.7	182	26.7	2258	94.3	10.8	3.2
PF2×NZ56	10.7	76.7	175	28.7	2251	96.7	9.2	2.4
PF1×NZS3-32	10.5	76.5	161	28.1	2302	98.3	8.0	3.1
Dea	12.4	76.2	163	30.8	2660	100.0	9.8	4.0
A676×NZ56	10.2	75.7	173	25.2	1962	99.3	8.1	2.8
N190×NZ2	10.9	75.6	175	24.9	1909	98.7	8.6	2.5
D1275×NZ84	10.9	75.3	177	24.5	1738	98.0	7.6	1.8
WGI207×BS22-22	11.2	75.3	176	22.0	1784	100.3	7.4	1.6
E1873×NZ3	10.3	75.2	178	25.8	2113	97.0	8.6	2.2
WGI207×M396-14	10.9	74.7	178	24.6	1989	95.7	9.1	2.3
P3902	11.1	74.5	177	25.7	1922	102.0	7.7	2.4
A665×M396-14	11.2	74.4	176	23.7	1848	100.7	7.1	1.8
WGI29×PF1	9.7	74.2	169	26.3	1956	96.3	8.7	3.0
P3787	10.9	73.5	174	23.7	1751	102.0	6.8	0.9
WGI207×M378-80	10.5	73.4	173	24.8	1863	98.3	8.7	1.7
A82-8×NZ84	10.1	73.1	167	24.8	1739	108.7	6.6	2.0
D1260×BS22-22	10.4	72.4	169	26.5	1802	103.3	7.0	1.5
Furio	10.3	72.2	162	26.5	1778	95.3	7.0	2.0
P3751b	10.5	71.2	174	26.2	1793	99.3	6.4	1.7
PAC42	10.4	70.9	166	25.6	1723	107.3	6.8	1.4
P3514	10.6	70.8	167	25.9	1671	102.7	6.2	0.7
A82-8×MBS847	10.4	70.4	170	22.2	1608	99.7	6.5	1.3
MBS847×NZS3-59	10.2	70.2	152	30.4	1750	104.0	7.2	1.4
P3901	10.0	70.2	163	23.9	1830	104.3	7.6	1.3
P3515	10.2	69.6	162	25.3	1625	104.0	5.6	0.7
P3751a	10.4	69.2	167	22.0	1726	102.3	7.5	0.6
P3394	10.4	69.1	168	24.2	1607	103.0	6.0	0.9
P3585	9.6	69.0	173	21.6	1539	101.7	5.9	0.8
PX74	10.6	68.6	171	25.5	1332	103.3	4.2	0.2
Test Statistic(F value)			*3.8*	*3.4*	*21.6*	*2.0*	*18.2*	*34.9*
Probability >F			*.0001*	*.0001*	*.0001*	*.0067*	*.0001*	*.0001*
Average CV within hybrids (%)			*3.5*	*8.4*	*6.1*	*4.2*	*7.9*	*18.7*

The data given in Table 4.1 show that trends exist. Higher bulk densities are associated with hybrids that have higher milling energies (E), greater resistance times (RT) and higher hard to soft endosperm ratios. Rankings of bulk density of entries which are duplicated but sourced from different areas of the field, P3751 and PF1 x BS22-39, are similar as are other measured parameters for these pairs of hybrids.

As one of the objectives of this work was to determine if the ratio of hard to soft endosperm was correlated with indirect but faster measurement techniques for grain hardness, a correlation analysis was carried out on the data. The correlation matrix resulting from measurements carried out on the 38 hybrids is illustrated in Table 4.2.

Table 4.2 Correlation matrix using SHT parameters, moisture content, bulk density and hard to soft endosperm ratio.

	MC:	BD:	PeakP	MT	E	H	RT	H/S
MC:	1.00							
BD:	0.33	1.00						
PeakP	0.18	0.32	1.00					
MT	0.13	0.36	-.26	1.00				
E	0.25	0.79	0.01	0.63	1.00			
H	-.05	-.43	-.31	-.17	-.34	1.00		
RT	0.18	0.73	0.23	0.40	0.79	-.50	1.00	
H/S	0.23	0.81	0.11	0.38	0.74	-.32	0.62	1.00

Note: MC: Moisture content
         BD: Bulk density
         PeakP   Peak power
         MT   Milling time
         E   Milling energy
         H   Height
         RT   Resistance time
         H/S   Ratio of hard to soft endosperm

It is clear from the data that some of the variables are correlated. The SHT milling energy and resistance time were highly correlated with H/S and therefore these properties are likely to be good estimators of grain hardness. However, H/S had a higher correlation coefficient with milling energy (E) (R^2=0.74) than with resistance time (RT) (R^2=0.62) suggesting that E was a better predictor of the proportion of hard endosperm.

In previous publications Pomeranz *et al.* (1985, 1986a, 1986b) measured only resistance time. However, the lower correlation coefficient between RT and H/S suggests that RT may be a less effective parameter for determining grain hardness than E or BD. The accuracy of the measurement of RT may be improved if a smaller tube is used to narrow the cross section thus reducing errors in the estimation of the tube filling.

Bulk density was also highly correlated with milling energy ($R^2=0.79$), resistance time ($R^2=0.73$) and H/S ($R^2=0.81$). It is therefore another good estimator of grain hardness. However, Hardacre (1997) have shown that bulk density may vary independently of E. This is due to variation in bulk density resulting from variation in grain shape which is independent of grain density. Correlation coefficients for peak power, milling time and the height of the meal in the collection tube with H/S were equal to or less than 0.5 and they are, therefore, less useful estimators of grain hardness.

A plot of E against H/S for all hybrids reveals some anomalies (Figure 4.1) which also occurred in plots of RT and BD against H/S (Figures 4.2 and 4.3). The hybrids, P3162, PF1 × BS22-78, and both entries of PF1 × BS22-39 had high H/S ratios but their milling energies and resistance times were lower than expected. P3162 also had a lower bulk density than expected from the H/S ratio. Deviations from the expected correlation may be due to these hybrids having softer kernel or endosperm texture than other hybrids. Another possible explanation is that the proportion of hard endosperm at the measured section of the kernel is not an accurate estimation of the volumes of hard and soft endosperm. Further evidence for this is discussed later. If these arguments are true, it is suggested that the modified Stenvert Hardness Test is a more accurate assessment of kernel hardness than the H/S ratio.

The presence of outliers in the correlation between milling energy and the ratio of hard to soft endosperm suggests that all measurements of kernel hardness should be based on a mechanical milling test such as that described here and not a visual assessment of the ratio of hard to soft endosperm area.

For the thirty eight hybrids harvested in the 92-93 season milling time was not correlated with the proportion of hard endosperm ($R^2=0.4$) and only moderately correlated ($R^2=0.6$) with the energy (E) required to mill the grains.
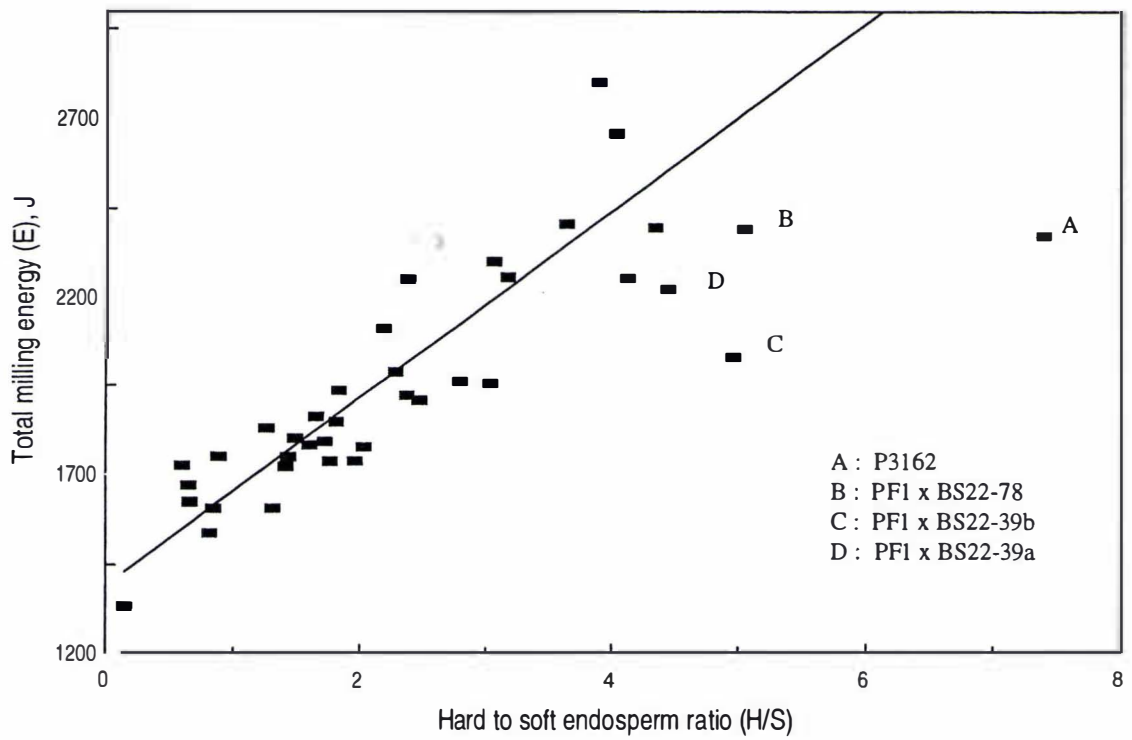
Figure 4.1 Relation between the hard/soft endosperm ratio (H/S) and the milling energy (E) for the 38 hybrids harvested in the 92-93 season. (Points represent 38 hybrids listed in Table 4.1.)
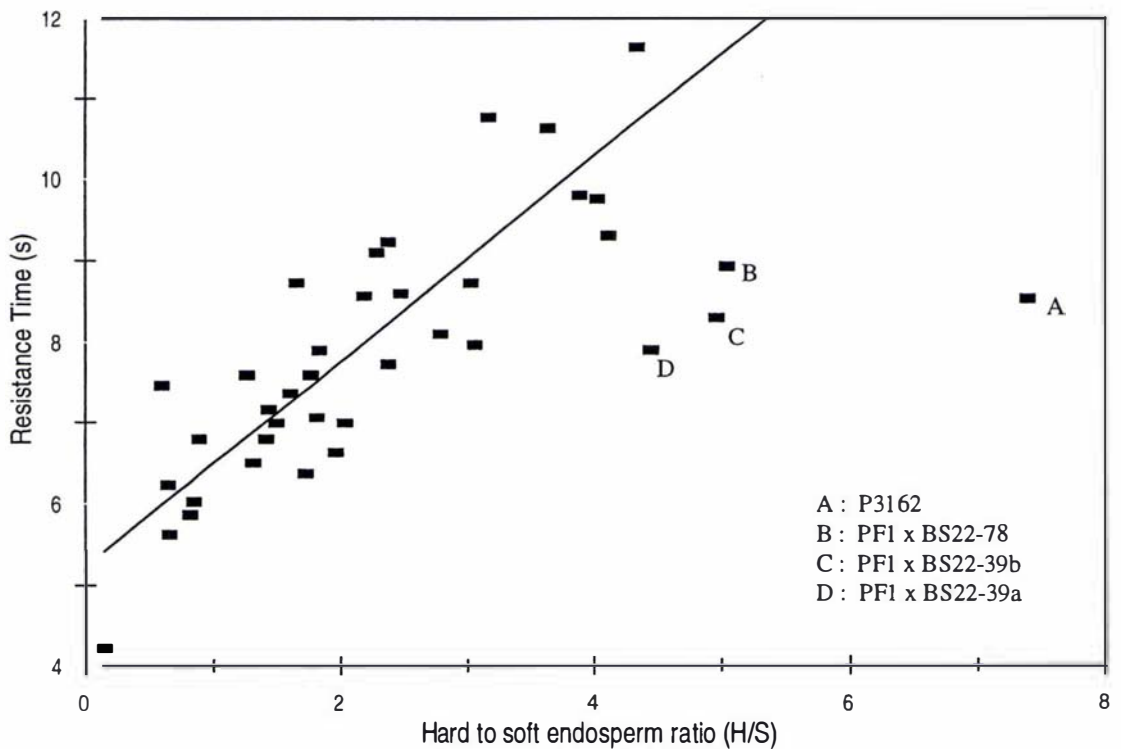
Figure 4.2 Relation between the hard/soft endosperm ratio (H/S) and the milling resistance time (RT) for the 38 hybrids harvested in the 92-93 season. (Points represent 38 hybrids listed in Table 4.1.)
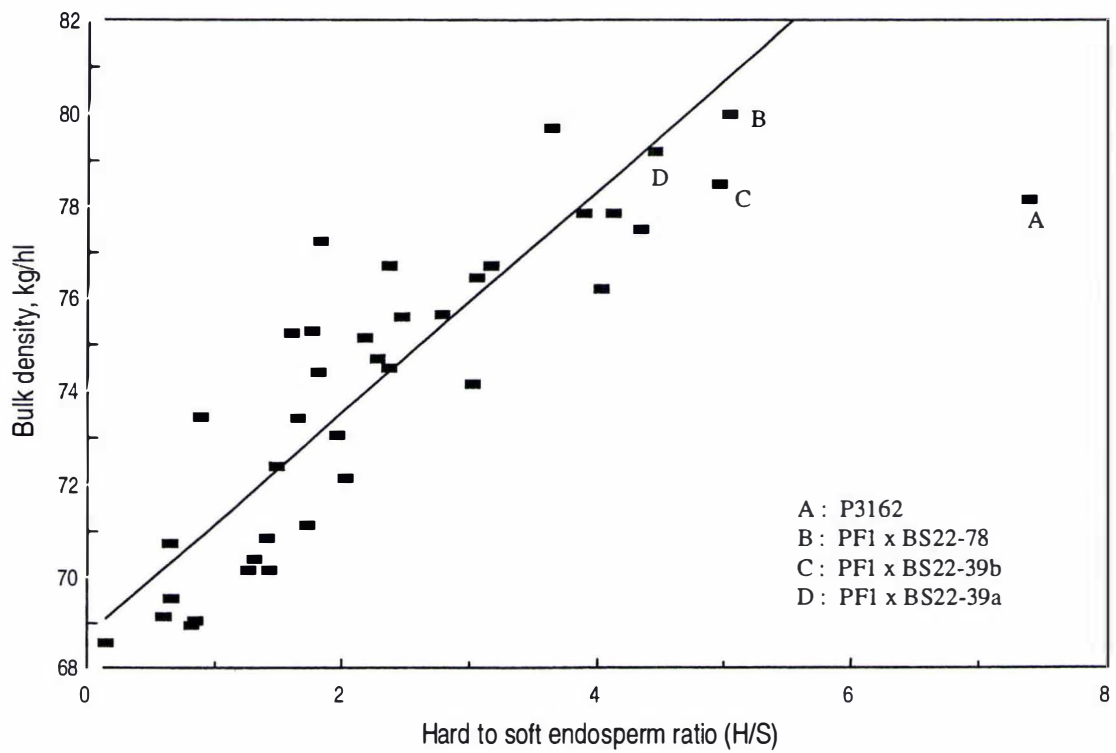
Figure 4.3 Relation between the hard/soft endosperm ratio (H/S) and the bulk density (BD) for the 38 hybrids harvested in the 92-93 season. (Points represent 38 hybrids listed in Table 4.1.)

4.2.1.2 Principal Component Analysis

Principal Component Analysis (PCA) (SAS 1988) is a multivariate technique used to reduce the number of variables in a data set. This is achieved by producing new variables called principal components (PC's) from correlated variables. The number of PC's produced equals the number of original variables since together these account for the total variation in the data. Typically, most of the variation is accounted for by the first one or two PC's. The eigenvectors generated by the PCA indicate the contribution of the original variables to the variation accounted for by each of the PC's. In contrast to multiple regression which fits variables sequentially and only considers improvements in an additive sense, PCA weighs all variables equally. Due to the high correlation among some of the variables obtained in this study it was considered appropriate to apply principal component analysis to combine the correlated variables and more accurately express a trait, in this case kernel hardness.

PCA revealed that 52% of the variability in the data could be accounted for by the first principal component (PC1) and only 22% by the second principal component (PC2). PC2 and the other five principal components were therefore ignored. From PCA, bulk density, milling energy and resistance time had the larger eigenvectors and are therefore the major contributors to the variability accounted for by PC1. Moisture content, milling time, peak power and sample height contributed less to the variation accounted for by PC1.

Table 4.3 shows that PC1 is highly correlated with bulk density ($R^2 > 0.74$), milling energy, resistance time and the ratio of hard to soft endosperm. In all cases, the correlations of these variables with PC1 are higher than the correlation of any of the variables with each other except for bulk density. Hardacre (1997) has shown that for some hybrids, bulk density gives a poor indication of milling energy. A rankings of hybrids based on PC1 is given in Table 4.4. Based in this ranking hybrid PF2×NZ40 is considered to be the hardest and PX74 the softest.

Table 4.3 Correlation matrix for the SHT parameters, hard to soft endosperm ratio and principal component PC1.

	PC 1	Bulk Density	Milling Energy	Resistance Time	Ratio of Hard to Soft Endosperm
PC 1	1.00				
Bulk Density	0.91	1.00			
Milling Energy	0.91	0.79	1.00		
Resistance Time	0.87	0.73	0.79	1.00	
Ratio of Hard to Soft Endosperm	0.84	0.81	0.74	0.62	1.00

Table 4.4 Rankings of hybrids according to the principal component PC1 and other parameters.

Entry names	PC1	PC1 Rnk	BD Rnk	E Rnk	RT Rnk	H/S Rnk
PF2×NZ40	3.38	1	8	4	1	5
Dea	3.07	2	13	2	5	7
PF1×AS3-94	2.99	3	2	3	3	9
PF2×NZ56	2.59	4	10	8	2	10
PF1×BS22-78	2.54	5	1	5	9	2
Hmv5313×PF1	2.39	6	7	1	4	8
PF2×NZ56	2.10	7	11	10	7	15
PF1×BS22-39a	1.82	8	3	11	18	4
PF1×NZS1-141	1.76	9	6	9	6	6
P3162	1.71	10	5	6	14	1
PF1×NZS3-32	1.09	11	12	7	17	11
WGI207×M396-14	1.06	12	19	14	8	17
E1873×NZ3	0.97	13	18	12	13	18
E1386×BS22-39b	0.97	14	4	13	15	3
D1275×M378-80	0.83	15	9	17	19	21
N190×NZ2	0.56	16	15	19	12	14
WGI29×PF1	0.34	17	22	16	10	12
A676×NZ56	0.18	18	14	15	16	13
WGI207×M378-80	0.06	19	24	20	11	25
D1275×NZ84	0.05	20	16	30	21	23
P3902	0.02	21	20	18	20	16
A665×M396-14	-0.43	22	21	21	26	22
WGI207×BS22-22	-0.51	23	17	25	24	26
Furio	-0.55	24	27	26	28	19
P3751b	-1.01	25	28	24	33	24
P3787	-1.09	26	23	27	29	32
D1260×BS22-22	-1.15	27	26	23	27	27
MBS847×NZS3-59	-1.52	28	32	28	25	28
P3514	-1.83	29	30	33	34	36
P3901	-2.01	30	33	22	22	31
A82-8×MBS847	-2.06	31	31	35	32	30
P3751a	-2.15	32	35	31	23	37
PAC42	-2.23	33	29	32	30	29
A82-8×NZ84	-2.27	34	25	29	31	20
P3394	-2.56	35	36	36	35	33
P3515	-2.76	36	34	34	37	35
P3585	-3.08	37	37	37	36	34
PX74	-3.28	38	38	38	38	38

Note: Rnk stands for 'Ranking'. Other abbreviations see Table 4.2.

4.2.1.3 Cluster Analysis of Different Hybrids

Cluster analysis (SAS 1988) was applied to separate the hybrids into groups based on all the variables (Figure 4.4). Two major groups were revealed, one with hard endosperm which includes the first 12 hybrids of Table 4.4 and a softer group comprising the remaining 26 hybrids. The group with hard endosperm comprises hybrids which are known to contain a high proportion of flint germplasm either from the inbreds PF1 and PF2 or of unknown origin for the commercial hybrids P3162 and DEA. It is interesting to note that most of the hybrids occurring in this group are based on crosses between flint and highland tropical germplasm. The crosses of highland tropical germplasm with non flint lines are intermediate in hardness, suggesting that highland tropical germplasm may be a useful source of grain hardness. These hybrid combinations also have good grain yield potential in the cool temperate New Zealand climate (Eagles and Hardacre 1985). The hybrids with softer grain often contain the inbreds MBS847 and A82-8 or are commercial hybrids which are used for grain production in New Zealand. Therefore it appears possible to improve kernel hardness by choosing the kernel properties of the hybrid parents.

Results from the cluster analysis show that the modified SHT was useful in assisting the maize breeding program as the hardness appears to be related to the hybrid parents.
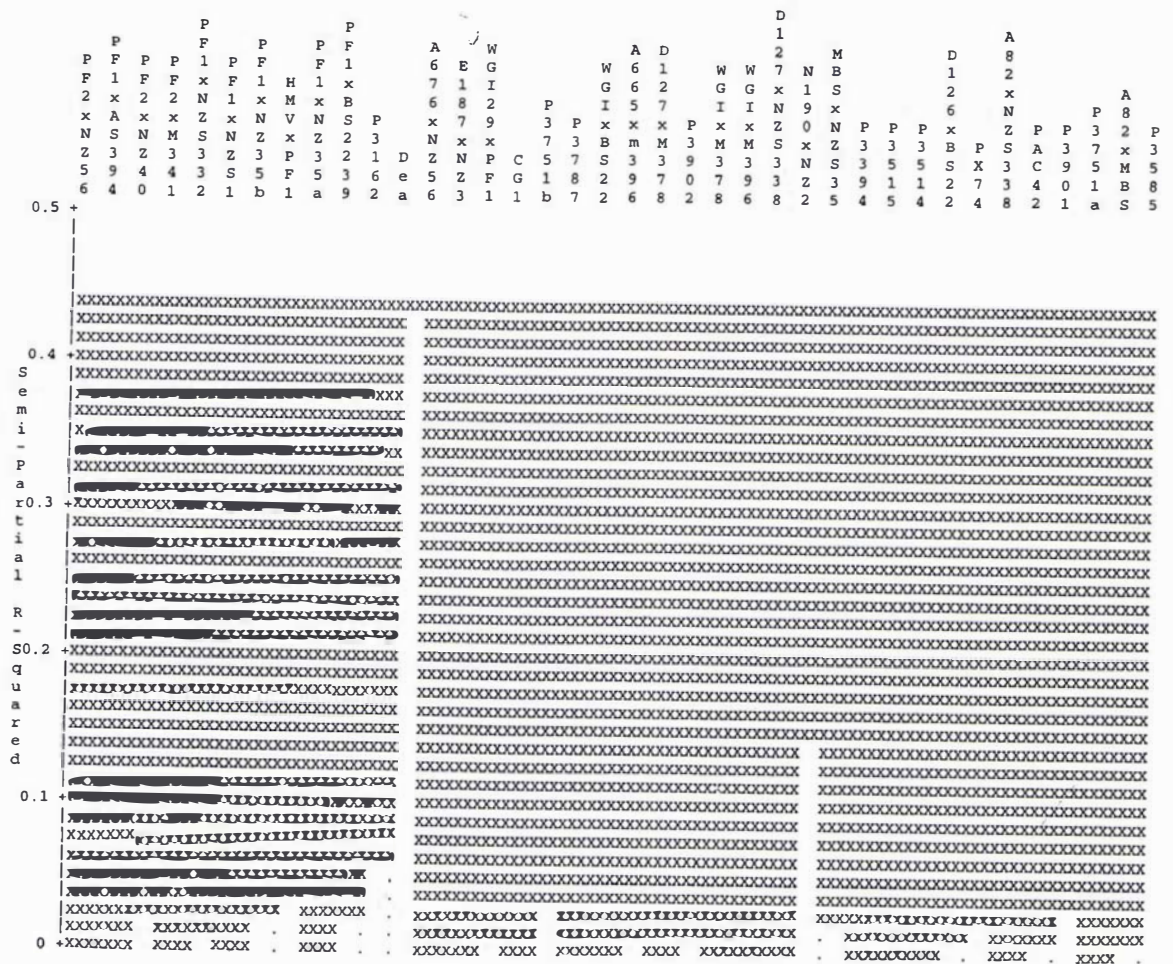
Figure 4.4 Plot of hybrids grouped by clusters.

4.2.2 Effect of Moisture Content on Grain Hardness and Bulk Density for 12 Hybrids Harvested in the 94-95 Season

As moisture content has a significant effect on the physical properties of corn grain, in order to compare differences in grain quality from different sources, these properties must be measured or corrected to the same moisture content. In industrial practice, it is difficult to obtain samples at a required moisture level. Therefore measurements of corn grain hardness and milling characteristics at different moisture contents need to be corrected to a standard moisture level. Thus, knowledge of the effect of moisture on these properties is important to ensure these data will be accurately corrected.

Twelve corn hybrids harvested in the 94-95 season (Table 3.2) were used to obtain bulk density and SHT data at different moisture contents ranging from 10.5 to 19.4%.

4.2.2.1 Effect of Moisture Content on Bulk Density

Figure 4.5 clearly shows that the relationship between grain moisture and bulk density was not linear. At moisture contents above 14% changes in bulk density with moisture content were greater than those at moisture contents below 14%. This was, probably, due to the fact that at high moisture levels, the grain size and volume played an important role in bulk density and it changed more significantly than the weight when gaining or losing water. At low moisture contents the grain becomes more solid and rigid, therefore, resisting shrinkage and large changes in volume. For moisture content less than 12%, bulk density changed only slightly with moisture content.

The effect of moisture content on the grain bulk density has been studied by several researchers. Chung and Converse (1971) used two lots of yellow dent corn and found that the relationship of moisture to bulk density followed the model given by Equation (4.1):

$$Bulk\ Density = \frac{7270 - 112 \cdot MC}{100 - MC} \qquad (4.1)$$

where $Bulk\ Density$ is in kg/hl and MC is the moisture content in percentage (%). Nelson (1980) summarised the relationship between moisture content and bulk density,

using averaged data from twenty-one lots of yellow dent corn, by the following equation:

$$BulkDensity = 68.3 + 1.422 \cdot MC - 0.09843 \cdot MC^2 + 0.001548 \cdot MC^3 \quad (4.2)$$

For given moisture contents, Equations (4.1) and (4.2) give different estimations of bulk densities. For instance, at a moisture content of 14%, bulk density is 66.3 kg/hl from Equation (4.1) but it becomes 73.2 kg/hl when calculated using Equations (4.2). Furthermore, these two equations do not take into account the effect of hybrid which is very significant as shown in the current research (Table 4.1). Corn grains with similar moisture content but from various sources can have different bulk densities. These indicate that the above relationships are not satisfactory and are less useful when different hybrids are considered. Thus, a more accurate and reliable estimation of bulk density for different moisture content and different hybrids is needed.

Figure 4.5 shows that grains from different hybrids had different bulk density but the slope of the curves for all hybrids were very similar. This suggests that these curves can be reduced to a master curve by shifting their position along the y-axis. A superposition technique was used to shift BD data into a master curve as showed in Figure 4.6. The shift factor was determined as:

$$F_{hybrid} = \frac{\int_{MC_{min}}^{MC_{max}} BD \cdot dMC}{MC_{max} - MC_{min}} \quad (4.3)$$

where F_{hybrid} is the hybrid dependent shift factor, kg/hl, MC_{max} and MC_{min} are the upper and lower limits of the moisture content measurements for each hybrid.

The master curve was fitted using a third degree polynomial equation as follows

$$BulkDensity = F_{hybrid} + b \cdot MC + c \cdot MC^2 + d \cdot MC^3 \quad (4.4)$$

where $b=0.0778$, $c=0.0372$ and $d=-0.0027$. The correlation coefficient of the model was 0.979.

The master curve can be used to determine bulk densities of grains of a particular hybrid at different moisture contents. The hybrid dependent factor F_{hybrid} can be determined

from equation (4.4) provided the bulk density of the grain at a given moisture content is known.
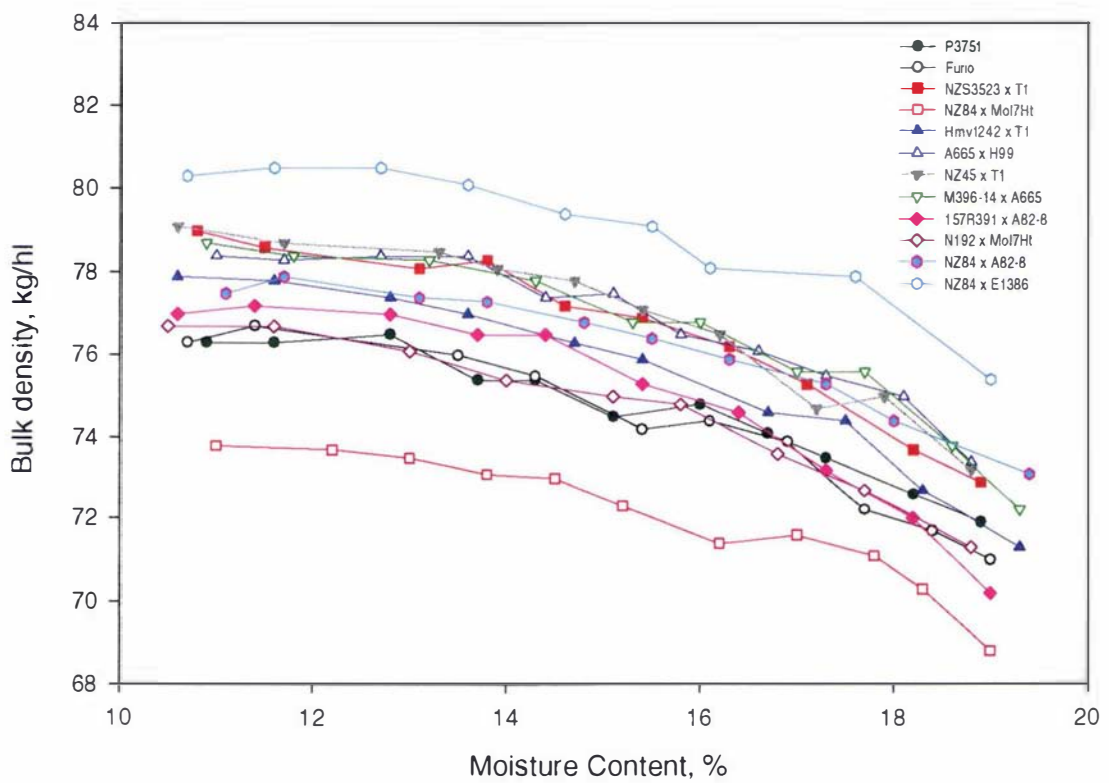
Figure 4.5 Effect of moisture content on bulk density for the hybrids harvested in the 94-95 season.
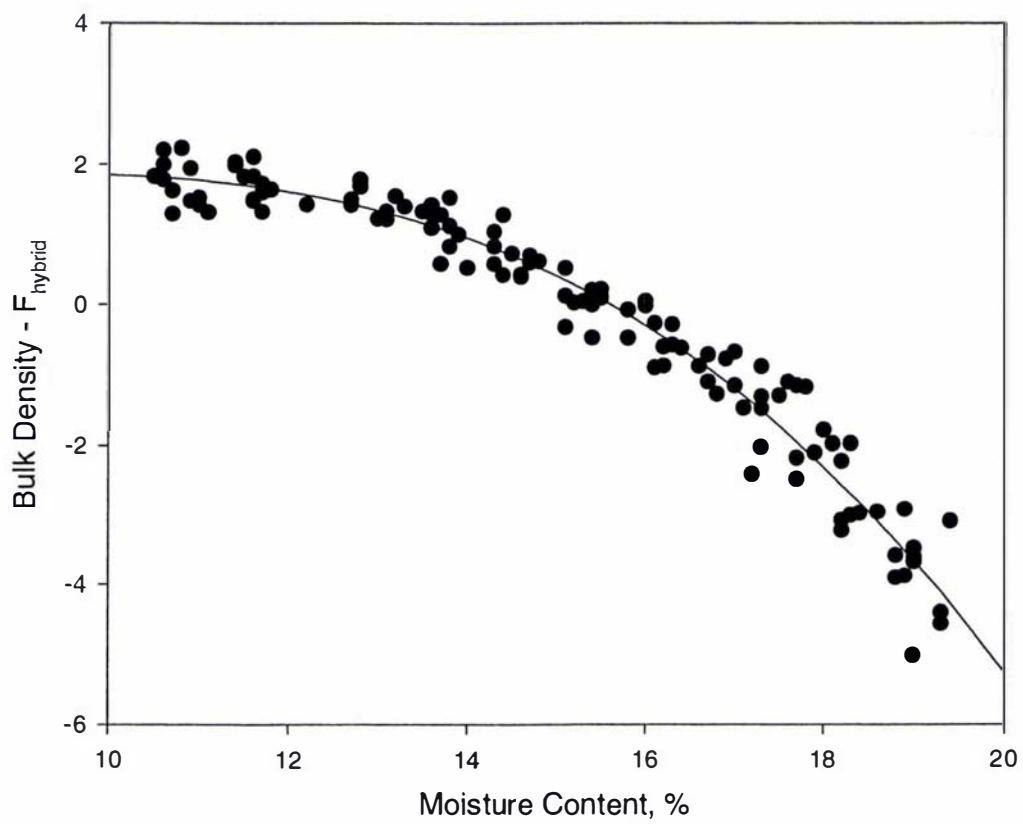
Figure 4.6 Master curve for effect of moisture content on bulk density.

4.2.2.2 Effect of Moisture Content on the SHT Milling Energy

The effect of moisture content on grain hardness, measured as the milling energy (E), follows the same tend for all the hybrids, Figure 4.7. With increases in moisture content, the corn grain becomes tougher and milling energy E increases.

Dorsey-Redding *et al* (1990) reported that moisture content had an effect on the sample column height of the SHT test. A single exponential function and a linear model were used to describe the relationship between moisture content and the column height of the milled sample. However, this model can not account for the differences among hybrids and is not appropriate to describe the relationship between moisture content and milling energy for all the hybrids studied in this research.

Figure 4.7 shows that for a single hybrid, a nearly linear relationship exists between moisture content and milling energy. However, moisture content affects the hardness differently in hard and soft hybrids. Therefore, it is necessary to develop a new model to describe this relationship. The model was obtained using a two-step regression. Firstly, by assuming that the milling energy (E) changes linearly with moisture content, a linear model for each hybrid was obtained using linear regression:

$$E = E_{ID} + C_{ID} \cdot MC \tag{4.5}$$

where E is milling energy, in joules, C_{ID} and E_{ID} are hybrid dependent coefficients, in joules, and MC is the moisture content between 10% to 20% wwb. Different values of E_{ID} and C_{ID} obtained using linear regression for all hybrid studied are shown in Table 4.5. Figure 4.8 shows that the parameters are linearly correlated. The equation that fits the values of E_{ID} and C_{ID} can be expressed as:

$$E_{ID} = 3403.3 - 6.97272 \cdot C_{ID} \tag{4.6}$$

Substituting E_{ID} given by Equation (4.6) into Equation (4.5) results

$$E = 3403.3 + (MC - 6.97272) \cdot C_{ID} \tag{4.7}$$

Equation (4.7) can then predict the milling energy (E) of corn hybrids at different moisture contents provided the coefficient C_{ID} is determined or known.

To evaluate the suitability of the model, plots of measured and estimated milling energy (E) for four typical hybrids ranging from soft to hard are shown in Figure 4.9. Values of

C_{ID} for each hybrid were determined using the milling energy measured at a range of moisture contents of 13.5% to 14%. The measured and estimated data have correlation coefficients of 0.96, 0.97, 0.99 and 0.98 for the four hybrids.
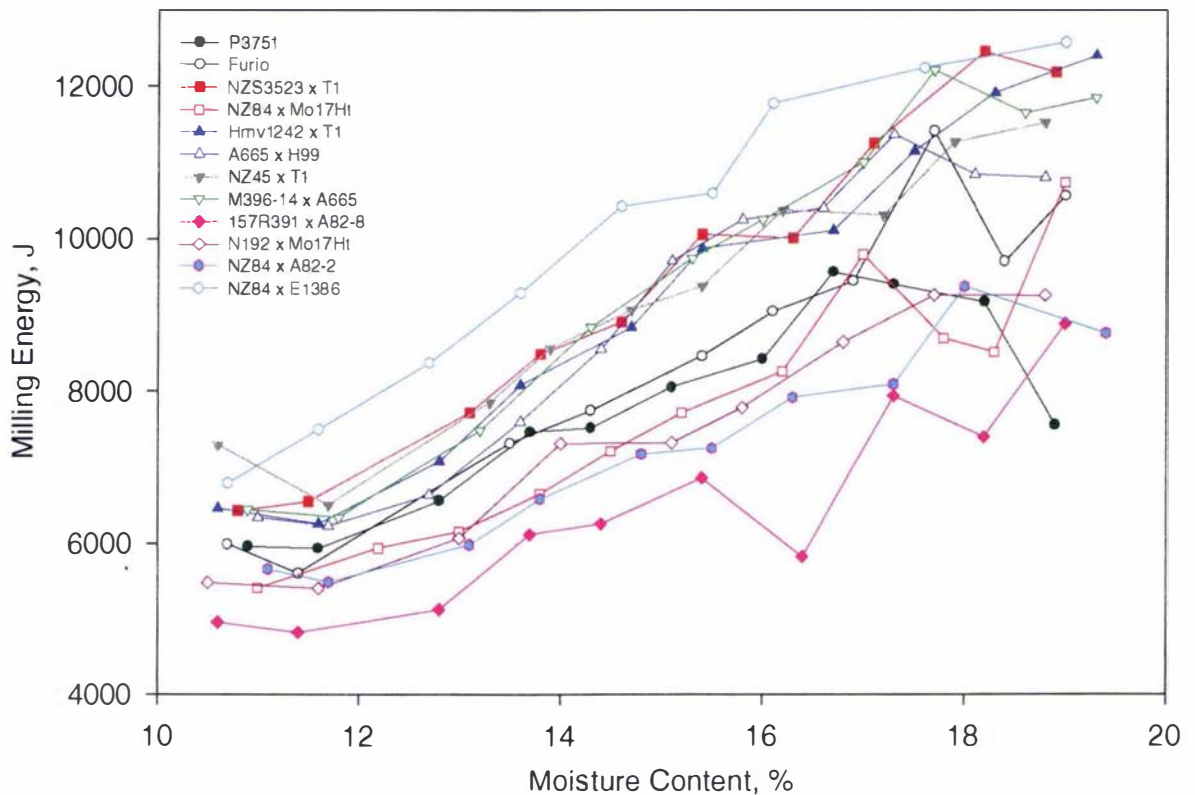
Figure 4.7 Effect of moisture content on the SHT milling energy for hybrids of the 94-95 season.

Table 4.5 Values of the hybrid-dependent parameters E_{ID} and C_{ID} given in equation (4.5) for the 12 hybrids of the 94-95 season.

Hybrid Name	E_{ID}	C_{ID}
P3751	430.85	499.41
FURIO	-767.04	601.42
NZS3-523-1-1-1 x T1	-2420.40	791.91
NZ84 x Mo17Ht	-1330.78	591.37
Hmv-124-2 x T1	-2203.83	759.84
A665 x H99	-1739.97	709.66
NZ45 x T1	31.32	613.66
M396-14-1-1-1-1-1 x A665	-2135.39	761.38
157-R-3-9-1 xA82-8	-312.91	465.4
N192 x Mo17Ht	-444.87	530.88
NZ84 x A82-8	86.81	475
NZ84 x E1386	-1007.66	751.33
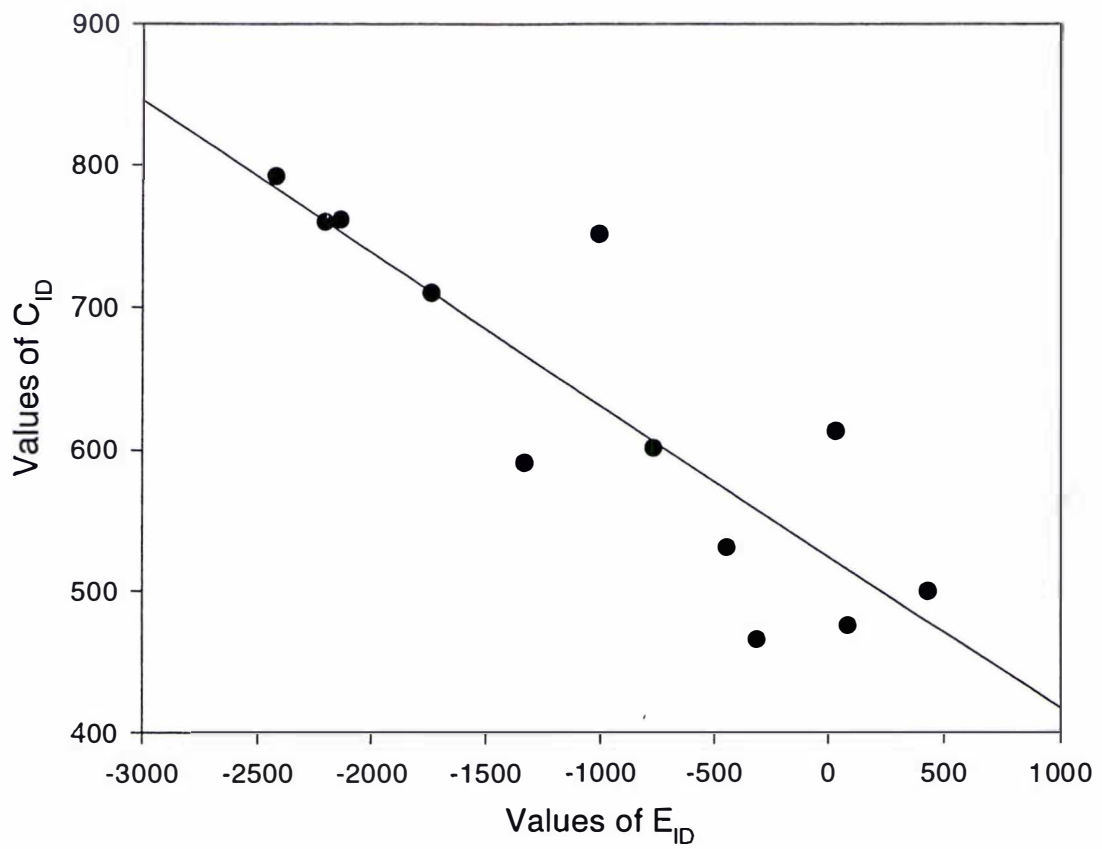
Figure 4.8 Plot of C_{ID} and E_{ID} parameters given in Table 4.5 for the 12 hybrids of the 94-95 season.
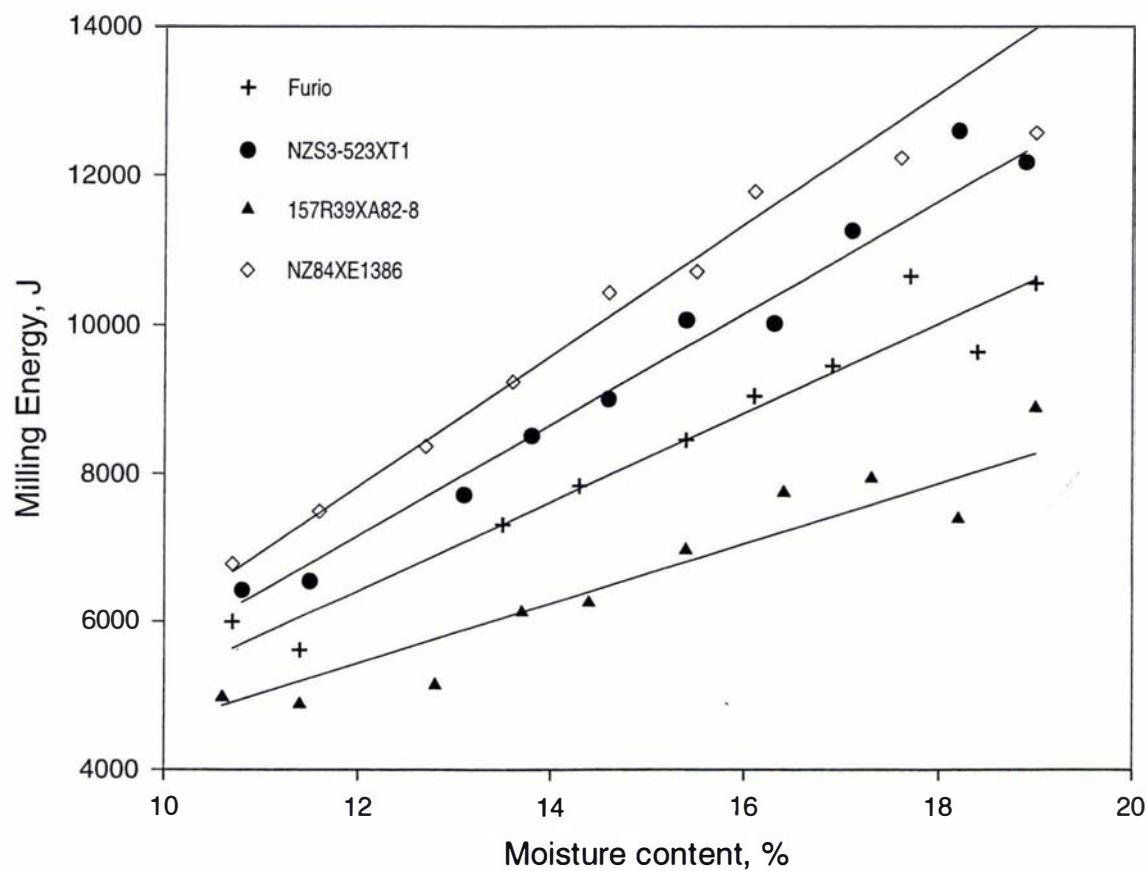
Figure 4.9 Comparison between experimental data and milling energy predicted by Equation (4.7). Symbols are experimental measurements and the lines represent the estimated values.

4.2.3 Particle Size Distribution of SHT Samples

In order to study the particle size distribution, ground samples obtained from the SHT tests for all 38 hybrids from the 92-93 and 12 hybrids from the 94-95 seasons were sieved. Soft grains have more soft endosperm that can easily be broken into small pieces and more flour is obtained. Conversely hard grains need more time and energy to reduce in size but grit can be swept out through the mill screen before they can be further processed, resulting more grit than flour.

At an average moisture content of 11% four typical particle size distributions selected among the 38 hybrids of the 92-93 season are shown in Figure 4.10. The particle size distributions of hard and soft grains show the same general trend. A large amount of flour (size<0.30mm) was produced with all samples, however, more flour was produced by soft grains PX74 (45.4%) and P3585 (30.7%) than by hard hybrids Dea (17.3%) and P3162 (27.5%). Differences among hybrids in producing grit of size ranging from 0.699mm to 1.000mm are also clearly shown in Figure 4.10. P3612 and DEA produced 29.5% and 24.2% of grits respectively, while soft grains P3585 and PX74 produced only 19.2% and 13.4% respectively. These results agree with the industrial practice of using hard grains for production of grits sizing from 0.699mm to 1.000mm.

SHT hardness (E) and grit recovery (Gr) with sizes ranging from 0.699 to 1.470mm were highly and positively correlated (R=0.92). Figure 4.11 shows that grit recovery of different hybrids can be predicted by measuring the milling energy E. The relationship between grit recovery and E measured at 10.5% MC can be expressed by the following equation (R^2 =0.943):

$$Gr=-39.87 + 0.0552 \cdot E - 0.0000089 \cdot E^2 \tag{4.8}$$

Grit recovery is also strongly correlated with bulk density (R=0.80) indicating that this parameter may also be used to predict grit recovery.

As moisture content largely affects milling energy E and bulk density, therefore, it is expected that grit recovery rate will be also affected by the grain moisture content. Figure 4.12 shows the effect of moisture content on grit recovery. For both hard and soft

hybrids, there is an optimum grit recovery for moisture contents ranging 13% to 14%. The grains become brittle at very low moisture content (10.1 to 10.6%) or soft at high moisture content (18%), therefore, they can be more easily reduced to small fragments producing more flours and a less grits.
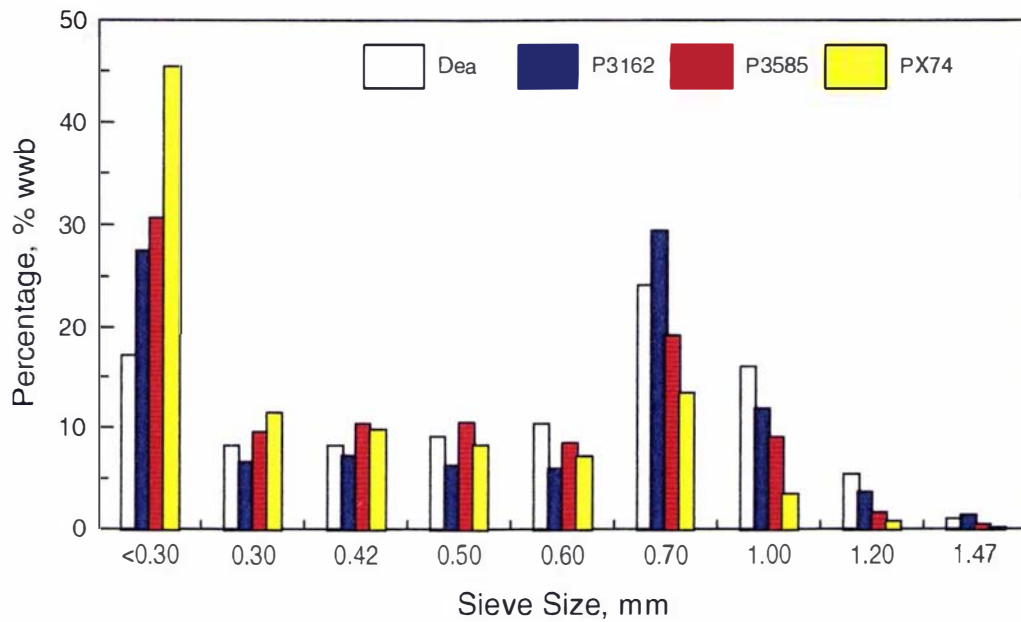
Figure 4.10 Particle size distribution of ground SHT samples.
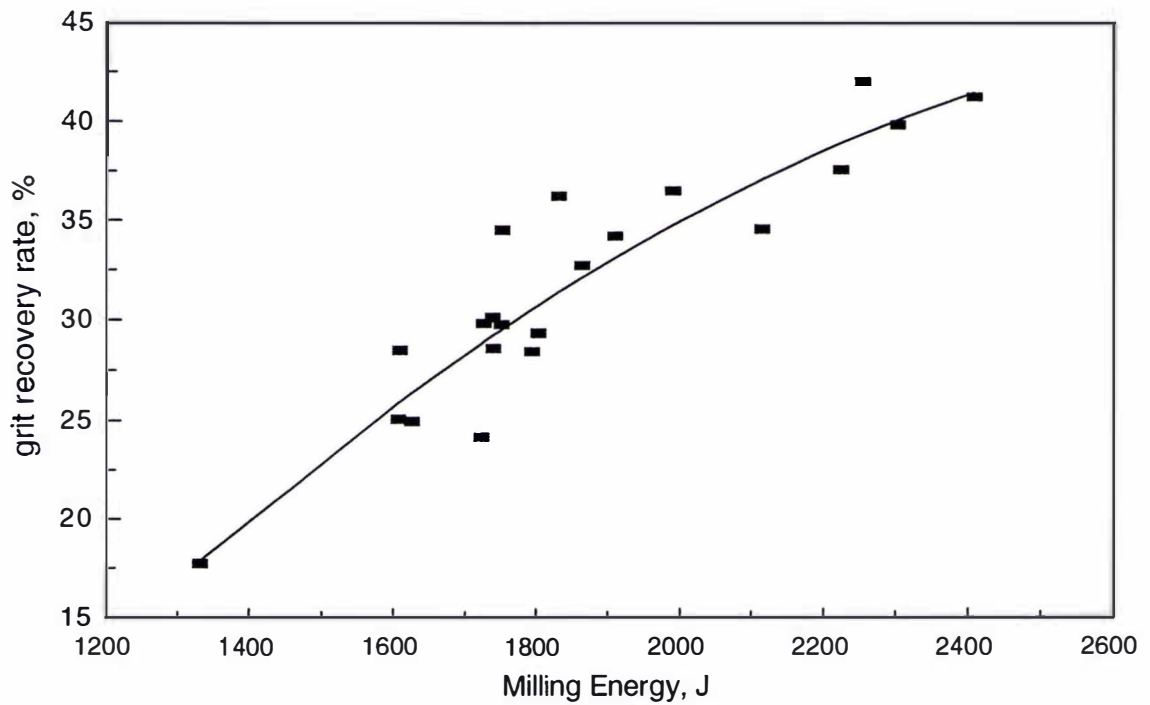
Figure 4.11 Relation between milling energy and recovery rate of grits with sizes ranging from 0.699 to 1.470mm.
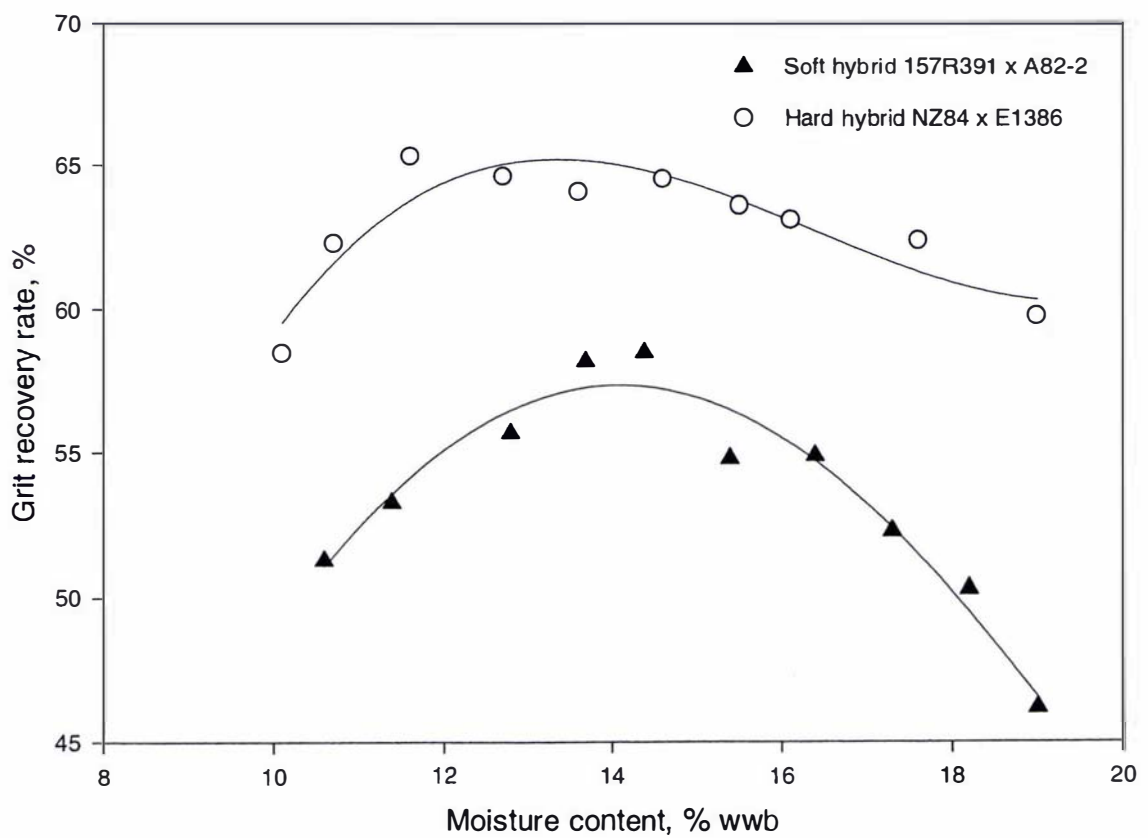
Figure 4.12 Effect of moisture content on grit recovery rate on hard and soft hybrids.

4.2.4 Roller Milling Test

Roller milling is one of the most commonly commercially utilised milling operation. There is not much reported information concerning the use of roller milling to characterise corn grain hardness. Therefore, it was necessary to study the interaction between the roller milling performance and corn grain hardness. A roller milling test was conducted for the twelve corn hybrids harvested in the 94-95 season at a moisture content of about 12%.

4.2.4.1 Grain Breaking Force in Roller Milling

The roller milling test used was a two-stage milling process. The force recorded for the first break showed little difference between hybrids. A plot of milling force measured for two typical hybrids, hard grain NZ84 x E1386 and soft grain 157-R-3-9-1 x A82-8, is illustrated in Figure 4.13. The data variability is very large (148% and 98% for NZ84 x E1386 and 157-R-3-9-1 x A82-8 respectively) indicating the poor quality of the data. The correlation between SHT hardness (E) and the milling force for the first break was very low (0.12). During the first milling stage a large gap (3mm) between rollers and a small pre-loading force (22kgf on load cell) were used. With this setting the rollers were easily pushed apart resulting in an uneven feed rate and large variation in the force measured. A large quantity of unbroken grains was observed with the milled corn. For this reason the measurements were not reliable and could not be used as an index of hardness.

Data recorded from the second stage milling process, however, showed significant differences among samples. Table 4.6 indicates a very good agreement between the breaking force measured by the roller mill and the milling energy (E) measured using the SHT. Figure 4.14 illustrates that higher values of breaking force were recorded when hard grains were tested. The correlation between SHT milling energy and the roller milling breaking force is very strong (r=0.87). These results clearly show that a better setting of the experimental conditions (a high pre-load force and a small gap) could provide better and more consistent results. There was less variation (10% and 8% for NZ84 x E1386 and 157-R-3-9-1 xA82-8 respectively) in the results of the second break. As the products produced during the first break were used for the second milling stage, data

measured for the second break should not be regarded as the true breaking force for the whole corn kernel. Products from the first-break milling consist of grit, partially broken grains and small amount of unbroken grains. When the materials were fed into the mill again for the second break, large pieces of endosperm together with unbroken grains underwent the second crush and tear during the process. Thus, the force applied on the rollers reflected the strength of the mixture of endosperm and grains, not the hardness of the whole grain or endosperm hardness alone.
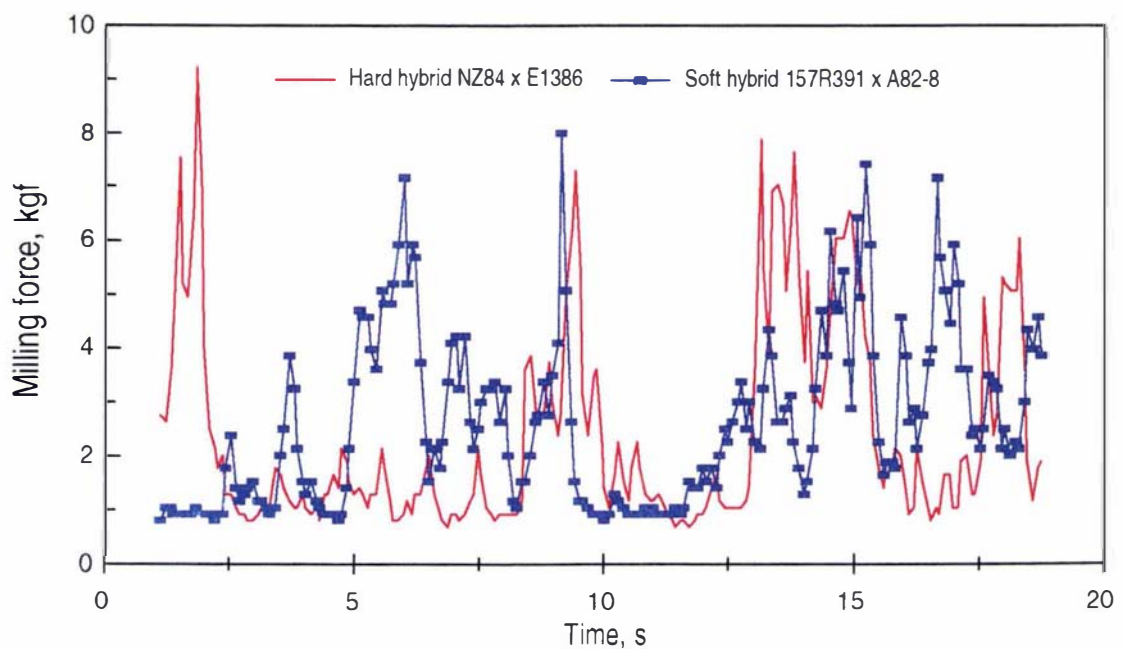
Figure 4.13 Plot of the transient roller milling breaking force during the first-break milling stage.

Table 4.6 Average force of roller milling 12 hybrids

Hybrid	2nd break force, kgf	SHT energy, J
157-R-3-9-1 xA82-8	4.11	4882.5
NZ84 x Mo17Ht	3.84	5356.5
N192 x Mo17Ht	4.01	5494.8
NZ84 x A82-8	4.30	5503.3
FURIO	4.31	5999.6
Hmv-124-2 x T1	4.62	6172.8
A665 x H99	4.49	6239.7
M396-14-1-1-1-1-1 x A665	4.68	6451.9
NZ45 x T1	4.87	6512.3
NZS3-523-1-1-1 x T1	5.19	6552.7
P3751	5.07	6561.2
NZ84 x E1386	5.38	8371.4
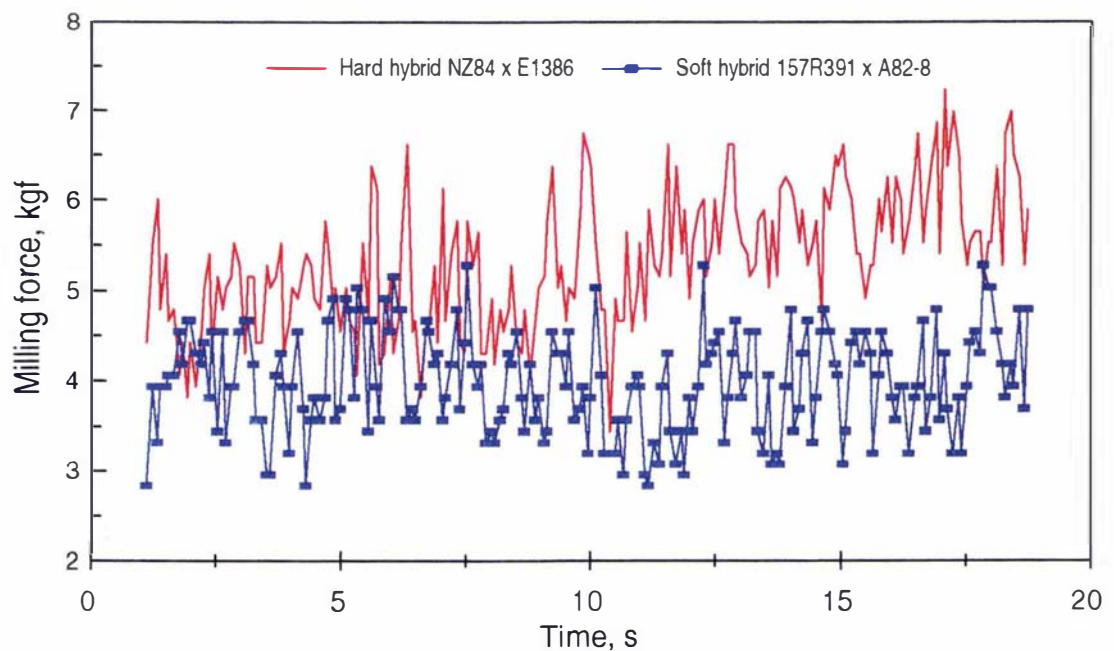
Figure 4.14 Plot of the transient roller milling breaking force during the second-break milling stage.

4.2.4.2 Particle Size Distribution of Samples from Roller Milling

Figure 4.15 illustrates the particle size distribution of all hybrids after the roller milling operation. In this figure the size distribution is unimodal and there are no major differences among hybrids.

Grit recovery, however, appears to be different. For instance, the hard hybrid NZ84 x E1386 produced about 10% more grit of size ≥2mm than did the soft hybrid 157-R-3-9-1 xA82-8. The correlation between milling energy and grit recovery during roller milling was not high. The highest correlation coefficient (0.64) occurred for grit of size ≥1.4mm, and confirmed again that harder grain increases the grit yield.

The set-up of the roller milling test for this study was not efficient as the data obtained had a high variability in some cases higher than variation among hybrids. The results, however, provided some valuable information about the dry milling performance of corn and confirmed the results obtained by the SHT. These results suggest that a roller mill suitable for corn would improve the accuracy of the dry roller milling test. Furthermore, the test could be carried out by using a single milling process to determine the strength of the grains.
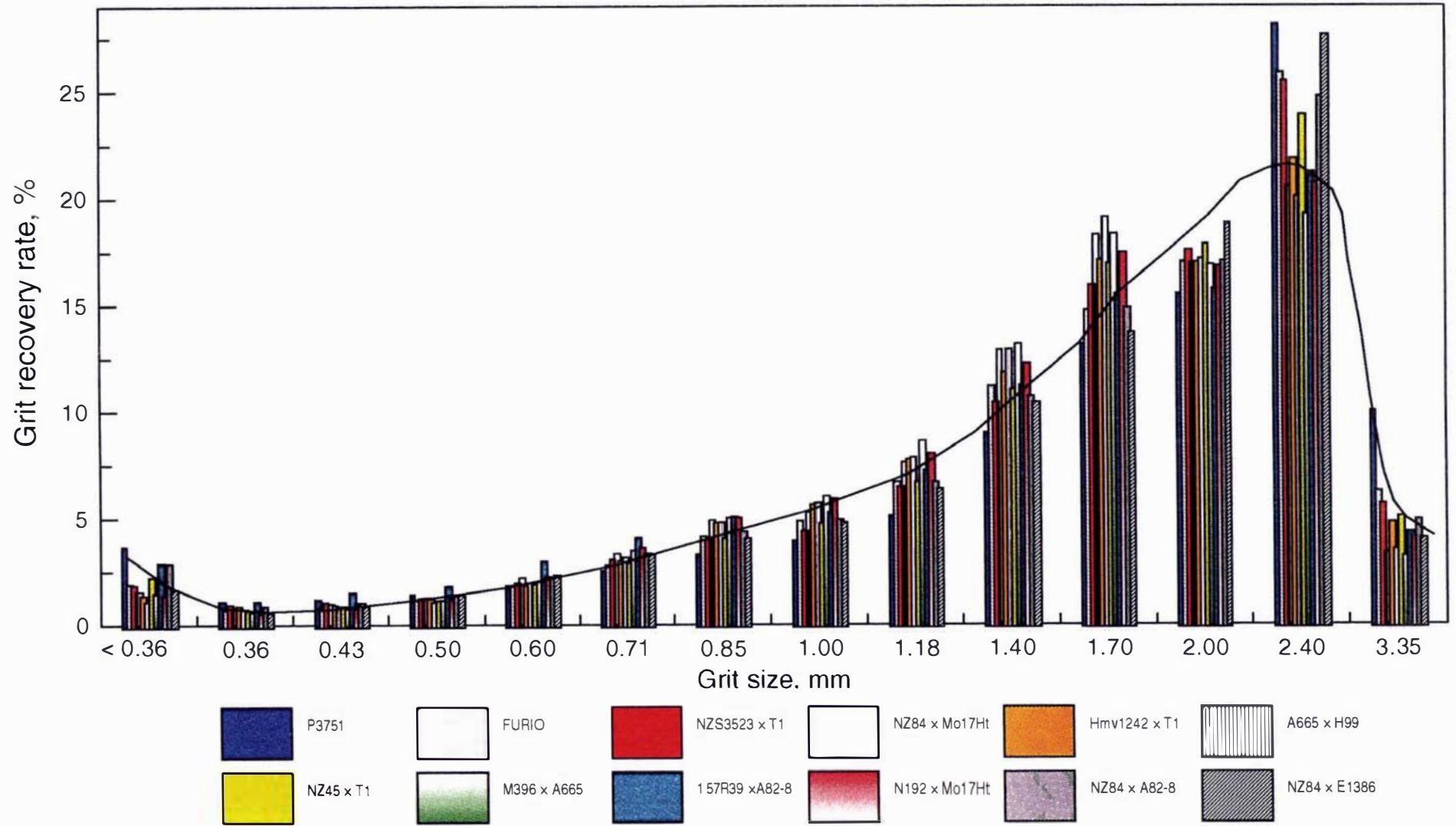
Figure 4.15 Particle size distribution of samples obtained from the roller milling operation, second break.

4.3 Conclusions

From these results, it is concluded that:

i)   The modified Stenvert Hardness Test (SHT) is a quick and simple method that can be used to compare the endosperm hardness of diverse maize hybrids.

ii)   At the same moisture content, SHT milling energy E can effectively distinguish differences in hardness among hybrids.

iii)   It is clear that the hard to soft endosperm ratio of the maize hybrids were closely correlated with grain hardness measured by the SHT and to some extent with bulk density.

iv)   The simplicity, accuracy and the small amount of sample required for the SHT suggest that this test is more convenient to carry out routine quality control of corn.

v)   All measurements of kernel hardness should be based on a mechanical milling test such as that described here and not a visual assessment of the ratio of hard to soft endosperm area.

vi)   Corn kernel hardness can be altered by choosing the kernel properties of the hybrid parents.

vii)   Grain moisture content has a large effect on bulk density, hardness, and the yield of grit during milling.

viii)   Corn grain hardness and milling characteristics should be determined at the same moisture content for all samples.

ix)   The roller milling experiments provided valuable information for the corn dry milling process. Grit recovery and breaking force were related to corn grain hardness.

Chapter 5

Literature Review: Extrusion Processing

5.1 Introduction

The quality of expanded extruded products is commonly determined by their physical properties such as bulk density, cell structure, expansion ratio, mechanical strength and sensory characteristics such as crunchiness and crispness. It is thought that these properties are closely related to and controlled by the rheological properties of the melt before it exits the extruder die (Fan *et al.*, 1994; Bouzaza *et al.,* 1996). The rheological properties of the melt vary largely with the product formulation and the operating conditions of the extrusion process (Colonna *et al.*, 1984; Chinnaswamy and Hanna, 1988; van Lengerich, 1990; Lai and Kokini, 1991; Harper and Tribelhorn, 1992; Vergnes *et al.,* 1993; Frame, 1994).

Figure 5.1 illustrates the variables involved in the extrusion process and their interactions. Despite intensive research on food extrusion during the last decade, the relationships between product quality, processing conditions, raw materials and melt rheology are not well understood yet.

As illustrated in Figure 5.1, the number of variables involved in the extrusion operation is large. It is clear that some of these variables, such as raw material and the extruder operational variables, can be regarded as input variables. The interactions among the input variables results in operational conditions which yield another group of variables such as Specific Mechanical Energy (SME), Residence Time Distribution (RTD), temperature, pressure and viscosity of the melt. These can be regarded as the process variables. The process variables, in turn, influence the product quality defined by a group of variables such as expansion, bulk density, and product morphology. Measurements defining the product quality can be regarded as output variables.

RAW MATERIAL
Composition: Oil, starch, protein, fiber
Moisture
Particle Size
Additives

SYSTEM DISTURBANCES
· Screw Wear
· Ambient Conditions

DIE PROFILE
· Open Area
· Flow Resistance

PRODUCT QUALITY
· Product Moisture
· Product Temperature
· Bulk Density or Expansion
· Morphology
  Shape
  Size
  Uniformity
· Texture/Sensory
· WSI/WAI
· Gelatinisation
· Dextrinisation

OPERATIONAL VARIABLES
Feed Rate
Water Addition
Screw Speed
Screw Profile
Cutter Speed
Barrel temperature

SYSTEM PARAMETERS
· SME
· Residence Time
· Thermal Energy
· Product Temperature
· Product Pressure
· Melt Viscosity

SME: Specific Mechanical Energy
WAI: Water Absorption Index
WSI: Water Solubility Index
GELATINISATION
DEXTRINISATION (Molecular Degradation)

Figure 5.1 List of extrusion parameters.

The large number of variables involved in extrusion and the complexity of the interactions occurring during the process make it very difficult to establish relationships among variables that could help to understand the process and the effect of theses variables on product quality.

Because of this difficulty, a simplified approach for research in food extrusion has been adopted in the past. In a large number of studies, the effect of only one variable at a time was investigated. This approach diverts the study into different directions or fields, which focused on only a part of the whole extrusion process. If the whole process is investigated, the approach would involve such a large number of trials as to make the study impractical. Although the approach has its disadvantages, it has been widely used and accepted in the field of extrusion research.

By defining important variables and establishing the basis of the interactions of these with the product quality, progress towards modelling the interactions among extruder performance and input and output variables can be made. One of the crucial measurements defining product characteristics is the viscosity of the melt within the extruder barrel. A considerable proportion of this work is devoted to this aspect. If the melt viscosity could be properly measured, it would help in understanding not only the extrusion process, but also would aid in the design of an extrusion control system.

The research focused on the analysis and measurement of selected and representative variables notably viscosity of the melt, degree of starch gelatinization and mechanical energy input. Since one of the main objectives in this research was concerned with the development of appropriate methods for on-line determination of melt viscosity, most of the literature review was concentrated on this subject. Although out of the scope of this project relationships between melt viscosity and the properties of final product are also reviewed. The review was carried out according to the following.

i)   The effect of rheological properties on the product expansion and the importance of the melt viscosity in extrusion.

ii) The effect of the raw materials and the extrusion operating conditions on the rheological properties of the melt.

iii)   On-line techniques for measuring viscosity during food extrusion and their advantages and disadvantages.

5.2 Rheological Model of the Melt and Product Expansion

5.2.1 Rheological Properties of the Melt

A power-law rheological model has been used to describe the behaviour of the melt. The model, although empirical, fits the data reasonably well, and predicts the fluid flow adequately (Steffe, 1992). The model can be expressed as:

$$\tau = K\dot{\gamma}^n \tag{5.1}$$

An apparent viscosity η_{app} can be defined as

$$\eta_{app} = \frac{\tau}{\dot{\gamma}} \tag{5.2}$$

By using the power law model given by Equation (5.1) the apparent viscosity η_{app} can be expressed as:

$$\eta_{app} = K\dot{\gamma}^{n-1} \tag{5.3}$$

τ is the shear stress in Pa (Pascal); η_{app} is the apparent viscosity in Pa·s; K is the consistency index in Pa·s^n; $\dot{\gamma}$ is the shear rate in 1/s; and n is the power law index.

The value of n defines three types of fluids. Liquids with value of $n=1$ are known as Newtonian fluids. Their viscosity is independent of the shear rate and is only a function of temperature. Fluids with $n<1$ are known as pseudoplastics or shear thinning fluids. Their viscosity is a function of shear rate and decreases when shear rate increases. Materials with $n>1$ are known as dilatant or shear thickening fluids. Their viscosity increases when shear rate increases.

The rheological properties of the melt are directly influenced by the structural changes such as starch gelatinization in the raw materials from which the melt is formed (van Lengerich, 1990). These structural changes in turn are influenced by the intensity and amount of mechanical and thermal energies introduced during extrusion.

It is important to note that the rheological properties of the melt control not only the extrudate properties, but also influence the transport of material (momentum and heat transfer) in the extruder and determine its pressure build-up. Data on the viscometric properties of the melt are also essential for reliable scale-up of laboratory processes (Bruin *et al.*, 1978; Lai and Kokini, 1990; van Lengerich, 1990).

5.2.2 Viscosity and Expansion

The structural and textural quality of directly-expanded extrudates depend on the expansion mechanism of the melt (Launay and Lisch, 1983). When the melt exits the die it is subjected to a large pressure drop between the die and the atmosphere. This sudden pressure drop causes an extensive flashing-off of superheated water which forms bubbles in the molten extrudate. The formation and expansion of the bubbles in the molten extrudate result in the rapid conversion of the melt into a foam (Bouzaza *et al.*, 1996). As a result of the cooling effect produced by water evaporation, the melt solidifies and a porous structure is retained.

Assuming that effects of elasticity and surface tension in the melt are negligible, Amon & Denson (1984) showed that the expansion process is controlled by the pressure-drop/viscosity ratio $\Delta P/(\mu_a)_d$. ΔP is the difference between the vapour pressure in the melt and atmospheric pressure and $(\mu_a)_d$ is the apparent viscosity of the melt at the die. Kokini *et al.* (1992a) showed that the specific volume of the extrudate is linearly related to the pressure-drop/viscosity ratio $(\Delta P/(\mu_a)_d)$ at constant moisture content.

Using a theoretical model, which includes glass transition theory, for the growth of a single bubble, Fan *et al.* (1994) showed that the bubble first grows by expansion of superheated water vapour and then, depending on the glass transition temperature, shrinks or hardens by cooling. The model clearly shows that the viscosity of the melt is the dominant factor that affects both cell growth and shrinkage.

Expansion of extrudates is commonly characterised by two main parameters: Sectional Expansion Index (SEI) measured in the radial direction and Longitudinal Expansion Index (LEI) measured in the axial direction (Alvarez-Martinez *et al.*, 1988). SEI is calculated by dividing the average cross-sectional area of the extrudate by the cross-sectional area of the die. If a circular die is used a radial expansion index (EI) can be

used to describe the expansion characteristics. EI is defined as the ratio between the diameter of the expanded extrudate and the diameter of the die, and it is equal to the square root of SEI ($SEI=EI^2$). LEI is defined as the ratio between the extrudate velocity after expansion and the velocity of extrudate inside the die. Alvarez-Martinez et al. (1988) found that SEI and LEI are inversely correlated and expressed as:

$$LEI = \left(\frac{\rho_d}{\rho_e} \right) \left(\frac{1}{SEI} \right) \left(\frac{1-MC_d}{1-MC_e} \right) \qquad (5.4)$$

ρ_d and MC_d are density and moisture content of the extrudate inside the die, ρ_e and MC_e are density and moisture content of the expanded extrudate.

SEI and LEI also depend on the rheological properties of the melt. Bouzaza et al. (1996) showed that SEI is proportional to the natural logarithm of the pressure-drop/viscosity ratio $\Delta P/(\mu_a)_d$. However, their results did not show a definite trend between LEI and melt properties. At constant die length, LEI decreased with increases in $\Delta P/(\mu_a)_d$ but, at constant die diameter, LEI increased significantly when $\Delta P/(\mu_a)_d$ increased.

5.3 Effect of Raw Materials and the Operating Conditions of the Extruder on the Rheological Properties of Melts

5.3.1 Raw Material

The microstructural properties of extrusion expanded products are created by the transformation of natural biopolymers, such as starch and proteins (Frame, 1994). When plasticized and molten at high temperatures these natural biopolymers provide a continuous matrix that can bind together other particulate matter which forms a dispersed phase. The continuous matrix retains gases released during the expansion process. The characteristics and the gas retaining ability of the continuous phase depend on its viscosity, pressure and temperature.

In food extrusion, starch is one of the most important ingredients and it plays a key role during the process, this literature review will concentrate on this component.

5.3.1.1 Starch

The Role of Starch

Starch is a key structure forming raw material in food extrusion (Guy, 1994) and it is described as a thermoplastic polymer. Depending on the thermo-mechanical treatment and the structure of the final product, extruded products containing starch may be characterised by several functional properties such as expansion, shape, mouth feel, cohesiveness, mechanical strength, solubility in water, and texture (Colonna et al, 1989). The discussion of these functional properties and how they are measured is beyond the scope of this study. However, as these properties are related to the rheological properties of the melt, it is important to understand the influence of the extrusion operating conditions and starch degradation (starch gelatinization or dextrinisation) on the melt viscosity.

Since the rheological properties of the melt are closely related to the size and shapes of molecules and/or particles forming the melt, it seems logical to assume that changes in the starch structure will greatly affect the rheological properties of the melt (Lai and Kokini, 1991).

Structure Changes in a Non-Shearing Environment

In a non-shearing environment, a minimum ratio of 14 molecules of water to one anhydrous glucose unit is required for complete starch gelatinization (Wang et al. 1990). With this theoretical calculation Wang et al. (1991) defined a minimum moisture of 63% for complete starch gelatinization without shearing at temperatures below 100°C.

Starch granules are made up of both crystalline and non-crystalline regions in alternating layers called "growth rings" (French, 1984). The crystalline layer is produced by the packing together of amylopectin that forms many small crystalline areas. The crystalline region is not able to fully swell owing to the constraints produced by the molecular chains that form the crystalline network. However, the less dense amorphous region between the dense, crystalline layer is readily penetrated by water and is more susceptible to acidic and enzymic erosion (French, 1984; Guilbot and Mercier, 1985; Bemiller and Whistler, 1996).

The mechanism of starch gelatinization with excess of water (>63%) is mainly the swelling of starch granules caused by water penetration and the dissolution of the swelled granules (Kokini *et al* 1992b). The viscosity of the starch paste increases rapidly when starch gelatinization takes off as shown in Figure 5.2. Before the temperature reaches the starting point of starch gelatinisation, starch granules are not soluble in cold water but can reversibly swell slightly. At this point the viscosity of the starch-water slurry is very low and is essentially the same value of the viscosity of water. On heating, water penetrates the amorphous region of the starch granules and the starch granules swell extensively. The swelled starch granules absorb a large amount of water and push tightly against each other in a honeycomb-like manner (Whistler and Daniel, 1985). The viscosity of the paste increases largely as a result of the flow resistance of the enlarged granules that now occupy the entire sample volume. With increasing temperature, the thermal motion of the molecules and the molecular solvation produced by the swelling lead to decreasing order and disruption of the crystalline regions. The highly swollen granules can be easily broken and disintegrated by stirring. When the number of granules being broken down exceeds the number of granules swelling, the viscosity of the paste decreases.

In limited water, gelatinization will not occur in the way described above. When temperature increases, the starch granules become progressively more mobile and eventually the crystalline region melts. The melting temperature increases when the water content decreases. As the crystalline regions melt, they become available for swelling and hydration, which results in water re-distribution within the starch granule. During swelling and hydration, strong stresses develop which strain the crystallites and contribute to the disruption of the starch granules (French, 1984). At low moisture contents, the melt would be expected to show viscoelastic behaviour (Kokini *et al* 1992b).
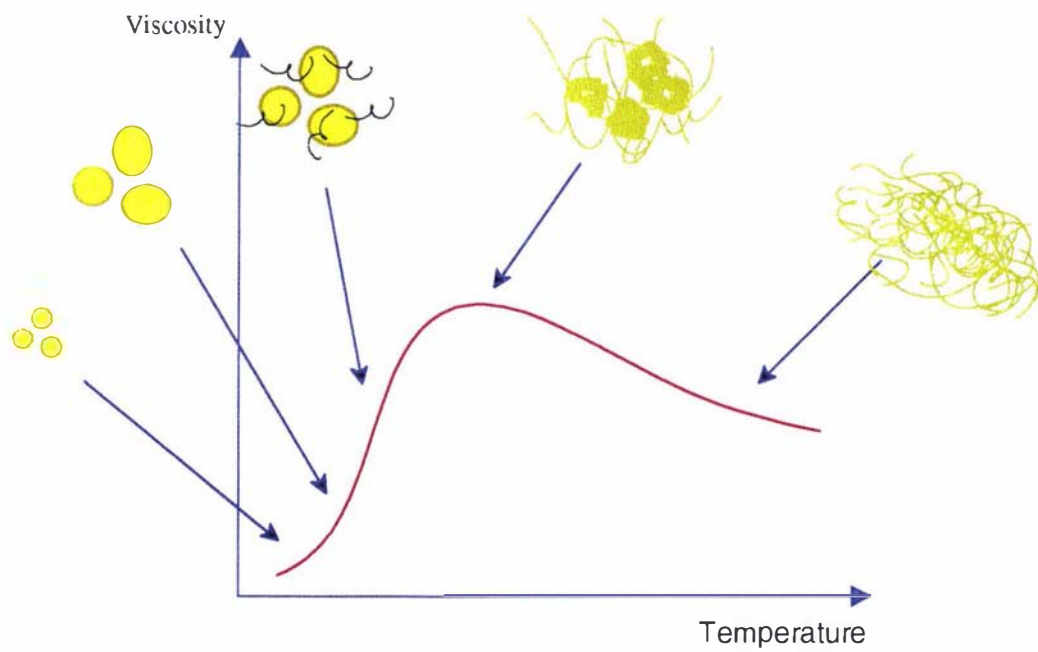
Figure 5.2 A schematic representation of starch gelatinization in the presence of excess water.

Structure Changes during Extrusion

The physico-chemical transformation of starch during extrusion is very different from that occurring in a shear-free high moisture environment. Snack food extrusion is usually conducted at moisture contents ranging from 12% to 16% wwb. This is well below the amount of water necessary for complete gelatinization as determined by Wang *et al.* (1991). In addition, the residence time of the material in the extruder (20-200s) is too brief for heat alone to transform the starch. During extrusion, shear force physically tears apart starch granules resulting in starch fragmentation allowing faster transfer of water into the interior of the starch molecules (Burros *et al.* 1987). Shear and high temperature also cause the melting of starch granules. Thus, starch fragmentation and melting are the primary mechanisms of starch degradation during extrusion (Bhattacharya and Hanna, 1987a; Chiang and Johnson, 1977; Colonna *et al.*, 1984; Davidson *et al.*, 1984a, 1984b; Diosady *et al.*, 1985; Faubion and Hoseney, 1982; Gomez and Aguilera, 1983, 1984; Kim and Hamdy, 1987; Lai and Kokini, 1990; Mercier and Feillet, 1975; Owusu-Ansah *et al.*, 1982a, 1982b, 1983; Wang *et al.*, 1991; Wen *et al.* 1990).

There are many definitions used to describe structural changes of starch molecules and granules in different environments. It appears that some of these definitions are rather confusing. For instance, starch fragmentation is also known as starch breakage. To clarify the technical definitions used in this dissertation to describe structural changes of starch, a few terms are defined. ***Starch gelatinization*** is used to describe the swelling of starch granules and the dissolution of the swelled granules caused by water penetration. ***Starch melting*** is the disruption of the crystalline regions of the starch granules at high temperatures. ***Starch fragmentation*** is the breakage of the starch granules and molecules due to both temperature and shear. ***Starch dextrinisation*** is a form of starch fragmentation and is defined as partial hydrolysis of starch molecules resulting in products that can be precipitated from aqueous solution by alcohol. ***Starch degradation*** is a general term that includes all the structural changes undergone by the starch granules and molecules including starch gelatinization, melting, fragmentation, and dextrinisation. Although gelatinization is mainly used to describe the structure changes in a shear free environment, it also occurs in extrusion. Historically, the degree of starch gelatinization was used to represent the proportion of gelatinised starch in excess water. However, in this study ***the degree of starch gelatinization*** is used to represent the

proportion of structurally modified starch over the total starch contained in the raw material. Thus, modified starch may include products of gelatinization, melting, fragmentation and dextrinisation.

Dextrinization appeared to be the predominant mechanism which usually took place in extrusion at low moisture content (Davidson et al., 1984a; Gomez and Aguilera, 1983, 1984; van Lengerich, 1990; Wen *et al.*, 1990) particularly when high screw speed was used. The structural modifications occurring during extrusion seemed to be limited to debranching of amylopectin, which in turn caused significant reduction in the overall molecular size without measurably changing the percentage of the α 1-6 bonds. Starch fragmentation was also thought to be random chain splitting in amylose (Colonna et al. 1984).

Chiang and Johnson (1977) showed that after extrusion, the extrudate contained a large amount of low molecular weight sugars that were not detected in the raw materials before extrusion. The mono- and oligosaccharides in the extruded sample were extracted with ethanol, concentrated and spotted on chromatographic papers. The sugars, which were detected by a silver nitrate reagent and identified by comparison of their mobilities with those of known samples, included fructose, glucose, melibiose, maltose, maltotriose, and maltotetraose. This suggested that the 2-1 glycosidic bonds of sucrose and raffinose and the 1-4 glucosidic bonds of malto-oligosaccharides and starch were broken by the combined action of high temperature, high pressure, and severe shearing during the extrusion process.

5.3.1.2 Amylose/Amylopectin Ratio

The starch granule is composed of two glucan polymers, amylose and amylopectin. Amylose is an essentially linear molecule of glucose units linked by α-(1-4) bonds. Typically amylose consists of 100-1000 glucose units. Amylopectin is a branched molecule with linear regions of α-(1-4) linked glucose units and α-(1-6) linked branch points (Figure 5.3). The structure of amylopectin is very complex and its linear chains consist of 12-60 glucose units (Boyer and Shannon, 1987).
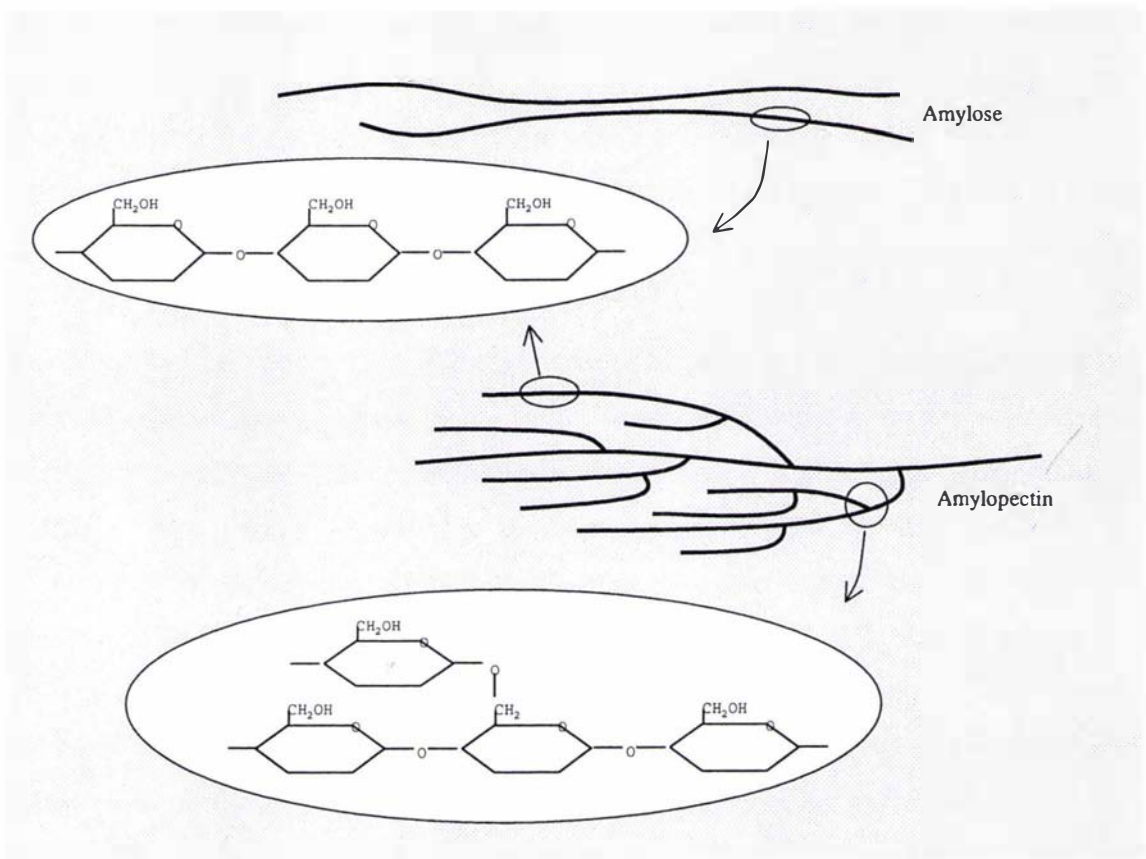
Figure 5.3 Schematic representation of amylose and amylopectin molecules.

Using Differential Scanning Calorimetry (DSC) Russell (1987) showed that amylopectin starch obtained from waxy maize has an onset temperature of 64°C at which starch gelatinization starts and a termination temperature of 90°C at which starch gelatinisation completes. Amylose starch from amylomaize showed a higher onset temperature (67°C) and a very high termination temperature (110°C). Plots of the DSC endotherms as a function of temperature showed a clear peak between the onset temperature and the termination temperature for amylopectin starch whereas amylose exhibited a broad, flat curve. These different behaviours in gelatinization show that amylose is more resistant to gelatinization than amylopectin (Lai and Kokini, 1990).

The pasting characteristics of starch are commonly measured using an amylograph (Corn Industries Research Foundation, 1964; Greenwood, 1979). A 5% to 7% of starch is added to water and heated from 50°C to 95°C at a constant heating rate of normally 1.5°C/min. The heated starch paste is held at 95°C for 30 or 60 minutes and then cooled to 55°C. During the heating, holding and cooling periods the torque of a rotating impeller, which is submerged in the paste, is recorded and the results expressed as amylograph viscosity units. Amylopectin starch had a lower onset temperature. It produced a very high viscosity at the completion of gelatinization, thereafter the viscosity decreased rapidly. The viscosity of the paste also decreased largely during the holding period. After cooling, the viscosity of the paste was low. Amylose starch showed quite different characteristics. The onset temperature was high and the viscosity increased during the entire heating period. The viscosity at 95°C was considerably lower than the peak viscosity measured for the amylopectin starch. However, after cooling the viscosity of the gel was very high.

Using flow curves Lai and Kokini (1990) showed that during extrusion a melt produced from a starch with 98% amylopectin content had a lower viscosity than that from a starch with 70% amylose. The degree of starch gelatinization for amylopectin starch was much higher (40%) than the amylose starch (8%) at low temperature (120°C) and at 30% moisture. At a higher temperature (150°C), the degree of starch gelatinization of amylose starch increased significantly from 8% to 60%.

Harper and Tribelhorn (1992) found that high amylose cornmeal produced samples with lower SEI and higher LEI, while a high amylopectin meal (waxy corn) gave products

with the opposite characteristics. Product manufactured from high amylose corn meal had a fine texture with very small cells of consistent size. The extrudates were mainly yellow in colour, with some orange to red casts and were chewy in character. Products manufactured from normal corn meal containing about 72% amylopectin had cells of irregular shape and were low in density and brittle. Products from high amylopectin waxy corn meal had physical characteristics more like the products from normal corn meal and were a light yellow colour.

Textural and rheological properties of extrudates manufactured from non-waxy corn grits (30% amylose) and waxy corn grits (1% amylose) using different extrusion conditions were studied by Bhattacharya and Hanna (1987a, 1987b). It was found that the waxy corn produced a melt with lower viscosity than the non-waxy corn. The SEI of waxy corn was higher (1.5 to 2.7) than that of non-waxy corn (1.1 to 2.1). The strength of the expanded product was measured using an Instron Universal Testing Machine and an Allo-Kramer shear press. The strength, calculated by dividing the maximum force by the cross-sectional area of the sample, was plotted as a function of moisture content and barrel temperature. These plots showed that samples made from non-waxy corn were tougher than samples made from waxy corn. Bhattacharya and Hanna (1987a,b) suggested that the waxy corn was more gelatinized and that the greater gelatinization caused the product to expand more. Microscopy showed a hollow, well-layered structure for products made from waxy corn, while the non-waxy corn products had a denser structure.

5.3.1.3 Protein and Oils

Corn grit contains 8-11% protein by weight. Protein plays a less important role in extrusion cooking of corn meal (Lai and Kokini, 1991). After extrusion the solubility of corn protein in ethanol and alkali is significantly decreased indicating that protein denaturation occurs (Wen *et al.* 1990).

Oils and emulsifiers have a very important function in the finishing of the products and are commonly added to obtain the desired product characteristics. For example, monoglycerides, diglycerides, and sodium stearoyl lactylate can be used as lubricants

and/or starch complexing agents (Cervone and Harper, 1978). Cervone and Harper (1978) found that the addition of sodium stearoyl lactylate increased the melt viscosity of pre-gelatinised corn flour during extrusion.

5.3.1.4 Corn Grits and Grains

The effect of the properties of corn grits and corn grains on extrusion has not been investigated extensively.

Corn grits are small fragments of corn endosperm. Grit size affected the colour of extrudate in single-screw extrusion (Manoharkumar *et al.,* 1978). Coarse corn grits (>1.2 mm in diameter) received inadequate heat and shear treatment in single screw extrusion (Anderson, 1969), whereas Garber *et al.* (1997) could not find such an effect in twin-screw extrusion. This is because the twin screw extruder provides a more uniform shear and a better mixing action than single-screw extruders. The residence time in twin screw extruders is also higher than that of single screw extruders.

5.3.2 Operating Conditions

Lawton *et al.* (1972) used two single screw extruders and corn starch to investigate the effects of fifteen extrusion variables on the gelatinization of starch. They included moisture content, barrel temperature, die temperature, screw geometry, screw tip geometry, screw speed, die composition (metal), die cross section area, die internal surface area, extruder barrel length, die block expansion chamber, and back pressure plate. Among these variables, moisture content, barrel and die temperatures, screw speed and screw geometry had the largest effect on starch gelatinization.

van Lengerich (1990) showed that moisture content, barrel temperature, and degree of fill are the variables that have large influence on the extrusion of starch. These variables determine how much mechanical energy (SME) can be introduced into the materials during extrusion.

The following discussions will concentrate on the effects of moisture content, barrel and die temperature, screw speed, feed rate and screw configuration on starch degradation, rheological properties of the melt, product expansion and SME.

5.3.2.1 Moisture Content

The moisture content of the melt is one of the most important processing variables. It affects not only starch gelatinization and viscosity of the melt but also the product expansion and product texture (Lawton *et al.,* 1972; Fletcher *et al.,* 1985; van Lengerich, 1990).

Using a single screw extruder and hard red winter wheat flour, Chiang and Johnson (1977) showed that at a very low temperature (65 to 80°C), the degree of starch gelatinization decreased from 40% to 35% when moisture content changed from 18% to 27%. However, extruded at 110°C the degree of starch gelatinization increased from 80% to 95% when moisture content increased from 18% to 28%. Owusu-Ansah *et al.* (1983) reported similar results when corn starch was extruded in a twin screw extruder at temperatures from 100 to 110°C. The degree of starch gelatinisation increased when moisture content increased from 11% to 23%. However, it decreased with increasing moisture content when the starch was extruded at very high temperatures >180°C.

Using Scanning Electron Microscopy, Brenner (1986) found that intact starch granules appeared in the melt of commercial maize grits (600μm) extruded with a twin-screw extruder at very high moisture content (83% dwb). With an excess of water acting as lubricant and plasticiser, even at high temperature (120°C), the starch granules are not deformed or torn apart by the process.

Melt viscosity decreases with the increasing moisture content (Cervone and Harper, 1978; Senouci and Smith, 1988a; Altomare *et al.,* 1992; Lai and Kokini, 1990; Padmanabhan and Bhattacharya, 1989, 1993a, 1993b; Vergnes *et al.,* 1993). By extruding corn starch, van Lengerich (1990) found that increasing water content from 25% to 30% decreased the power law consistency K from 5841 to 2412 $Pa \cdot s^n$ and the power law index n increased from 0.31 to 0.43. Furthermore, melts with moisture contents >35% had an almost Newtonian flow behaviour ($n \approx 1$). The addition of water

provided lubrication and resulted in a less viscous melt. This was evidenced by a decrease of the screw torque, thus providing less mechanical energy to the material and resulting in a lower SME input (van Lengerich, 1990).

Increased expansion ratios with decreasing moisture content were reported by Fletcher *et al.* (1985) and Bhattacharya and Hanna (1987b). Owusu-Ansah *et al.* (1984) found that the moisture content of the melt was inversely correlated to product expansion. Scanning electron micrographs revealed that a low moisture content in extrusion was essential to develop a porous structure with large number of air pockets.

Chinnaswamy and Hanna (1988) reported that there was a maximum expansion rate at about 14% moisture content for normal corn starch extruded in a single screw extruder at 140°C. The expansion rate increased when the moisture content of the melt decreased from 30% to 14% dwb, and then decreased sharply when moisture was lower than 14% dwb. At low moisture content, the material became very viscous and high pressure in the extruder die was generated. This resulted in longer residence times and further starch degradation and dextrinization occurred. With a large degree of dextrinisation and hence a reduction in average molecule size, the glass transition temperature of the melt is much lower (Fan *et al.*, 1994). Melts with lower glass transition temperatures remain soft after expansion, therefore, bubbles produced in the melt can shrink by the negative pressure through vapour condensation, thus resulting in less expanded products.

5.3.2.2 Barrel and Die Temperatures

The temperature of the melt is controlled by the barrel and die temperatures and has a large effect on the rheological properties of the melt and its expansion rate.

Owusu-Ansah *et al.* (1983) showed that the interaction of barrel temperature and moisture contributed largely to increasing the degree of starch gelatinization during extrusion. Barrel temperature alone was the next important contributor, followed by moisture content and then screw speed. Lawton *et al.* (1972) also found that barrel temperature and moisture content were the variables that exert the greatest effect on starch gelatinization. High barrel

temperature and low moisture content resulted in a large degree of starch gelatinization. These results are consistent with studies on wheat flour, where the interaction between barrel temperature and moisture content was shown to be the most important factor affecting starch transformation during single screw extrusion (Chiang and Johnson, 1977).

Increase of die temperature also contributed to a higher degree of starch gelatinization of the melt (Lawton *et al.,* 1972) resulting in products with a higher expansion (Fletcher *et al.,* 1985).

The temperature of the melt increased and viscosity decreased when barrel temperature increased (Cervone and Harper, 1978); Padmanabhan and Bhattacharya, 1989; van Lengerich, 1990). The power law consistency K decreased nearly 50% and power law index n increased slightly when barrel temperature increased from 160°C to 180°C (Padmanabhan and Bhattacharya, 1993a; 1993b). This is consistent with the results of Lai and Kokini (1990), Senouci and Smith (1988a) and Vergnes *et al.* (1993).

Using a twin screw extruder and maize grits, Fletcher *et al.* (1985) showed that the expansion ratio increased when the extruder barrel temperature increased from 100°C to 140°C. By studying one variable at a time Chinnaswamy and Hanna (1988) found that the expansion ratio was highly dependent on barrel temperature, moisture content, extruder die configuration, screw speed and feed rate. An expansion ratio as high as 16.1:1 was obtained when corn starch was extruded at a barrel temperature of 140°C, 14% moisture content, 150 rpm screw speed, 60g/min feed rate, and a die with a L/D ratio of 3.1 in a single screw laboratory extruder (C. W. Brabender Model 2902). For temperatures higher than 140°C the expansion rate decreased.

The decrease in viscosity and in expansion rate at very high barrel temperatures was also observed by Chiang and Johnson (1977), Chinnaswamy and Hanna (1987), Colonna *et al.* (1984), and Davidson *et al.* (1984a, b). This decrease in viscosity and expansion was thought to be the result of starch degradation notably starch fragmentation and dextrinisation. As discussed in the previous section, starch fragmentation and dextrinisation reduce average molecular size therefore lower the glass transition temperature resulting in less expanded products.

5.3.2.3 Effect of Screw Speed, Feed Rate and Degree of Fill

Although screw speed appears to have little influence on the expansion ratio (Bhattacharya and Hanna, 1987a,b), it is an important variable affecting gelatinization of corn starch (Lawton *et al.*, 1972) and melt viscosity. Increasing screw speed in a single screw extruder decreased starch gelatinization due to a decrease in residence time (Chiang and Johnson, 1977). Using maize grits, potato powder and a twin screw extruder Senouci and Smith (1988a) found that at constant barrel temperature and constant feed rate, viscosity of the extrudates decreased and SME increased with increasing screw speed. The result was independent of the barrel temperature used, although, these rheological measurements could be affected by the thermo-mechanical history and could not be reliable.

Fletcher *et al.* (1985) showed that the expansion ratio increased when the extruder feed rate and screw speed increased from 30 to 60kg/h and 150 to 430 rpm respectively.

Degree of fill is an extrusion parameter that gives an indication of the proportion of the extruder barrel that is being used for starch transformation at a given screw speed and feed rate. It is usually defined as the ratio between the feed rate and the screw speed (van Lengerich, 1990). Increases in screw speed or decreases in feed rate result in a lower degree of fill. It is related to the residence time distribution (RTD) and Specific Mechanical Energy (SME) and the viscosity of the melt.

van Lengerich (1990) observed that a lower degree of fill always corresponded to a higher SME, regardless of whether it was achieved through a decrease in feed rate or an increase in screw speed. Starch melts with smaller average molecular weights were produced at lower degree of fill resulting in melts with decreased viscosity. These results agreed with those of Senouci and Smith (1988a)'s who found that increases in SME caused more starch degradation during the extrusion process resulting in melts with lower viscosity. van Lengerich (1990) demonstrated that a high SME introduced in a relative short residence time affected molecular breakdown more than a low SME introduced over an extended time period.

Chinnaswamy and Hanna (1988) found that both screw speed and feed rate affect product expansion rate and there exists an optimum screw speed or feed rate for expansion. Normal corn starch was extruded using a C. W. Brabender Laboratory model single screw extruder. The optimum expansion rate was related to a maximum value of $\Delta P/(\mu_a)_d$. The change in screw speed or feed rate resulted in a change in the melt viscosity and die pressure. The lower expansion rate at either side of the optimum was attributed to lower values of $\Delta P/(\mu_a)_d$, which may be a direct result of lower levels of starch gelatinization at high feed rates, or extensive molecular degradation of starch at lower fill levels. Again, extensive starch degradation reduced the starch average molecule size lowering the glass transition temperature and resulting in a less expanded product.

5.3.2.4 Screw Configuration and Die-Block Design

Screw geometry is an important variable in the extrusion operation. Using two single screw extruders (C. W. Brabender Laboratory model), Lawton *et al.* (1972) found that increasing the compression ratio of the screw resulted in an increase in the degree of starch gelatinization.

Sokhey *et al.* (1994) showed the effect of mixing elements on the product expansion. Corn starch was extruded in a single screw extruder at 140°C and a constant screw speed of 140rpm with three different screw configurations: no mixing, one mixing, and two mixing elements. The screw configuration without the mixing element produced a slightly higher expansion rate (4.8) than the other two (4.0 for one mixing element and 3.9 for two mixing elements).

Kirby *et al.* (1988) used a twin screw extruder and four sets of screw configurations to study the effect of screw conveying efficiency on extrusion cooking of maize grits. The screw without any mixing paddles was defined as a high conveying efficiency configuration while the others with one to four sets of mixing paddles as low conveying efficiencies. At a moisture content of 20%, the low conveying efficiency screws produced products with very low bulk density ≤ 0.25g/cm^3 at all temperatures from 90 to 150°C. Conversely, the high conveying efficiency screw without the mixing paddles could not produce well expanded product if the temperature was lower than 150°C. The

lowest screw torque was produced with the screw with the highest conveying efficiency. It increased when more mixing paddles were used.

Chinnaswamy and Hanna (1987, 1988) using a laboratory extruder (Brabender) studied the effect of die length and diameter on extrudate expansion using corn starch containing 25% amylose. They used dies with different length to diameter ratios (L/D). The expansion ratio of the product increased from 4.5 to 13.0 as L/D increased from 2.5 to 3.4 and then decreased gradually to 8.5 as L/D was further increased to 10.3. The initial increase in expansion ratio was thought to be related to the degree of starch gelatinization. Higher L/D ratio resulted in higher die pressure and longer residence times with an increased degree of starch gelatinization. A further increase of L/D resulted in a much higher die pressure with even longer residence time, which caused appreciable starch dextrinisation. Thus, the glass transition temperature of the melt decreased resulting in shrinkage of the product after expansion.

Since different absolute values of L and D can be used to achieve the same L/D ratio, it was suggested that the die pressure instead of the L/D ratio may be a better indicator for the prediction of product expansion (Chinnaswamy and Hanna, 1987).

5.4 On Line Measurements of Rheological Properties

The puffing and expansion phenomenon is mainly dependent on the rheological properties of the extrudate after plasticization and before exiting the die opening. The rheological properties are also closely related to the extrusion process, the properties of the raw materials, and the properties of the final product. Therefore, the measurement of rheological properties is very important to control the extrusion process. However, the measurement of viscosity is only valid when the extrudate is still in a molten state under conditions of high temperature and pressure and in a high shear environment. Furthermore, the rheological properties of the melts are very sensitive to the process thermo-mechanical history making on-line determination the only possible alternative to measure these properties.

The high pressures and shear existing in the extruder are difficult, if not impossible, to achieve in conventional rotational viscometers, precluding their use in the determination of the rheological properties of the melt. Therefore, commercial capillary viscometers and rotational viscometers are not suitable for measuring viscosity of the melt during extrusion (Altomare *et al.*, 1992) and the rheological properties of the melt must be measured on-line (Bhattacharya and Padmanabhan 1992, 1994).

Specially developed capillary and slit die viscometers were used to measure the rheological properties of melts on-line. In both viscometers, the pressure profiles along the die and the flow rate of the material through the die were measured. The use of the pressure profile enables the calculation of the shear stress whereas the flow rate enables the calculation of the apparent shear rate. The capillary viscometer has a small circular channel while the slit viscometer uses a rectangular channel to create the viscometric flow for calculating shear rate and shear stress.

5.4.1 Capillary Viscometer

A capillary viscometer is a device that can create a very well defined and simple shear flow. The apparent viscosity of the fluid can be calculated by using equation (5.2) once the wall shear stress and the shear rate are determined at varying conditions by the following equations (Steffe, 1992):

$$\tau_w = \frac{R}{2} \frac{\partial P}{\partial x} \cong \frac{R}{2} \frac{\Delta P}{L} \tag{5.5}$$

$$\dot{\gamma}_{app} = \frac{4Q}{\pi \cdot R^3} \tag{5.6}$$

$$\dot{\gamma} = \left(\frac{3n+1}{4n} \right) \frac{4Q}{\pi \cdot R^3} \tag{5.7}$$

τ_w is the shear stress at the capillary wall, $\dot{\gamma}_{app}$ is the apparent shear rate, $\dot{\gamma}$ is the corrected shear rate using the power law index, R is the radius of the capillary, P is the pressure inside the capillary tube, ΔP is the pressure difference between the entrance and the exit of the capillary, L is the length of the capillary, Q is the flow rate in the capillary, and n is the power law index.

It is important to note that the shear stress can be calculated using Equation (5.5) only when a linear pressure profile along the capillary is obtained. By using capillary dies of different length to diameter (L/D) ratios and applying various pressure difference between the entrance and exit, a wide range of shear rates (10^{-1} - 10^{4} s^{-1}) and shear stresses (10^{2} - 10^{7} Pa) can be measured.

Although capillary viscometers have been used extensively to determine the shear properties of viscous fluids, their success as on-line systems in food extrusion still has to be demonstrated. Two main designs of capillary viscometer are discussed in this section. One consists of a short capillary die with the pressure being measured before the capillary entrance. The other design uses a long capillary with pressure transducers mounted along the capillary tube.

5.4.1.1 Capillary Die

Jao *et al.* (1978) used four sets of capillary dies to investigate the effects of moisture content, temperature, and shear rate on the rheological properties of defatted soy dough in a single screw extruder. Only one pressure transducer was used to monitor the pressure before the capillary viscometer and this pressure was considered as the sum of the entrance and capillary pressure drops. Viscosity was calculated by knowing the flow rate and the pressure difference between the measured pressure and the atmospheric pressure. The screw speed was adjusted to vary the shear rate in the capillary die. Jasberg *et al.* (1981) used the same approach and capillary dies with different length/diameter ratios to measure viscosity of soy dough and polyethylene melt on-line. It was found that data obtained for polyethylene melt were within the expected range. However, the rheological properties of the soy extrudate were affected by the extruder operating conditions.

Capillary dies were also used to measure the viscosity of extruded blends of corn gluten meal and soy protein concentrate (Bhattacharya and Hanna, 1986) and of pregelatinized corn flour (Cervone and Harper, 1978) on a single screw extruder. The rheological properties of the melt were found to follow the power law model described in section *5.3.1.*

5.4.1.2 Capillary Viscometer with Pressure Transducers Mounted along the Capillary Tube

The pressure drop in extruder dies was analyzed by Padmanabhan and Bhattacharya (1989) using a capillary die fitted with three pressure transducers along its length. The die was actually acting as a capillary viscometer and the apparent viscosity was calculated from the pressure distribution and the flow rate in the die. It was concluded that entrance pressure drop could be as high as 37% of the total pressure drop and varied proportionally to the length-to-die diameter ratio.

Wang *et al.* (1990, 1993) used a capillary viscometer before the extruder die exit to measure the extrudate viscosity on-line. Moore *et al.* (1990) and Bouzaza *et al.* (1996) also used the same design. The shape of the capillary was changed to have a flat section for fitting two pressure transducers. In order to prevent interference with the extrusion process, a short capillary was used. This design forced the position of the first pressure transducer to a location which was too close to the capillary entrance and entrance effects for non-Newtonian fluids could not be avoided. To get a proper calculation of viscosity without entrance effects, it is essential that a linear pressure gradient exists along the capillary die. Most of the past work, however, was carried out using two pressure transducers which makes it impossible to discern whether the pressure distribution is linear. A minimum of three pressure transducers should be used to get suitable data. Furthermore, as the cross section of the capillary was altered to fit the pressure transducers, theoretically, the equation derived for a capillary with a circular shape could be no longer valid.

Lai and Kokini (1990) also used a capillary viscometer with four pressure transducers mounted on the wall of the capillary to examine the rheological properties of corn starches during single screw extrusion.

5.4.1.3 Disadvantages of the Capillary Viscometer

There are some disadvantages in using capillary viscometers. It is necessary to use at least two dies with different L/D ratio in order to correct for entrance effects. This is

known as the Bagley correction (Bagley, 1957). Changing the capillary die is a time consuming procedure and impractical if on-line measurements are required. Cases, as in this project, with a limited availability of raw material could preclude the use of a capillary viscometer with frequent stops and wastage of material.

The disadvantages in the use of capillary viscometer discussed in this section clearly show that this type of viscometer was not appropriate for this project.

5.4.2 Slit-Die Viscometer (SDV)

The working principle of slit viscometry is similar to that of the capillary one. It uses a thin rectangular shape, rather than a capillary to produce the viscometric flow. The advantage of the slit viscometer is the measurement of pressure along the slit because it is possible to flush mount the pressure transducers on the wide flat sides of the slit geometry. Furthermore, if pressure transducers are located far from the slit entrance, no entrance correction is needed. Because of these advantages, extruder-fed slit die viscometers have been used extensively in food extrusion to obtain rheological properties of melts (Altomare *et al.,* 1992; Bhattacharya and Padmanabhan, 1994; Lai and Kokini, 1990, 1992; Padmanabhan and Bhattacharya, 1991, 1993a, 1993b; Seethamraju *et al.,* 1994; Senouci and Smith, 1988a, 1988b; van Lengerich, 1990; Vergnes *et al.,* 1993).

Lai and Kokini(1992) studied the effect of viscous heating in slit flow and found that the viscous heating was not a major source of errors in rheological measurements using this type of viscometer. Frictional heating in the die can be further reduced if the material is less viscous, or if the temperature of the viscometer is controlled.

5.4.3 Interference between On-Line Viscosity Measurements and the Operating Conditions of the Extruder

In order to measure the viscosity of the melt on-line, the viscometer is mounted as a die block at the exit end of the extruder. Different flow rates and pressures are applied through the capillary or slit channel to obtain different shear rates. By measuring the

volumetric flow rate and pressure gradient along the channel, shear stress and shear rate can be calculated.

In the classical design of an on-line slit viscometer, the output of the extruder goes directly into the viscometer. In order to obtain different shear rates, the throughput of the extruder is modified. Most of the researches reviewed in this chapter utilised this approach (Altomare *et al.,* 1992; Lai and Kokini, 1990, 1992; Padmanabhan and Bhattacharya, 1991; Senouci and Smith, 1988a, 1988b).

Depending on the type of extruder used, two methods were used to vary the extruder throughput. (1) Many single screw extruders use flood feed with an excess of raw material available at the feed of the extruder. In this condition, the throughput is dependent on the screw speed. In order to vary the throughput and the shear rate in the viscometer, various screw speeds can be used. (2) Starved feed mode is commonly used with twin screw extruders and many single screw extruders designed for this purpose. In order to vary the shear rate in the viscometer, the throughput of the extruder is adjusted by varying the feed rate.

In both cases, varying screw speed and feed rate alter the conditions under which the extruder is operating. Consequently, the extruded material is subjected to different thermo-mechanical treatments at each set of operating conditions. Thus the flow curves obtained are affected by uncontrolled variation in the operating conditions of the extruder and may not represent the flow behaviour of a single product. Thus the resulting flow curves using the classical on-line approach should be treated with caution.

Experiments using both polymeric and food materials clearly showed that extreme care has to be taken with food materials because their sensitivity to thermo-mechanical treatment. Using an on-line SDV attached to a twin screw extruder, Senouci and Smith (1988a) measured the viscosity of low density polyethylene (LDPE), corn grits, and potato powder. Rheological measurements on LDPE were not affected by the processing history while the data for corn grits and potato powder were strongly affected by the extrusion processing history. Padmanabhan and Bhattacharya (1991) used a Slit-Die-Viscometer attached to a Brabender single screw extruder to investigate

the flow behaviour of corn meal and LDPE at various extrusion conditions. Various screw speeds ranging from 60 to 240 rpm were used to vary the extruder throughput and therefore to obtain various shear rates at the SDV. Their results showed that this procedure affected the rheological measurements during the extrusion of corn meal resulting in small or negative values of power law index n. The severe shear thinning behaviour observed ($n \leq 0$) was rather unusual and was explained by Padmanabhan and Bhattacharya (1991) as a result of molecular degradation, the pressure dependence of viscosity, viscous dissipation, slip flow and yield stress of the melt. The reason for their results, however, lies in the fact that changes in the screw speed affected the thermo-mechanical treatments of the product. The flow curves plotted from this data did not represent the true rheological properties of the melts. Thus, their conclusions were invalid as they were developed from data of different products.

Altomare *et al.* (1992) used a twin-screw extruder and rice flour to examine the suitability of using a long slit die as a rheometer in food extrusion. The slit die viscometer had gaps of 0.1, 0.075, and 0.05in and corresponding height to width ratios of 7.9, 10.5, and 15.8. The feed rate of the extruder was varied to obtain different shear rates. The slit with a large width to height ratio (>10) produced a linear pressure gradient distribution along the slit. The large gap (0.10in) produced a higher power index n (0.67) than that of the smallest gap (0.05in) for which the power law index n was 0.49. The authors concluded that the SDV was a useful but imperfect tool for determining the melt viscosity on-line, and it was virtually impossible to ensure that all materials experience identical time-temperature-shear histories during the measurements. However, from their experimental approach it is clear that the variation in feed rate used to obtain different shear rates varied the thermo-mechanical history of the melt at each run. The flow curves therefore represented products subjected to different processing conditions. Furthermore, the changes in the slit height resulted in dies with different flow resistance. Thus, for a similar throughput and screw speed, the degrees of fill in the extruder were different.

Lai and Kokini (1990) also used on-line viscometers (slit and capillary geometry) attached to a single screw extruder (Brabender) to measure the rheological properties of high amylose and high amylopectin corn starch during extrusion under various conditions. The extruder was flood fed and the screw speed was varied from 20 to 210

rpm to obtain different shear rates. The differences in viscosity between the slit and the capillary die were large at high shear rates and low moisture levels. Some flow curves yielded a very low value of the power index n. These results clearly showed that interference between the rheological measurements and the operating conditions of the extruder could not be avoided.

This review shows that the classical method to measure melt viscosity using an on-line viscometer must be used with great caution as it interferes with the operation of the extruder. Several designs to reduce or eliminate the interference between the SDV measurements and the extruder operating conditions have been investigated recently.

5.4.4 Improvements on On-Line Viscosity Measurement.

The idea of using a side-stream valve to vary the flow rate at the SDV was introduced by Padmanabhan and Bhattacharya (1993a) and used in the work of Padmanabhan and Bhattacharya (1993b), Bhattacharya and Padmanabhan (1994), Seethamraju and Bhattacharya (1994) and Seethamraju et al. (1994). In this design a side-stream valve was placed at the last section of a single screw extruder which was flood fed at fixed screw speeds. Adjusting the opening of the side-stream valve, the flow rate through the slit die was controlled. The flow curves obtained using this technique were significantly different (n=0.3 to 0.44) from those obtained by varying the screw speed and the throughput ($n \leqslant 0$). Padmanabhan and Bhattacharya (1993a) claimed that this design reduced the interference between the SDV and the extruder. However, this approach still interfered with the operation of the extruder. The melt pressure before the SDV was measured using a pressure transducer but there was no indication whether the pressure was maintained constant to ensure the operation of the extruder remained unchanged. The side stream valve was positioned at the last section of the extruder barrel and part of the extrudate was diverted as waste. As the extruder was flood fed and the screw speed was constant, the slit of the SDV plus the opening of the side stream valve defined the total restriction to the extrudate flow and determined the pressure built-up in the extruder and the pressure drop in the SDV. The slit was not adjustable, therefore, when the opening of the side stream valve was varied, the change in the flow area was also varied resulting in pressure changes in both the extruder and the SDV. Thus, the

problem of interference between the SDV and the extruder operating conditions still remained unresolved.

Using a twin-screw extruder, van Lengerich (1990) suggested that if the specific throughput, defined as the ratio between the extruder throughput and the screw speed, is kept constant, the degree of fill in the extruder also remains constant and therefore the product undergoes the same thermo-mechanical history. Hence it is possible to vary the throughput to obtain different flow rates at the SDV without modifying the thermo-mechanical treatment of the product. The method, however, could lead to a very long experimental procedure. When the feed and screw speed are changed, it takes time for the extruder to reach a new equilibrium status before measurements can be taken. Furthermore, the method can not be used with single screw extruders.

Springer *et al.* (1975) presented a novel designed SDV to measure on-line rheological properties of polystyrene. The design was based on a dual slit geometry with a double valve to eliminate the effect of SDV on the extruder operation. Based on this principle, a new SDV for food extrusion called Rheopac was described by Vergnes et al (1993). Through a balanced diversion of the feed rate between the two channels the flow rate and shear rate in one of the channels could be modified without changing the flow conditions in the extruder. Each channel was provided, at its entrance, with a piston valve, which could be moved up and down in order to partially obstruct the flow section. Because the die pressure was not monitored, the relationship between the two valve openings had to be calculated before the experiment to maintain the same operating conditions. The relationship between the two valve openings was dependent on the power law index n. Vergnes et al (1993) showed that if a proper ratio between the valve and slit lengths is chosen and also the power law index n of the melt is greater than 0.4, the valve opening is weakly dependent on the power law index n. However, the power law index n for maize grits melt under different extrusion conditions was reported to be in the range 0.19 to 1.10 (Vergnes and Villemaire, 1987; Senouci and Smith, 1988a; Bhattacharya and Padmanabhan, 1994). This makes the above assumption inaccurate.

The evidence presented in this section clearly shows that a new viscometer, with a design suitable for single and twin screw extruders and capable of measuring the

rheological properties of the melt independently of the extruder operating conditions, was needed in the area of food extrusion and rheology.

5.5 Summary

The viscosity of melt plays an important role in the quality of expanded products. Expansion rate or bulk density is directly influenced by the melt viscosity and the die pressure. The relationships between rheological properties of the melt, extrusion operation conditions and raw materials are important in understanding the food extrusion process and in product quality control. The viscosity of the melt and the expansion rate are controlled by the extrusion operating conditions and the properties of raw materials. The most important extruder operating parameters are moisture content, temperature, and degree of fill. Usually a higher moisture content results in a lower viscosity of the melt. Increases in temperature or shear in the extruder increase the degree of starch gelatinization and starch dextrinisation, therefore, resulting in variations in the viscosity of the melt.

Rheological properties can be measured on-line with a SDV. On-line measurements carried out by altering the throughput of the extruder to obtain different shear rates in the viscometer can interfere with the extruder operation. Therefore, a new design for on-line measurements is required in order to assure that correct viscosity measurement is taken.

In New Zealand, varieties of corn hybrids are used to produce corn grits or corn meal for extrusion cooking. Study of the effect of different hybrids on the rheological properties of melts has not been researched, and that information could be very valuable to both industry and basic research.

<center>**Chapter 6**</center>

<center># Extrusion Experiments: Materials and Methods</center>

A new Slit-Die-Viscometer was developed as part of this work and attached to a twin screw extruder to measure melt viscosity on-line. It was used to investigate the effects of different raw materials and extrusion operating conditions on the properties of the melt. Starches of different characteristics, grits of different properties and grits from different corn hybrids were used. Details of the new SDV, equipment, raw materials, and methods are described in this chapter. The common experimental approaches and procedures for all experiments are also discussed. Specific extruder operating conditions and experimental set up for each of the tests are given along with results in Chapter 7.

6.1 Materials

6.1.1 Normal Corn Grits

Corn grits from a single batch were obtained from Seedbank Ltd NZ. The grits were produced from a Pioneer brand corn hybrid P3515. The grits had a moisture content of 14.1% w/w and a oil content <0.9%. The true density of the grits was 1430 kg/m^3. These grits are referred as *normal corn grits* or *normal grits* in Chapter 7. They were used as the raw material for experiments aimed to investigate the effect of extruder operating conditions and the temperature of the SDV on the rheological properties of the melt. The particle size distribution of the normal grits is shown in Table 6.1.

Table 6.1 Particle size distribution of normal corn grits.

<0.2 mm	<1 mm	<1.18 mm	<1.4 mm	<1.7 mm	>1.7 mm
Nil	20-35%	40-45%	20-35%	0-5%	Nil

6.1.2 Starches of Different Amylose/Amylopectin Ratio

Two starches were purchased from National Starch and Chemical Company, NZ. One was *Colflo67*, a waxy corn starch, which contains amylopectin (>99%) and amylose (<1%). Another was *Crispfilm*, which contained about 55% amylose and 45% amylopectin. Both starches had approximately 11% moisture content.

6.1.3 Grits of Different Sizes

Corn grits from a single hybrid (Pioneer brand P3514) but with three size distributions (Seedbank Ltd. NZ) were used to investigate the effect of particle size on extrusion. Figure 6.1 shows the particle size distribution for each grit sample. For convenience, these grits will be referred as *fine*, *medium* and *coarse* grits as indicated in Figure 6.1. They had different moisture contents and true densities as shown in Table 6.2.
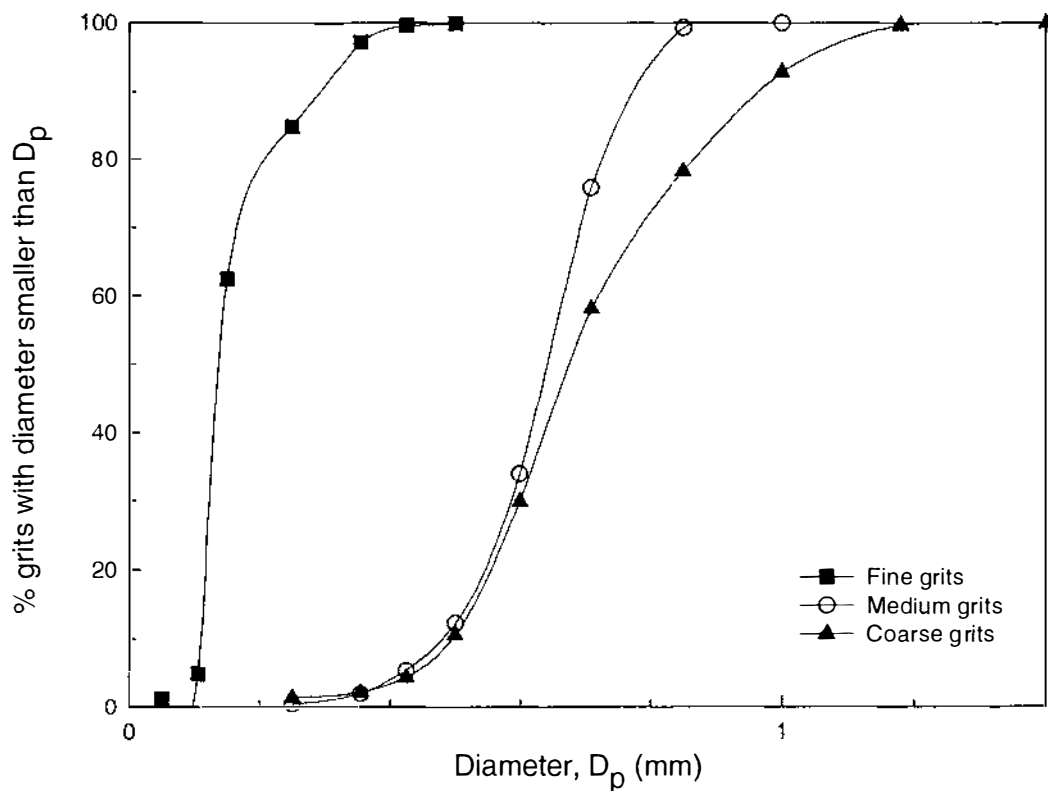
Figure 6.1 Particle size distribution of grits used in extrusion trials to investigate the effect of grit size on melt viscosity.

Table 6.2. Moisture content and true density of different size grits.

	fine	medium	coarse
Density, kg/m^3	1430.2	1378.5	1381.8
MC, %	14.3	16.1	15.2

6.1.4 Grits from Different Hybrids of the 92-93 Season

Grits of 11 different hybrids were produced for extrusion to investigate the effect of hybrid type on extrusion performance. Eleven hybrids were selected from the 38 hybrids listed in Table 3.1 (Chapter 3) for the 92-93 season. They were selected to have four hard samples (PF1×AS3-94, PF2×NZ56, PF1×BS22-39, PF1×NZS1-141), three hybrids of medium hardness (D1275×M378-80, N190×NZ2, WGI207×M378-80) and four soft hybrids (A82-8×MBS847, P3751, P3394, P3585). Whole grains of each sample were ground then sieved to produce grits with size between 0.5mm to 1.7mm. The amount of grains available for each hybrid was limited and about 5 kg grits were produced for each hybrid studied. The oil content of this material was approximately 2.2 to 3.8% and the protein content was in the range from 6.5 to 10.8%.

6.1.5 Grits from Different Hybrids of the 94-95 Season

Grits were produced from grains of the 12 hybrids listed in Table 3.2 (Chapter 3) in the 94-95 season. To this set, five hybrids produced by the Institute for Crop & Food Research were added. The grain was degermed, then milled to grits with sizes ranging between 0.5mm to 1.7mm. The amount of grits available for extrusion was about 5 kg for each hybrid. Moisture, oil and protein contents of each sample were measured (Table 6.3).

Table 6.3 Moisture, oil and protein contents of grits obtained from the 94-95 season.

Test #	Hybrid Name	Hardness	Moisture content, %	Oil content, %	Protein content, %
9409#01	P3751	medium	12.1	1.0	7.3
9409#02	FURIO	medium	11.7	1.0	7.9
9409#03	NZS3-523-1-1-1 × T1	medium	11.3	1.5	9.9
9409#04	NZ84 × Mo17Ht	soft	11.6	1.0	8.1
9409#05	Hmv-124-2 × T1	medium	11.7	1.4	8.8
9409#06	A665 × H99	medium	10.6	1.0	8.3
9409#07	NZ45 × T1	medium	10.3	1.1	9.1
9409#08	M396-14-1-1-1-1-1 × A665	medium	9.9	1.1	8.5
9409#09	157-R-3-9-1 ×A82-8	soft	1.8	1.0	6.8
9409#10	N192 × Mo17Ht	soft	11.7	1.2	7.8
9409#11	NZ84 × A82-8	soft	11.1	1.2	6.6
9409#12	NZ84 × E1386	hard	10.3	0.8	8.1
CF01	A665 × NZ77	medium	12.7	2.1	9.3
CF02	D1260 × NZ84	hard	12.8	1.0	7.3
CF03	A82-8 × NZ43	soft	12.9	1.2	7.1
CFHD	E1386 × NZ84B	hard	12.8	1.1	9.5
CFSFT	Mo17Ht ×NZ84	soft	12.7	1.2	6.7

6.2 Methods

6.2.1 Extruder

A twin screw co-rotating extruder Clextral BC21 was used in this study. The extruder barrel has four sections. Each section is equipped with an independent temperature control unit (Figure 6.2). Figure 6.3 illustrates the screw configuration used in this research. The configuration combined forward screw elements with different pitches, mixing paddles and a pair of reverse elements. Dry raw materials were fed into the extruder by a volumetric feeder and water was directly injected into the mixing section of the extruder with a dosing pump. The rotational speed of the screw, feed rate and water rate were controlled and screw torque and thrust pressure measured using the developed data acquisition system (described in section 6.2.3).
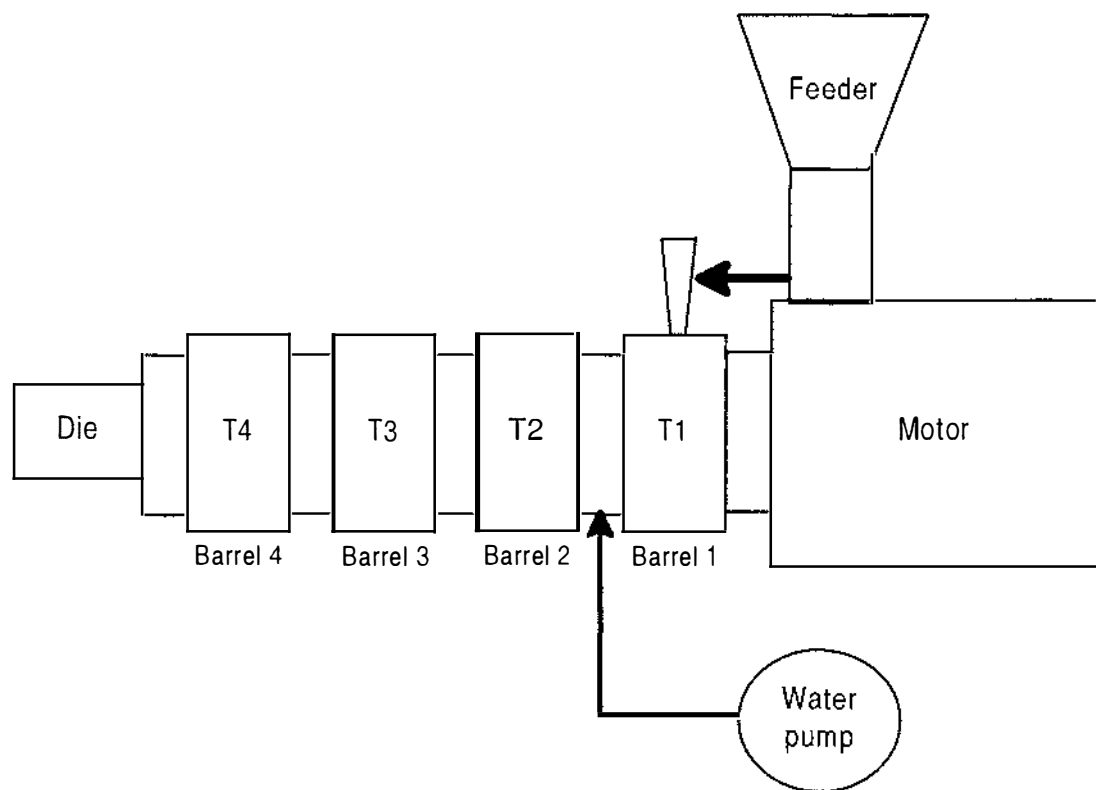
Figure 6.2 Schematic of the twin screw co-rotating extruder Clextral BC21.

Flow direction

←

|◄——————————————————— 400 mm ———————————————————►|

D   RSE   C   C   KD   B   KD   B   A   A

A: Forward screw element, 13mm pitch, 50mm length;
B: Forward screw element, 10mm pitch, 50mm length;
C: Forward screw element, 7mm pitch, 50mm length;
D: Forward screw element, 7mm pitch, 25mm length;
RSE: Reverse screw element, 7mm pitch, 25mm length;
KD: Bi-lobal kneading disks, forward conveying;
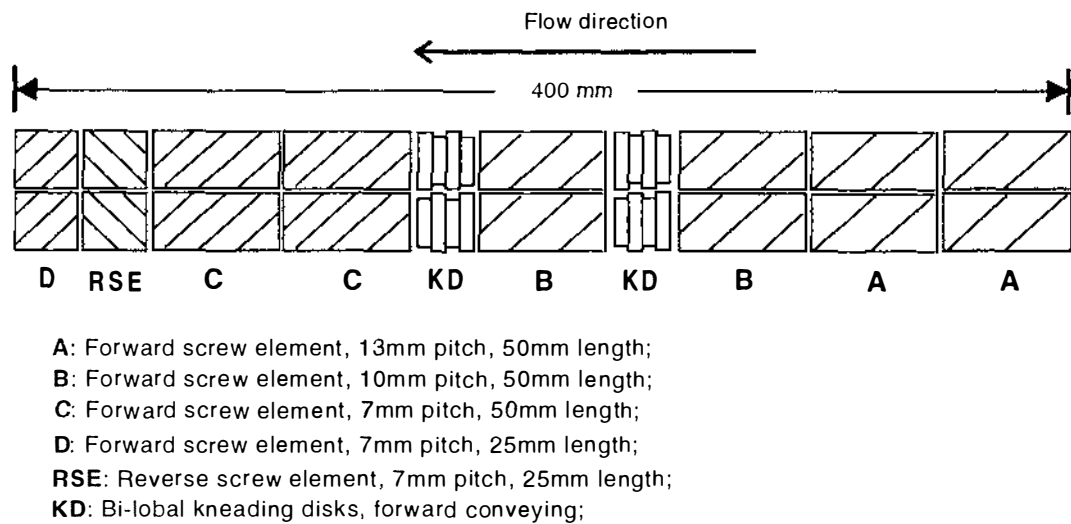
Figure 6.3 Schematic of the screw configuration used.

6.2.2 The New Slit-Die-Viscometer (SDV)

The new SDV used in this project is shown in Figure 6.4. It consists of an adapter and a slit die block which is used to measure the shear rate and shear stress. The slit height is adjustable. The slit used in this research has dimensions of 2×30×250mm. Its width to height ratio was 15:1. This was considered large enough to meet the necessary conditions to generate a well defined slit flow (Whorlow, 1992). Five combined pressure and temperature transducers (Dynisco TPT463) were flush mounted on one side of the slit. The pressure transducers were mounted at positions 58mm away from the entrance region to ensure that the measurements were taken in the region where the slit flow was fully developed. The tip diameter of the pressure transducer is about 8 mm, much smaller than the slit width, thus the pressure measurements were free from edge effects. Heating elements controlled by a computer were employed to maintain the temperature in the viscometer. Before the slit viscometer, an adapter was fitted to allow the diversion of flow. By carefully adjusting the openings of valves A and B indicated in Figure 6.4 the flow rate in the SDV was varied to achieve different shear rates, whereas at the same time, the die pressure and SME were maintained constant to ensure that the extruder operating conditions remained unchanged.

The die pressure was monitored by a pressure transducer (Dynisco PT415). The design is relatively simple and has the advantage that any typical SDV can be easily converted

without changing the original design. Compared with the design of Vergnes *et al.* (1993), this design has the advantage that the position of the valves A and B is independent of the power law index *n*.

The mass flow rate was determined by weighing the amount of sample collected in a given time. The density of the extrudate was calculated on line and the mass flow rate converted to volumetric flow rate Q using the following formulas.

$$Extrudate\ Density = \frac{Feed\ Rate_{water} + Feed\ Rate_{corn\ grit}}{Feed\ Rate_{water}/Density_{water} + Feed\ Rate_{corn\ grit}/Density_{corn\ grit}} \qquad (6.1)$$

$$Q = \frac{Feed\ Rate_{water} + Feed\ Rate_{corn\ grit}}{Extrudate\ Density} \qquad (6.2)$$
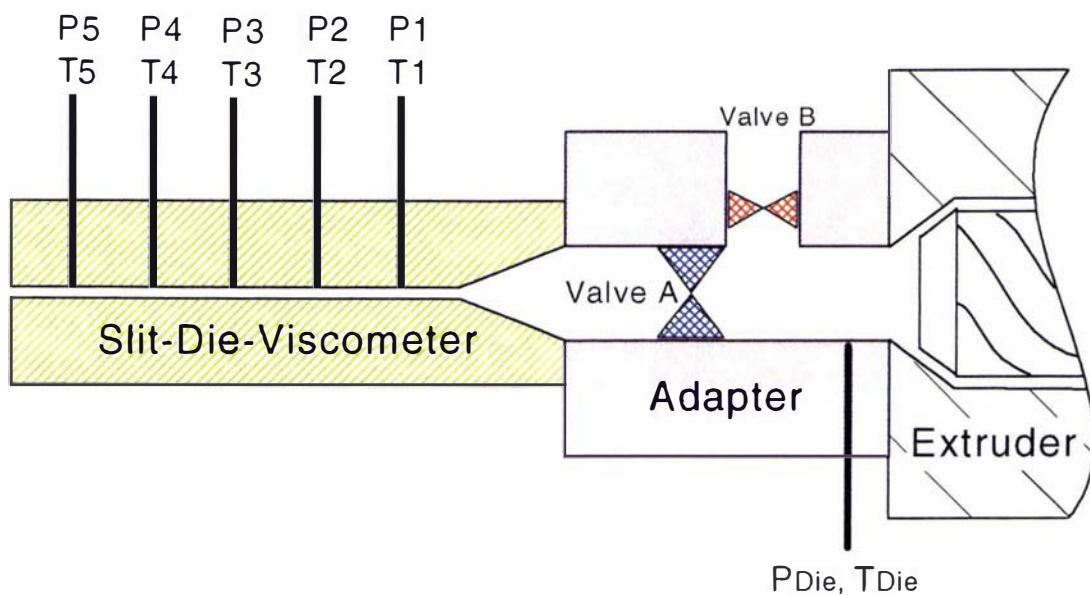
Figure 6.4 Schematic diagram of the new SDV and the adapter.

6.2.3 Data Acquisition and Control System

A computerised data acquisition and control system was developed for this study. All extrusion and SDV parameters were measured and stored by this system. The software contains data processing functions to perform real time noise filtering, error handling, statistical analysis, and on-screen data plotting. The data displayed included the SDV pressure profile, linear and log-log graphing of shear stress versus shear rate curves.

Temperature of the extruder barrels, screw speed, feed rate, water injection rate, extruder shaft torque and thrust pressure, die pressure, die-block temperature, temperature of the SDV, and the five pressures measured along the slit die were recorded. Raw data for each variable were taken at a rate of 1 Hz and written onto a computer file. Values of the rheological parameters K and n were calculated in real time and were written into another computer file together with the operating conditions as a summary report.

With real time on-screen plotting, the steadiness of the extrusion process could be easily visualised thus increasing the confidence and efficiency in operating the equipment and therefore the accuracy of the measurements.

A detailed flow chart of the program is shown in **Appendix C**.

6.2.4 Calibration of Pressure Transducers and Slit-Die-Viscometer

The accuracy of the pressure transducers used and linearity of their output signals were calibrated using a dead-weight tester at room temperature. All transducers have minimum 0.5% accuracy of the measurements.

The accuracy and reliability of the new SDV together with the data acquisition system was evaluated by comparing viscosity measurements of a Polycell paste (Polycell Products, Wattyl (NZ) Ltd.) determined using a home-built capillary viscometer and a rotational viscometer (Paar Physica, UDS 200, DIN Z3 bob & cup). Figure 6.5 shows that results obtained from the SDV are in a good agreement with those obtained from the capillary and rotational viscometers.
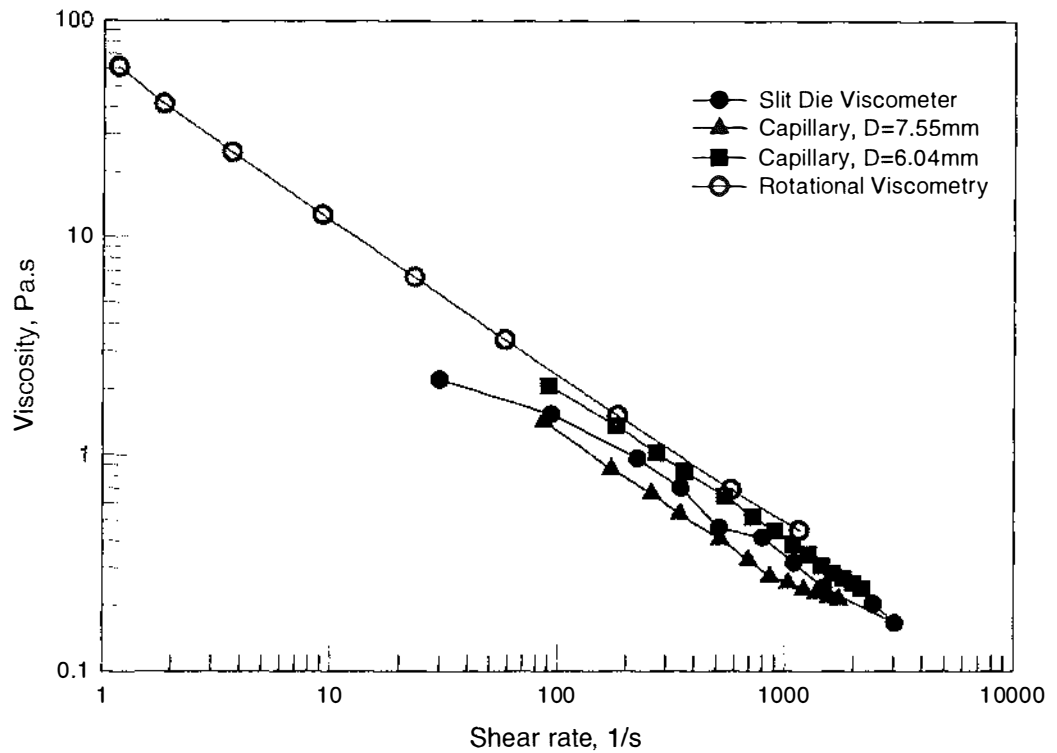
Figure 6.5 Viscosity of Polycell measured using the SDV, a capillary viscometer (two different capillary sizes) and a rotational viscometer.

6.2.5 Rheological Parameters Calculation

Wall shear stress, apparent shear rate and shear rate were calculated as

wall shear stress:
$$\tau_w = \frac{H}{2}\left(\frac{\partial P}{\partial x}\right) \cong \frac{H}{2}\frac{\Delta P}{L} \tag{6.3}$$

apparent shear rate:
$$\dot{\gamma}_{app} = \frac{6Q}{WH^2} \tag{6.4}$$

shear rate:
$$\dot{\gamma} = \frac{(2n+1)}{3n}\frac{6Q}{WH^2} \tag{6.5}$$

τ_w is the shear stress at the slit wall, $\partial P/\partial x$ is the pressure gradient along the flow direction, H is the height of the slit, W is the width of the slit, L is the length between two pressure transducers, Q is the volume flow rate and n is the power law index.

The calculation of corrected shear rate requires a sequential procedure because n is unknown. The index n is determined by the slope of a plot of $\log\left(\dfrac{\partial P}{\partial x}\right)$ vs $\log\left(\dfrac{6Q}{W \cdot H^2}\right)$.

Detailed calculations of the power law index n and consistency K can be found in **Appendix B**.

6.2.6 Calculation of the Specific Mechanical Energy (SME)

During extrusion, energy was applied, in the form of thermal and mechanical energies, to transform the raw materials into extruded products. In extrusion most of the energy applied is mechanical energy. The amount and intensity of mechanical energy received by the product during the process is measured with a parameter known as Specific Mechanical Energy (SME).

SME was calculated from the screw torque, screw speed and throughput as:

$$SME = \frac{\pi \times N \times (Tq - Tq_0)}{30 \times Q} \tag{6.6}$$

SME is Specific Mechanical Energy in $W \cdot h/kg$, N is screw speed in *rpm*, Tq is screw torque in $N \cdot m$, Tq_0 is the screw torque at empty load in $N \cdot m$, and Q is extruder throughput in kg/h. It was found that the screw torque Tq_0 at empty load was dependent on the screw speed and determined by the following equation:

$$Tq_0 = \frac{8.2755 \times N^{0.6361} - 1.5670}{8.4253 + N^{0.6361}} \tag{6.7}$$

6.2.7 Residence Time Distribution (RTD)

Data on the residence time distribution (RTD) were obtained by measuring the colour change in the extrudate samples after a quick injection of a dye solution. One millilitre of a 2% red dye solution (tetraiodofluorescein sodium salt Erythrosin B) was injected into the extruder through the feed inlet. The time taken for the red dye to appear in the extrudate was recorded. The extrudate was pulled from the die and guided onto a stainless steel table with a smooth surface to maintain a regular belt shape. Samples were taken until the red colour disappeared from the extrudate. The length of the sample

and time were recorded. During the sample collection period the operating conditions of the extruder was maintained constant. The samples were then dried, cut into pieces of equal length and ground. The length of the cut sample was used to calculate the time interval. The colour of the ground samples was measured by a Minolta Chroma Meter CR-200 using absolute chromaticity (L*a*b* colour space). In this study, the red colour co-ordinate +a* was used to measure the amount of red dye in the extrudate samples. A higher amount of red dye in the sample gave higher readings of +a*.

The RTD for a typical set of operating conditions using standard commercial corn grits is shown in Figure 6.6. The time corresponding to the average red colour co-ordinate +a* on Figure 6.6 was defined as the average residence time (t_{avg}) whereas the time to reach the highest value of +a* was defined as the peak time (t_{peak}). In this study, the average residence time was determined to be approximately 100s and t_{peak} was about 75s. The RTD had a skewed distribution and there was 25s difference between the t_{peak} and t_{avg}. The plot of RTD shows that appreciable back mixing occurs during extrusion. The melt flows back through the gaps between the screw flights and the extruder barrel because of the high pressure at the die.
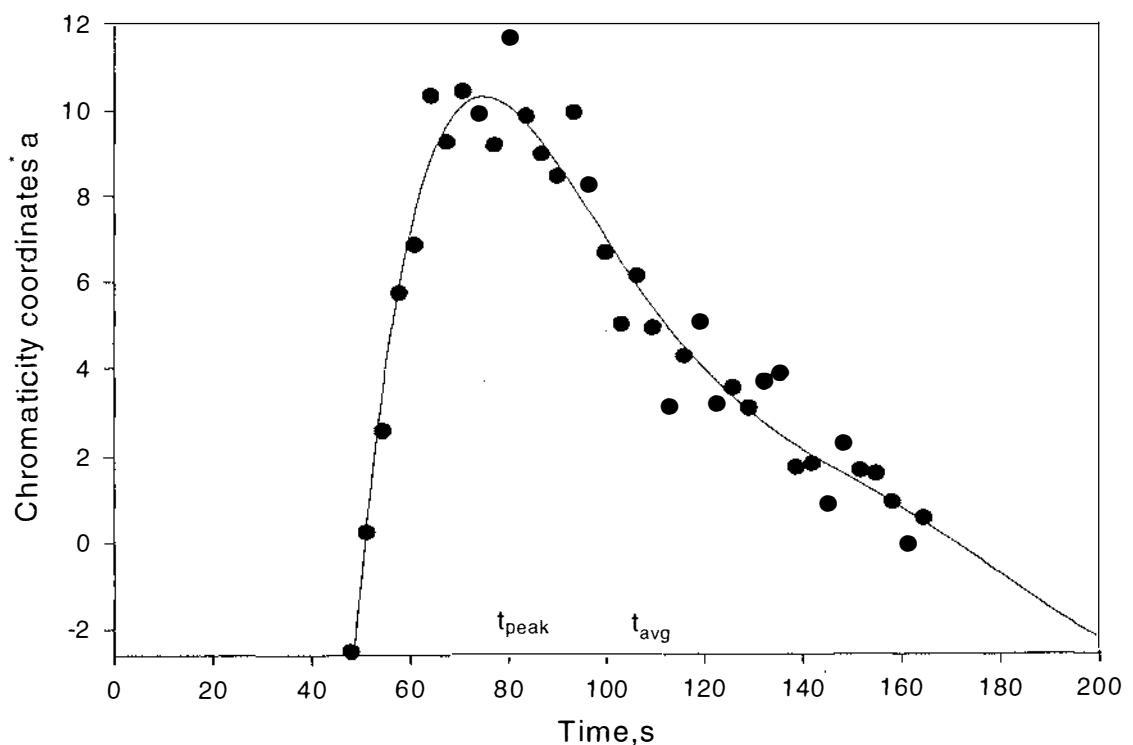
Figure 6.6 Measurements of the residence time distribution.

6.2.8 Grit Density

True density of corn grits is used to determine the amount of water to be added into the extruder to obtain a required level of moisture content during extrusion. The true density of corn grits was measured using a pycnometer-type apparatus. About 20 to 40 grams of corn grits were weighed and put into a tared 100ml flask at 25°C. About 40 to 60 ml of distilled water was added into the flask. The flask was gently shaken to release air bubbles that could be trapped inside the grit and water mixture. Distilled water was added to top the mixture up to 100ml and the mixture was weighed. By assuming that the volume changes in water and grits due to absorption of water by the grits are negligible, the following relationships exist:

$$V_m = V_g + V_w \qquad (6.8)$$

$$G_m = G_g + G_w \qquad (6.9)$$

V is volume in m^3; G is weight in kg. The subscripts m, g, and w represent the mixture, grits and water respectively. The density of the grit ρ_g (kg/m^3) is calculated as:

$$\rho_g = \frac{G_g}{V_g} \qquad (6.10)$$

By substituting G_g and V_g (equations 6.8 and 6.9) into equation (6.10), the density of the grit could be obtained by knowing the density of water ρ_w and measuring G_g, G_m, and V_m:

$$\rho_g = \frac{G_g}{V_g} = \frac{G_g}{V_m - V_w} = \frac{G_g}{V_m - (G_m - G_g)/\rho_w} \qquad (6.11)$$

6.2.9 Extrusion Sample Collection and Drying

For all extrusion experiments described in Chapter 7, two groups of samples, before the SDV and after the SDV, were collected to measure the degree of starch gelatinization. The sample after the SDV was collected when the valve A was fully open and valve B fully shut (Figure 6.4). In contrast, samples before the SDV were collected when valve A was at its minimum opening and valve B at its corresponding opening for that test. All samples were dried in a vacuum oven at 35°C for one week.

6.2.10 Degree of Starch Gelatinization

The degree of starch gelatinisation was determined by measuring the ratio of gelatinised to total starch in the sample. This method was described by Wootton *et al.* (1971) and modified by Owusu-Ansah et al. (1984). Dried samples of the collected extrudates were ground into fine powder (<200µm) using a cyclone mill (Cyclotech 1092 Sample Mill). Two 0.1g samples were weighed and put into labelled screw capped test tubes. 5ml of 0.25M KOH solution was added to the first sample test tube to solubilize the gelatinised starch. To the second sample 5ml of 0.7M KOH solution was added to obtain a solution of the total starch. The mixtures were shaken for 15 minutes and centrifuged at 3000g for 15 minutes. 1 ml of each supernatant was neutralised with 1 ml of HCL of the appropriate molarity (0.25M or 0.7M). The neutralised supernatants were diluted 20 times using distilled water. To 0.5 ml of the diluted starch solution, 4.5 ml distilled water was added followed by 50µl of iodine solution. The concentrations of the starch-iodine complex formed in the two aqueous suspensions were determined using a spectrophotometer (Model 240 Gilford Instrument, USA) measuring the absorbances at 600nm, which is a linear function of the concentration of the starch-iodine complex in the solution. The absorbance of the gelatinised sample, A_1, and that of the total soluble starch sample, A_2, were used to calculate the degree of starch gelatinisation from the following equation:

$$Degree\ of\ starch\ gelatinisation\ \% = \frac{A_1}{A_2} \times 100\% \qquad (6.12)$$

6.3 Experimental Approach

For the experiments, the extruder was allowed to reach stable operating conditions after startup. At the beginning of an experiment, valve *A* was fully open and valve *B* shut (see Figure 6.4). Die pressure and SME were carefully monitored during experiments. By carefully adjusting the openings of valves *A* and *B* by hand to maintain a constant extruder die pressure and constant SME, shear rate in the SDV was varied while the extruder operating conditions remained unchanged.

After the adjustments of valves *A* and *B* for a new shear rate at the SDV, the SDV and extruder were allowed to reach a new stable condition. The flow rate at the SDV slit

was then measured by weighing the extrudate product flowing out from the slit for a short period. However, as the melt was at high temperature, water loss due to evaporation could not be avoided before the sample was weighed. This loss must be added to the weight of the sample to obtain a corrected flow rate in the SDV.

The water loss was estimated by comparing the extruder throughput, which was calculated using the feed rate and the water injection rate, with the measured throughput by the weighing procedure. The difference between the calculated and the measured throughput was assumed to be the water loss during the weighing process and was expressed as a percentage of the calculated throughput. It was used to obtain a corrected flow rate in the SDV during that experiment using Equation 6.13.

$$MC_c = \frac{MC_0}{1 - L_w/100} \tag{6.13}$$

MC_c is the corrected sample weight, MC_o is the initial weight and L_w is the water loss in percentage. The corrected mass flow rate in the SDV was entered into the data acquisition system for calculation of the shear rate in the SDV using Equation 6.5.

Chapter 7

Experimental Results and Discussion (Extrusion)

7.1 Introduction

The new on-line Slit-Die-Viscometer (SDV), which includes the slit channel and the adapter valves (Chapter 6, Figure 6.4) was used to measure the rheological properties of melts produced by extrusion of corn grits. Five experiments using normal corn grits were carried out to investigate the effects of the extruder operating conditions on the viscosity of the melt. In the first experiment, the readings of five pressure transducers mounted in the SDV were used to examine the pressure profile along the slit at different flow rates. The pressure profile was found to be linear. Therefore, it was possible to calculate shear rate and shear stress in the SDV accurately. Appropriate procedures for using the new SDV were also set and verified in these experiments.

The general aim of the work presented in this chapter was to establish the relationships among raw materials, operational variables and system parameters of the extruder. The experiments were designed to study the effects of the following operational variables on the rheological properties of the melt and the extruder performance:

i)   Moisture content;

ii)   Temperature of the last two barrel sections;

iii)   Screw speed;

iv)   Feed rate;

v)   Degree of fill;

vi)   SDV temperature;

vii)   Starch composition;

viii)   Grit size;

ix)   Hybrids from the 92-93 season (grits manufactured from whole grains);

x)   Hybrids from the 94-95 season (grits manufactured from degermed grains).

7.2 Effects of Operating Conditions

7.2.1 Moisture Content

The purpose of this experiment was to investigate the effect of moisture content on the rheological properties and the degree of starch gelatinisation of the melt. It was also used to verify the operation of the new SDV and to validate the experimental procedures for using the SDV to measure shear rate and shear stress.

Normal corn grits were fed into the extruder by a volumetric feeder at 7.5kg/h. The moisture content of the melt was controlled by altering the water injection rate during the extrusion cooking process. Three water injection rates of 1.6, 2.0 and 2.7 litres/h were used to modify the moisture content of the melt to 31.7%, 35% and 40% respectively.

The temperatures in each of the barrel segments were kept constant at 60°C, 90°C, 120°C, and 120°C for barrel sections #1 to #4 respectively. The temperature of the SDV was constant at 120°C. Screw speed was set at 400rpm.

7.2.1.1 Pressure Distribution in the SDV

As the valve A was closed and valve B opened (Chapter 6, Figure 6.4) in 4 to 6 steps for each of the moisture contents, the shear rate at the SDV was modified and a pressure profile obtained. The pressure profiles along the SDV at various flow rates are plotted in Figures 7.1, 7.2 and 7.3 for melts with 31.7%, 35%, and 40% moisture content respectively. As indicated in the figures and in Table 7.1, the pressure increased linearly with distance measured from the exit of the SDV as all the profiles had a linear regression coefficient (R^2) greater than 0.97. This indicates that a fully developed flow was established in the slit channel and the necessary conditions for the calculation of shear rate and shear stress were met (Appendix B).

7.2.1.2 Effect on Melt Viscosity and Extruder Operational Parameters

Table 7.2 shows the effects of moisture content on the extruder operational parameters. For the three moisture contents, shear rates at the SDV were varied over a similar range by altering the apertures of the two valves on the new SDV. For each moisture content shear rate at the SDV had no effect on the operational variables SME, torque, thrust pressure and die pressure. As the moisture content was increased, the mean values for SME, torque, thrust pressure and die pressure decreased. These results are expected as the viscosity of the melt decreases when its moisture content is increased. The operating conditions, given in Table 7.2 along with their coefficients of variance (COV), show that the extruder was operating at stable conditions for each of the moisture contents. Thus, the raw material was subjected to the same thermo-mechanical treatment during the rheological measurements. It is concluded that the operation of the new SDV does not interfere with the operating conditions in the extruder.

As discussed in Chapter 5, the behaviour of the melt is non-Newtonian and the viscosity changes with shear rate. Therefore, it is more appropriate to use the term apparent viscosity. However, in this chapter *apparent viscosity* and *viscosity* will both be used to refer to the rheological properties of the melt without implying any distinction. Flow curves of the extrudate melt and plots of viscosity versus shear rate at different moisture contents are given in Figures 7.4 and 7.5. These figures illustrate that all the melts exhibited shear thinning behaviour and follow a power law model. As moisture content decreased from 40% to 31.7%, the power law consistency K increased from 2377 to 4963 (Table 7.3), indicating that moisture content has a very large effect on the melt viscosity. This result is consistent with those obtained by van Lengerich (1990), Padmanabhan and Bhattacharya (1993a, 1993b), and Vergnes *et al.* (1993). At high moisture content, the melt had a lower viscosity providing less resistance for the extruder screw rotation and flow through the extruder die. Thus the torque, the screw thrust pressure and die pressure decreased (Table 7.2). The SME applied to the material during the extrusion process also decreased when the moisture content of the melt increased from 31.7% to 40.0%. This was due to the fact that the torque on the screw decreased whereas the screw speed and extruder throughput remained constant.

It is worth noting that the slope of the plots for each moisture content in Figures 7.4 and 7.5 are similar indicating that the power law index n did not change with moisture content (Table 7.3). The values of n (0.30 to 0.37) indicate that the melt was highly shear thinning. Although the values of the rheological parameters K and n given in Table 7.3 appear as single data points, they represent the means of more than one thousand data points with an error less than 2% for the entire experiment.

7.2.1.3 Effect on Degree of Starch Gelatinisation

As a result of the lower mechanical energy input and decreased internal friction, at higher moisture contents, the starch was less gelatinised (Table 7.4). Starch gelatinisation occurs below 120°C, therefore, if gelatinisation is incomplete in the barrel, it will continue in the SDV when it is operated at 120°C. As available water in the melt was limited and the residence time of the melt in the SDV was short, the amount of starch gelatinisation occurring in the slit viscometer was small.
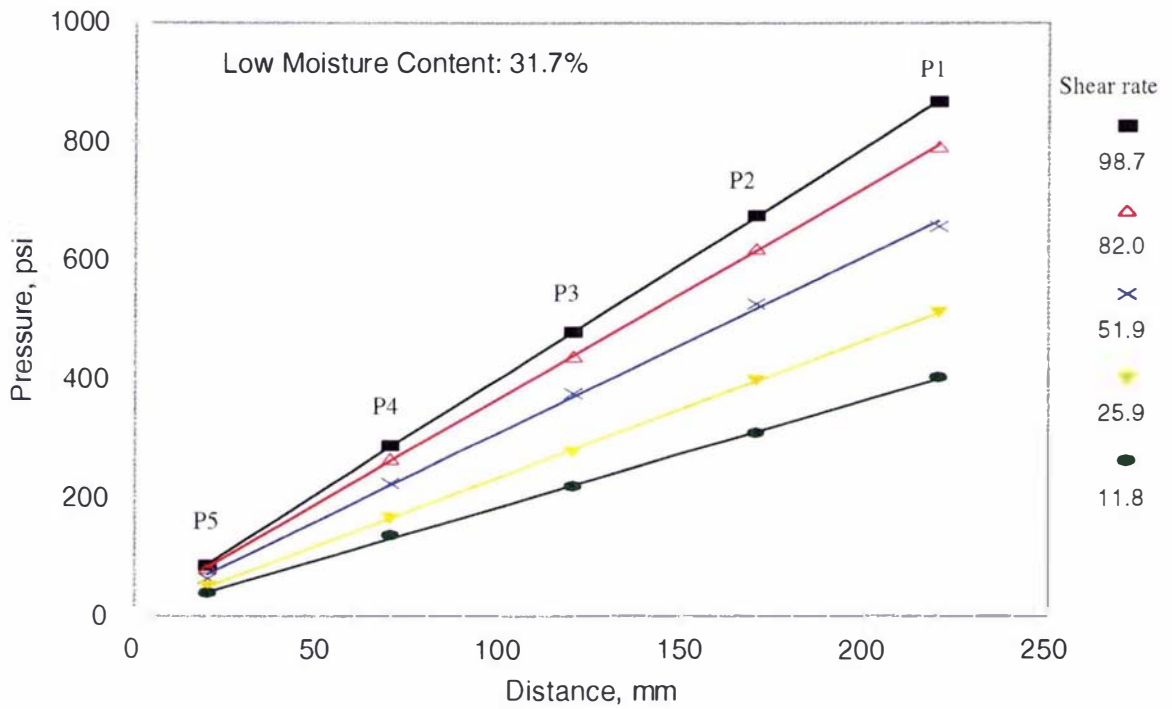
Figure 7.1 Pressure profiles along the SDV slit for various shear rates at 31.7% moisture content.
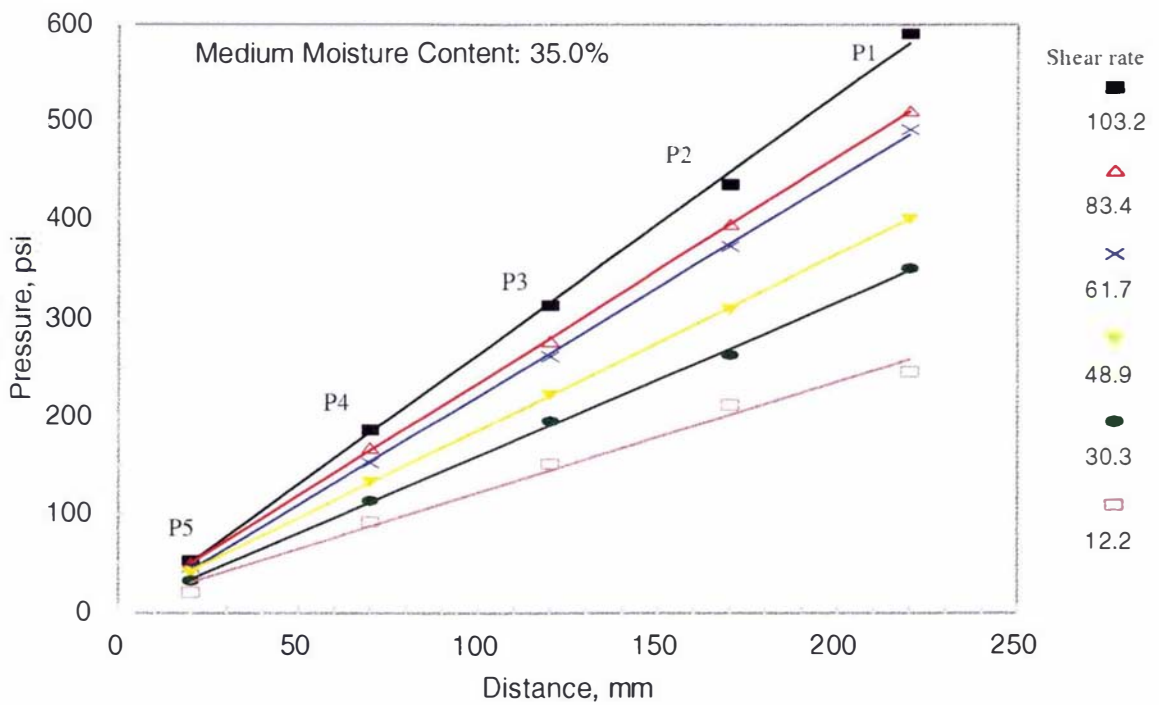
Figure 7.2 Pressure profiles along the SDV slit for various shear rates at 35.0% moisture content.
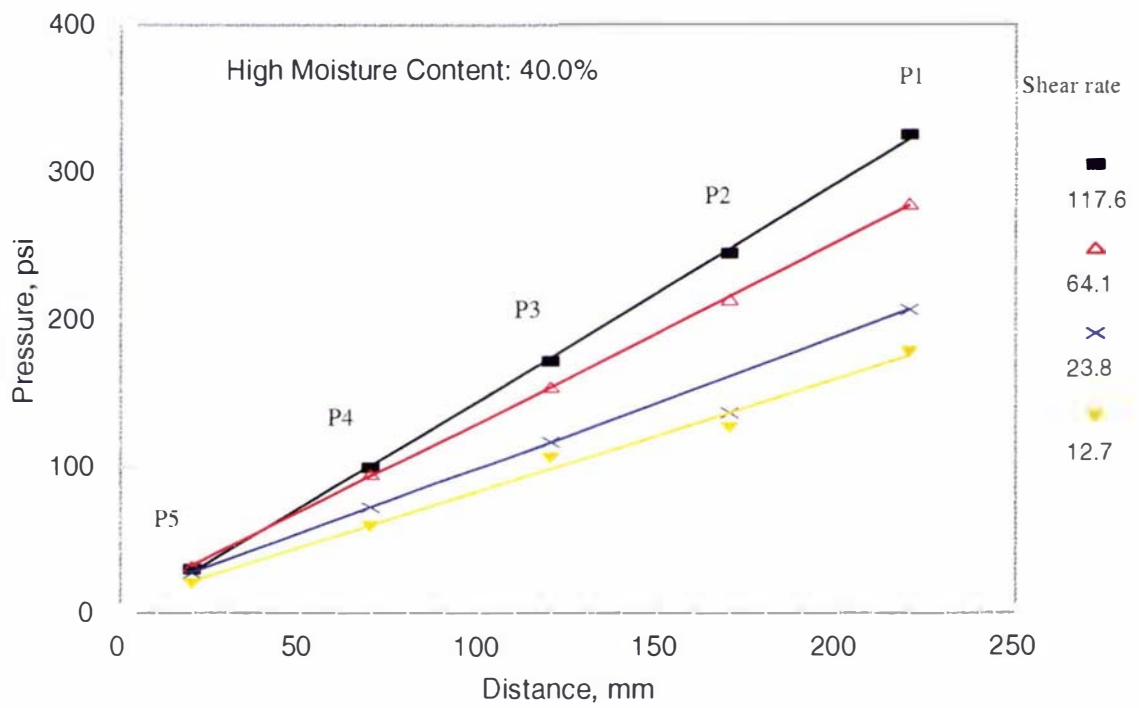
Figure 7.3 Pressure profiles along the SDV slit for various shear rates at 40.0% moisture content.

Table 7.1 R^2 of linear regression for pressure profiles in the SDV at different shear rates.

Moisture content, %	App. shear rate, 1/s	R^2
31.7%	98.7	0.9999
	82.0	0.9999
	51.9	0.9988
	25.9	0.9998
	11.8	0.9993
35.0%	103.2	0.9984
	83.4	0.9999
	61.7	0.9995
	48.9	0.9999
	30.3	0.9989
	12.2	0.9868
40.0%	117.6	0.9994
	64.1	0.9998
	23.8	0.9763
	12.7	0.9887

Table 7.2 Effect of moisture content on extruder operating conditions.

Moisture content (%)	Test #	Apparent shear rate (1/s)	SME (W•h/kg)	Torque (Nm)	P_{thrust} (bar)	P_{die} (Psi)
	1	98.7	50.8	18.2	72.7	1228.9
	2	82.0	52.2	18.5	74.8	1261.2
	3	51.9	50.3	18.1	73.0	1222.0
31.7	4	25.9	51.3	18.3	73.7	1248.9
	5	11.8	50.0	18.0	72.7	1217.7
	Mean	-	50.9	18.2	73.4	1235.7
	COV	-	1.7%	1.0%	1.2%	1.5%
	1	103.2	30.7	14.0	48.5	830.7
	2	83.4	29.6	13.7	47.6	810.2
	3	61.7	29.3	13.6	47.7	810.2
	4	48.9	28.6	13.5	47.3	810.1
35.0	5	30.3	29.9	13.8	48.9	832.3
	6	12.2	29.9	13.8	49.2	843.5
	Mean	-	29.7	13.7	48.2	822.8
	COV	-	2.4%	1.2%	1.6%	1.8%
	1	117.6	12.0	10.0	26.4	468.5
	2	64.1	12.7	10.2	26.4	471.1
	3	23.8	11.9	10.0	26.0	463.5
40.0	4	12.7	13.0	10.3	27.0	481.8
	Mean	-	12.4	10.1	26.5	471.2
	COV	-	4.3%	1.4%	1.6%	1.6%
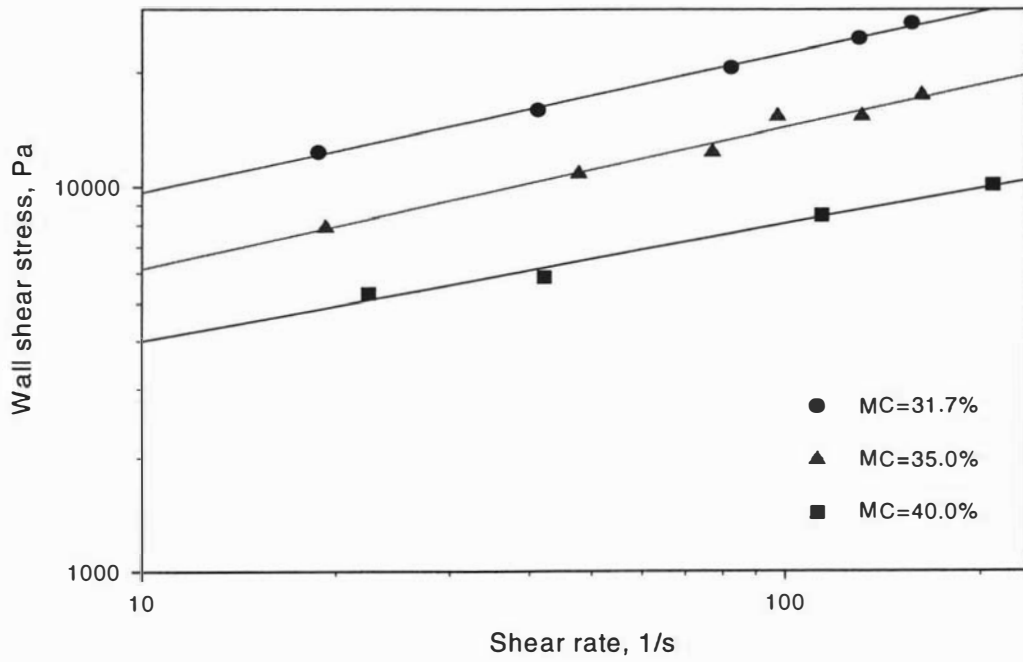
Figure 7.4 Flow curves for melts produced at three different moisture levels.
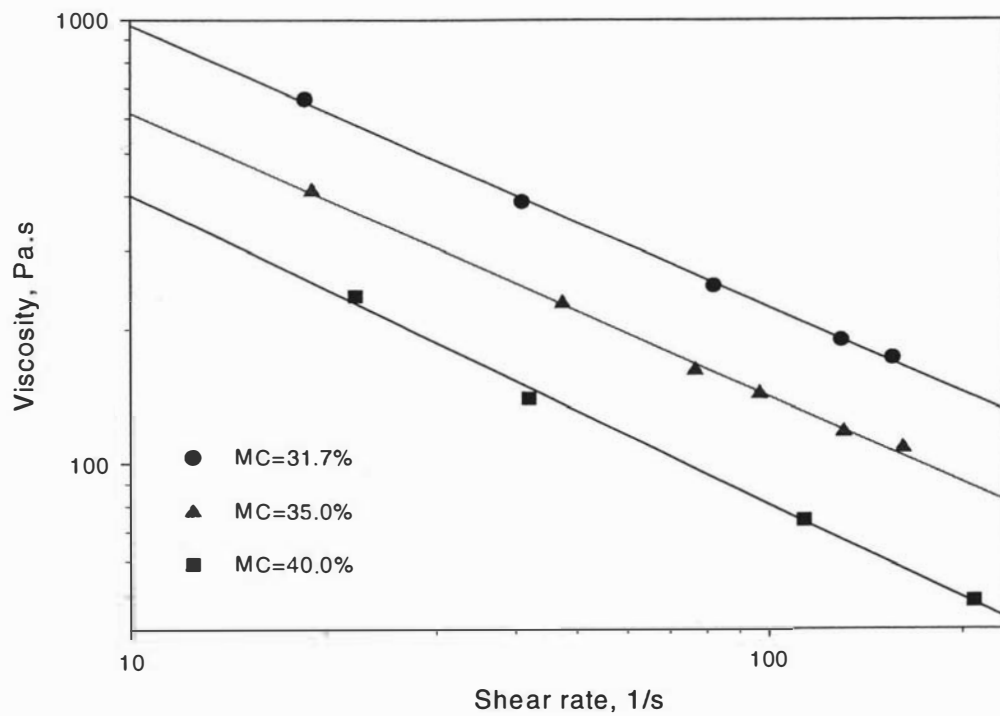
Figure 7.5 Plots of viscosity versus shear rate for melts produced at three different moisture levels.

Table 7.3 Values of power law index n and consistency K of melts produced by extrusion at three moisture contents.

Moisture content, %	Power law index, n	Power law consistency, K
31.7	0.36	4962.9
35.0	0.37	3106.0
40.0	0.30	2377.0

Table 7.4 Degree of starch gelatinisation of melts produced by extrusion at different moisture contents (95% confidence interval).

Moisture content, %	Sample before SDV degree of starch gelatinisation, %	Sample after SDV degree of starch gelatinisation, %
40.0%	80.1 ± 0.9	82.9 ± 1.3
35.0%	91.2 ± 1.3	88.0 ± 2.6
31.7%	86.2 ± 1.3	88.3 ± 0.7

7.2.2 Barrel Temperature (Last Two Barrel Sections)

The effect of the barrel temperature on the viscosity of the melt was investigated by changing the temperature profile in the extruder barrels. For this work, barrel temperature refers to the temperature of the last two barrel sections. The temperatures for the first two sections were kept constant at 60°C and 90°C, whereas, the last two sections were set at different temperatures increasing by 10°C steps from 90°C to 160°C. The temperature of the SDV was kept at 120°C for all the experimental runs.

Normal corn grits were used and fed at a constant feed rate of 7.5kg/h. Screw speed was set at 450rpm and the moisture content of the melt at 35% wwb.

Figure 7.6 shows the changes of starch gelatinisation with the barrel temperature. The figure clearly illustrates that increasing the barrel temperature caused a large increase in the degree of gelatinisation. When the last two sections were set to 90°C, only 64% of starch gelatinised whereas at temperatures above 130°C, more than 95% of the starch gelatinised during the extrusion process. Statistical analysis of the data shows that there is no difference in the degree of starch gelatinisation at temperatures above 130°C. Between 90°C and 130°C (Figure 7.6) the degree of starch gelatinisation varies linearly with temperature.

Figure 7.7 shows that changes in the barrel temperature in the range 90°C to 160°C resulted in small changes in the apparent viscosity of the melt measured at 120°C. As barrel temperature increased from 90°C to 160°C, the melt became less shear thinning as the power index n increased from 0.40 to 0.49 (Table 7.5). A similar result was also observed by Vergnes and Villemaire (1987) using corn starch, Senouci and Smith (1988a) using maize grits, Lai and Kokini (1990) using corn starch, and Padmanabhan and Bhattacharya (1993a, 1993b) using corn meal. It is thought that the rheological properties of the melt are closely related to the molecular weight distribution of the starch (van Lengerich, 1990). Change in the shear thinning behaviour has been attributed to a decrease in the size of the starch molecules due to its degradation at high temperatures (Vergnes et al., 1993).

There is no consistent relationship between barrel temperature and the consistency index K but a maximum value of K occurred at temperatures between 120°C and 140°C. The power law model is characterised by two parameters K and n. The apparent viscosity is determined from both K and n using the formula $\eta_{app} = K \dot{\gamma}^{n-1}$. Since the parameters K and n can not vary independently (Schowalter, 1978), it is more appropriate, for comparison purposes, to calculate the apparent viscosity at different shear rates. Values of apparent viscosity, calculated using the rheological parameters given in Table 7.5 and shear rates that can exist inside the extruder, are plotted in Figure 7.8 as a function of the barrel temperature. These plots clearly show that the maximum apparent viscosity occurs at about 130°C.

It is thought that the increase in melt viscosity with temperature up to 130°C is due to the increase in the degree of starch gelatinisation, whereas the decrease in the melt viscosity above 130°C indicates that further starch degradation, probably dextrinisation occurred. This phenomenon has been reported by Colonna *et al.* (1984) and Davidson *et al.* (1984a) during extrusion of corn and wheat starch at temperatures above 120°C. In these two reports, the average macromolecular weight of starch was found to be substantially reduced during extrusion at temperatures above 120°C.

As illustrated in Figures 7.9, 7.10, and 7.11, SME, torque, die pressure and thrust pressure all had peak values at barrel temperatures near 130°C and followed the same trend as that followed by the apparent melt viscosity. The variations in torque and SME were caused by changes in the viscosity of the melt. When the barrel temperature was lower than 130°C, the melt had a lower viscosity, therefore, the resistance to the rotation of the screw was smaller resulting in lower torque. When the viscosity of the melt decreased at temperatures above 150°C, torque, SME, die pressure and thrust pressure all decreased.
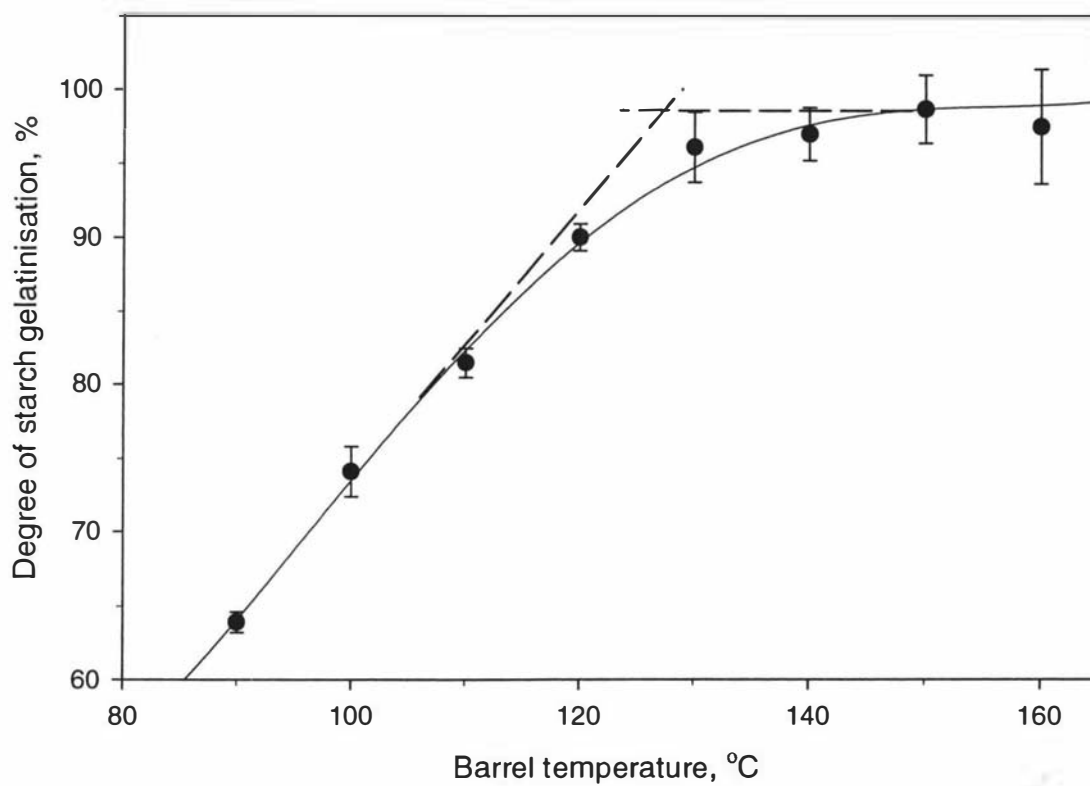
Figure 7.6 A plot of the effect of extruder barrel temperature (last two barrel sections) on the degree of starch gelatinisation. Error bars were calculated using a 95% confidence interval.
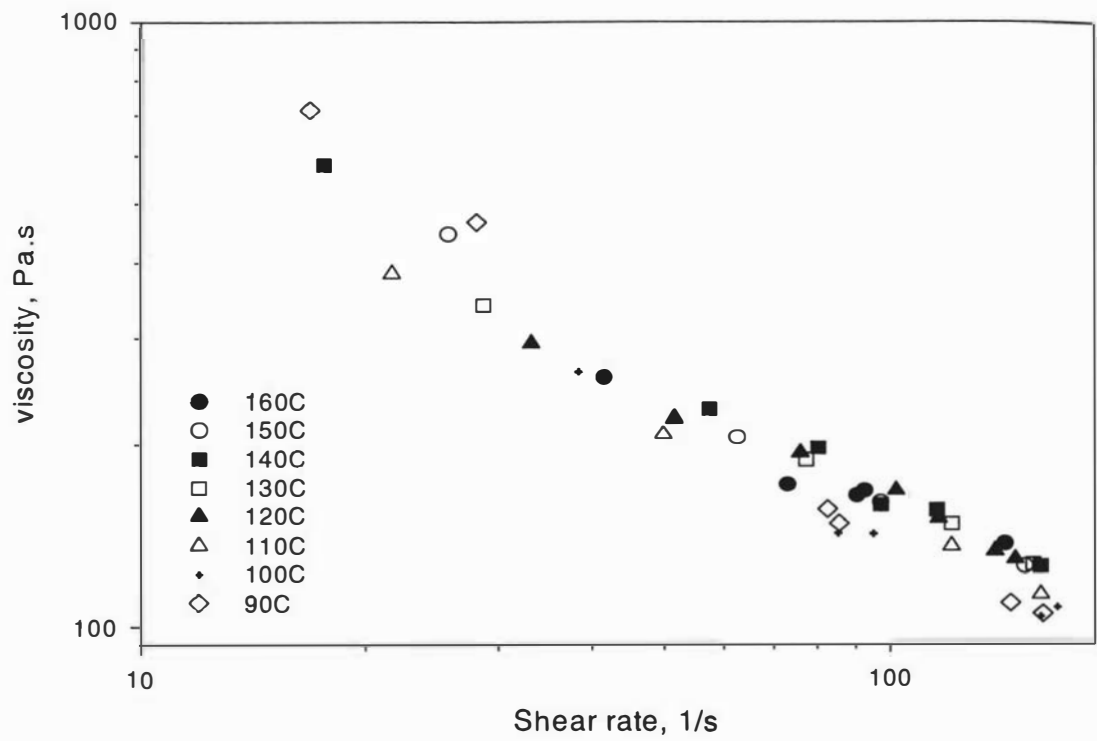
Figure 7.7 Effect of barrel temperatures (last two sections) on melt viscosity measured at 120°C.

Table 7.5 Rheological properties of the melt produced 'at different barrel temperatures.

Temperature of the last two barrels	Viscosity	
	Power law index, n	consistency, K
160°C	0.49	1959.3
150°C	0.43	2528.5
140°C	0.40	3199.3
130°C	0.41	2878.5
120°C	0.39	3106.2
110°C	0.40	2809.5
100°C	0.37	3009.1
90°C	0.40	2558.7
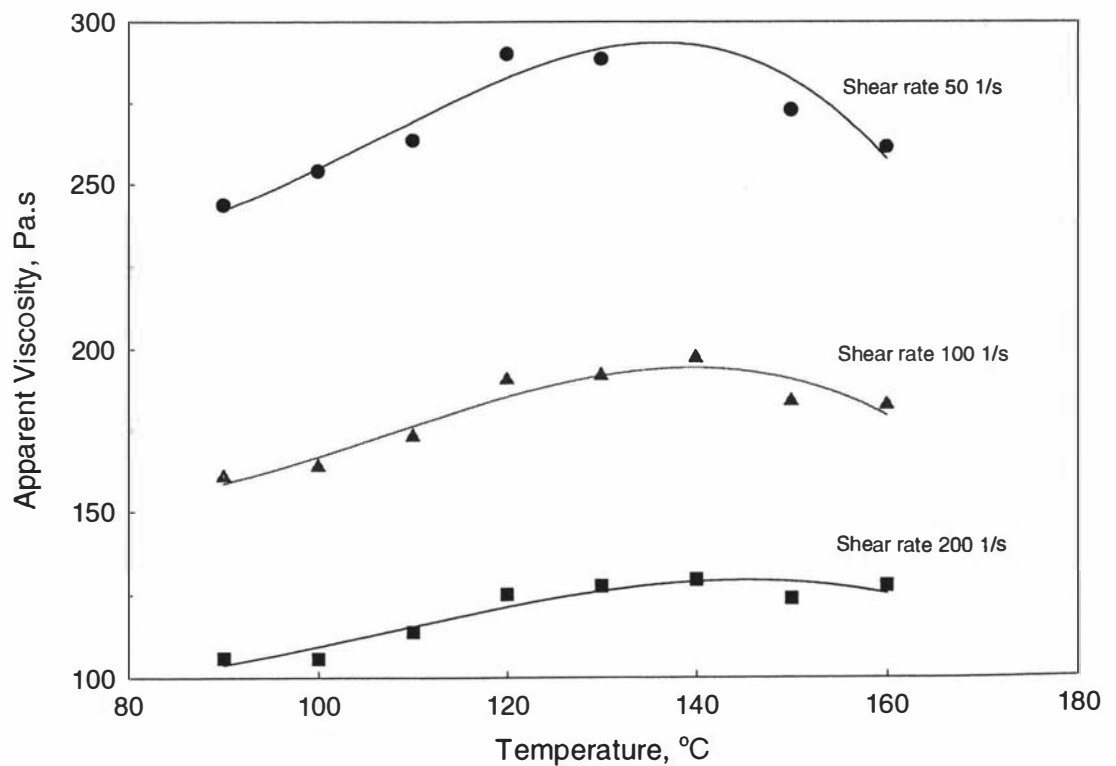
Figure 7.8 Effect of barrel temperature on apparent viscosity calculated at different shear rates.
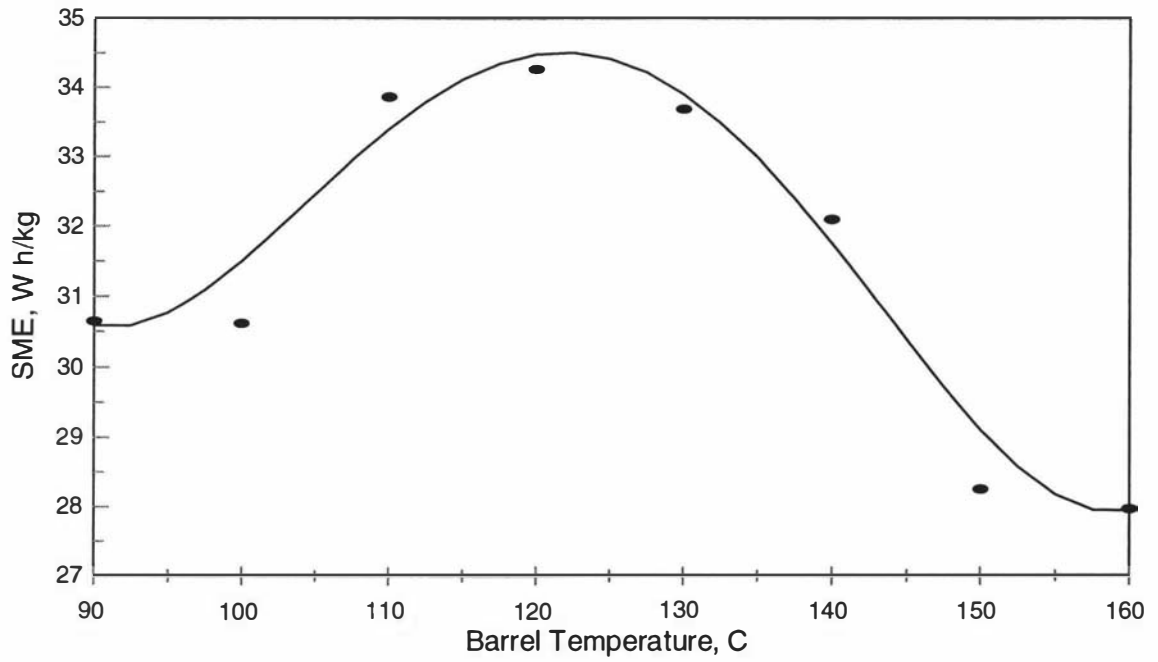
Figure 7.9 Effect of barrel temperature on SME.
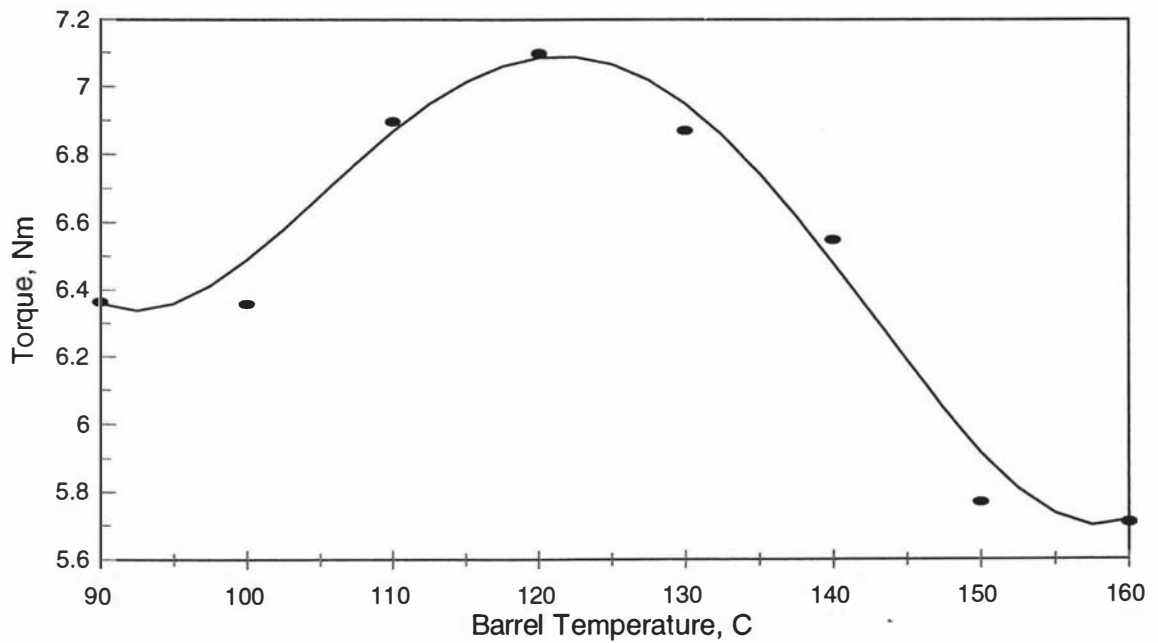
Figure 7.10 Effect of barrel temperature on torque.
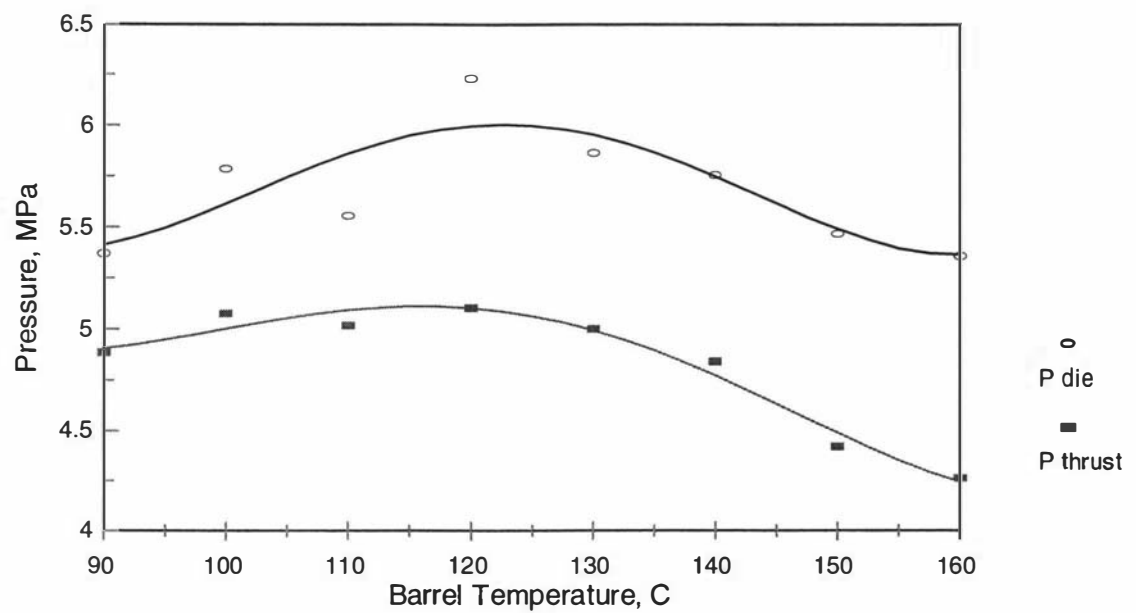
Figure 7.11 Effect of barrel temperature on die pressure (P_{die}) and thrust pressure (P_{thrust}).

7.2.3 Degree of fill - Screw Speed and Feed Rate

Degree of fill is defined as the ratio between the extruder feed rate and the screw speed (van Lengerich, 1990). The effects of degree of fill on the rheological properties and the degree of starch gelatinisation of the melt were investigated in three experiments. The experiments were conducted by: (1) increasing the screw speed at constant feed, (2) increasing the feed rate at constant screw speed, and (3) simultaneously increasing both feed rate and screw speed proportionally to keep the degree of fill constant. Increases in screw speed or decreases in feed rate result in lower degree of fill.

For the three experiments, the temperatures of the four barrel sections were constant at 60°C, 90°C, 120°C, 120°C. Normal corn grits were used. Moisture content of the melt was maintained at 35% wwb and the temperature of the SDV was kept constant at 120°C.

7.2.3.1 Effect of Screw Speed at Constant Feed Rate

In this experiment, the feed rate was kept constant at 9.5kg/h while the screw speed was varied. Three speeds 300rpm, 400rpm, and 500rpm were used.

SME increased and torque decreased when screw speed increased from 300 rpm to 500 rpm (Figures 7.12 and 7.13). This agrees with the results reported by van Lengerich (1990) using wheat starch and a conventional SDV. Increasing screw speed increases the shear and friction in the extruder resulting in a higher rate of mechanical energy transfer. However, an increase in screw speed also reduces the residence time and the degree of fill if feed rate is kept constant. Thus, the increase in mechanical energy due to the increase in screw speed is in part compensated by the shorter residence time. When the screw speed was increased from 300rpm to 500 rpm, the net result was an increase of SME (Figure 7.12) and a 6.8% increase in the degree of starch gelatinisation (Table 7.6). The increase in the degree of starch gelatinisation with the increase in SME is consistent with data reported by Senouci and Smith (1988a) and van Lengerich (1990).

The effect of screw speed on the rheological properties of the melt is, however, more difficult to see. Figure 7.14 shows that there is negligible change in these properties as

screw speed varies indicating that, in rheological terms, melts produced at different screw speeds have similar properties.

The melts exhibited a shear thinning behaviour as the values of n obtained were less than one ($n<1$) (Table 7.6). This behaviour explains the results shown in Figures 7.12 and 7.13. The mean shear rate produced by the screw rotation in the extruder barrel can be estimated by the following equation:

$$\dot{\gamma} = \frac{\pi \cdot ScrewSpeed \cdot ScrewDiameter \cdot \cos(ScrewFlightAngle)}{60 \cdot ScrewFlightChannelDepth}. \tag{7.1}$$

Thus, the apparent viscosity of the melt at the three screw speeds can be calculated using the rheological parameters given in Table 7.6 and the shear rates calculated by Equation 7.1. The shear thinning behaviour of the melt is illustrated in Figure 7.15 where a decrease in the apparent viscosity with increasing screw speed is noted. Values of the shear rate for each screw speed calculated by Equation 7.1 are included in the figure. This shear thinning behaviour is the main reason for the decrease in torque observed in Figure 7.13.

The rheological behaviour of a non-Newtonian fluid is somewhat more complex than that described by the power law model. At very low and very high shear rates, the fluid exhibits Newtonian behaviour having a limiting viscosity at very small shear rates η_0 and a limiting viscosity at very large shear rates η_1 (Steffe, 1992). They are illustrated as zones A and C in Figure 7.16. Both η_0 and η_1 are independent of shear rate. At a middle zone B, where the apparent viscosity decreases with shear rate, the power law equation is a suitable model to describe the behaviour of the fluid. In this experiment for screw speeds higher than 400 rpm, values of shear rate within the extruder are considered to be in zone B (Figure 7.16) but closer to zone C. Thus, the changing rate of apparent viscosity with screw speeds from 400 rpm to 500 rpm (0.35 Pa·s/rpm) is smaller than that obtained when the screw speed increased from 300 to 400 rpm (0.6 Pa·s/rpm). Given the relationship of SME with torque and screw speed at a constant feed rate ($SME = \dfrac{Torque \times ScrewSpeed}{Feed\ Rate}$), the small change observed in SME when the screw speed increased from 300 to 400 rpm was due to the large decrease in viscosity and therefore torque. When the screw speed increased further to 500 rpm, the viscosity

decreased to a lesser extent resulting in a smaller decrease in torque (Figure 7.13). Thus, the increase in screw speed from 400 rpm to 500 rpm resulted in a large increase in SME (Figure 7.12).

The above discussion clearly illustrates that the value of SME is related to the rheological properties of the melt. The torque can also be considered a function of the melt viscosity and the screw speed. Thus, the definition of SME could not be physically well defined as it contains variables that are not truly independent.
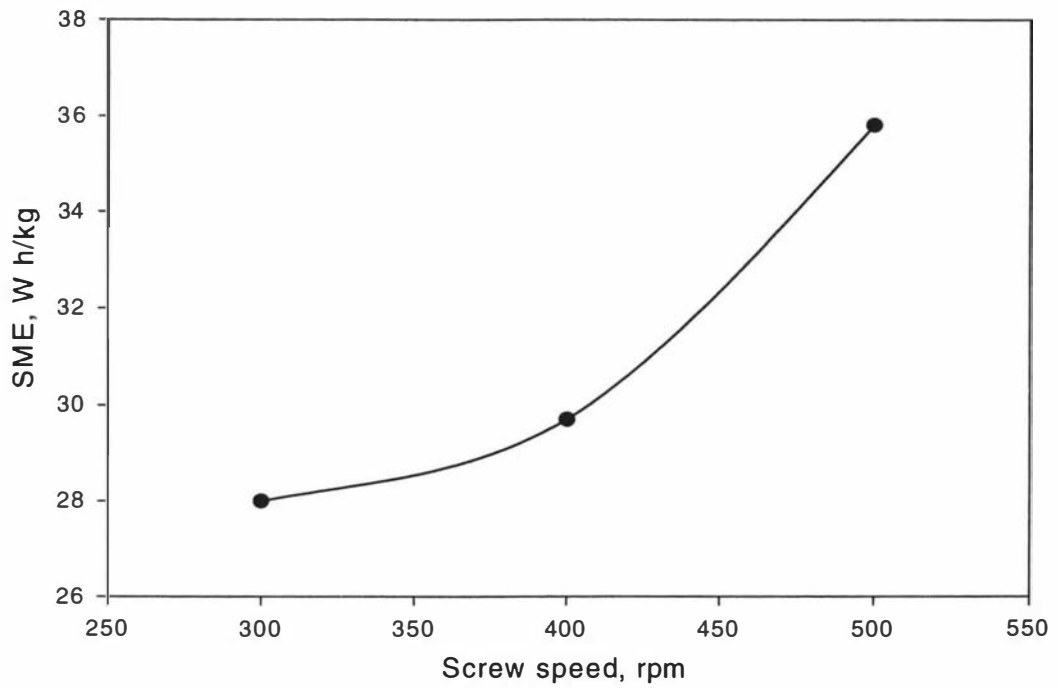
Figure 7.12 The effect of screw speed on SME at a constant feed rate of 9.5kg/h.
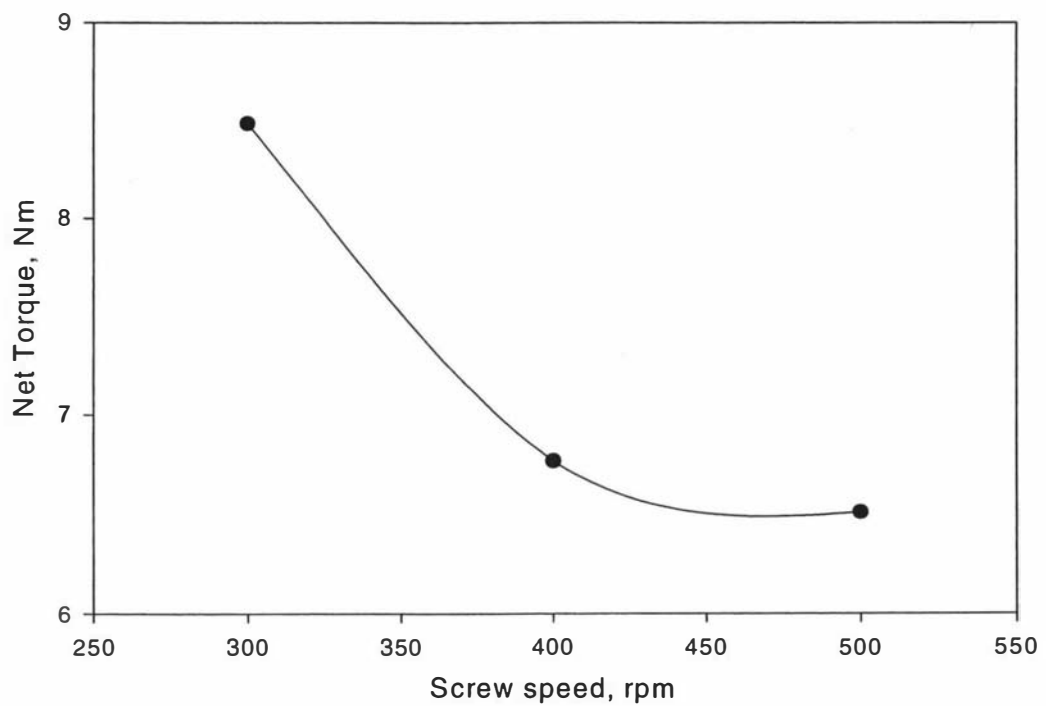
Figure 7.13 The effect of screw speed on screw torque at a constant feed rate of 9.5kg/h.

Table 7.6 Rheological properties and degree of starch gelatinisation of the melts at different screw speeds at a constant feed rate 9.5kg/h.

Screw speed rpm	Viscosity		Degree of starch gelatinisation, %
	Power law index, n	Power law consistency, K	
500	0.35	3326.6	93.1 ± 4.6
400	0.37	3106.0	91.2 ±1.4
300	0.39	3005.9	86.3 ±1.2
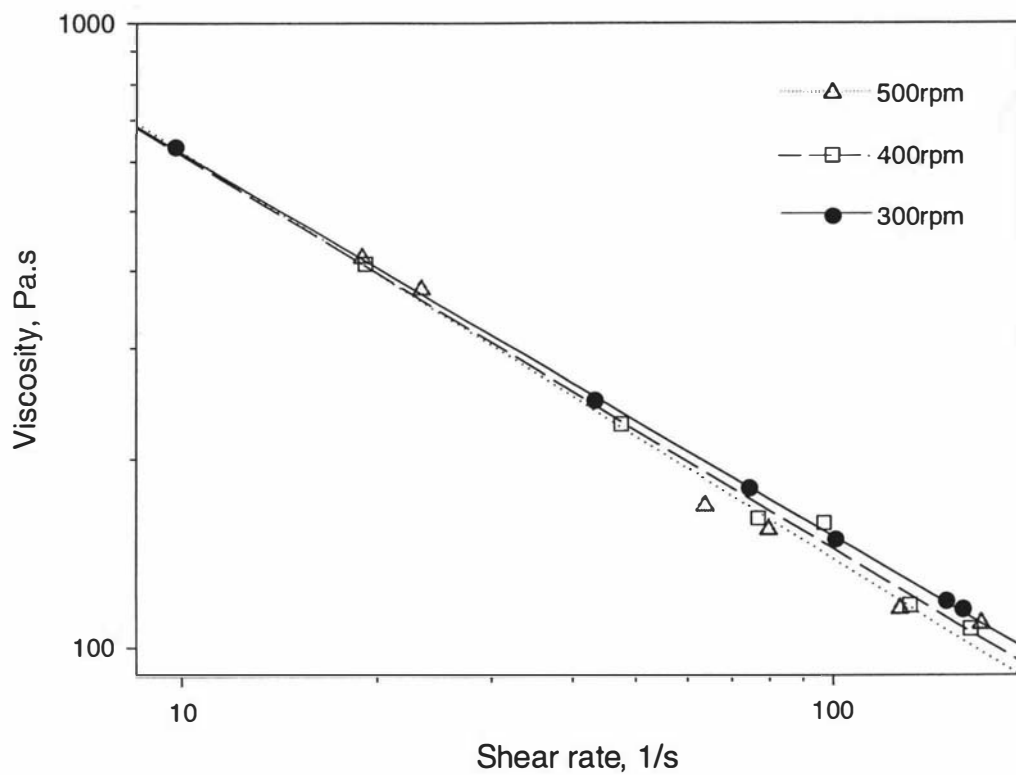
Figure 7.14 The effect of screw speed on the melt viscosity measured at 120°C. Feed rate was kept constant at 9.5kg/h.
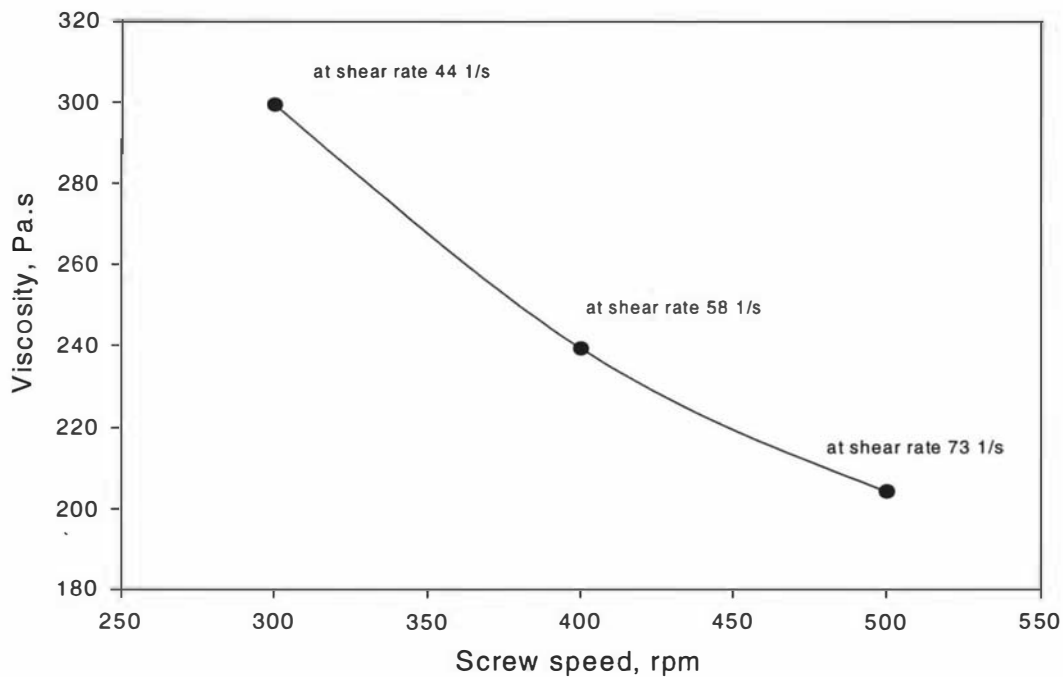
Figure 7.15 Viscosity of the melt in the extruder barrel at the three screw speeds.
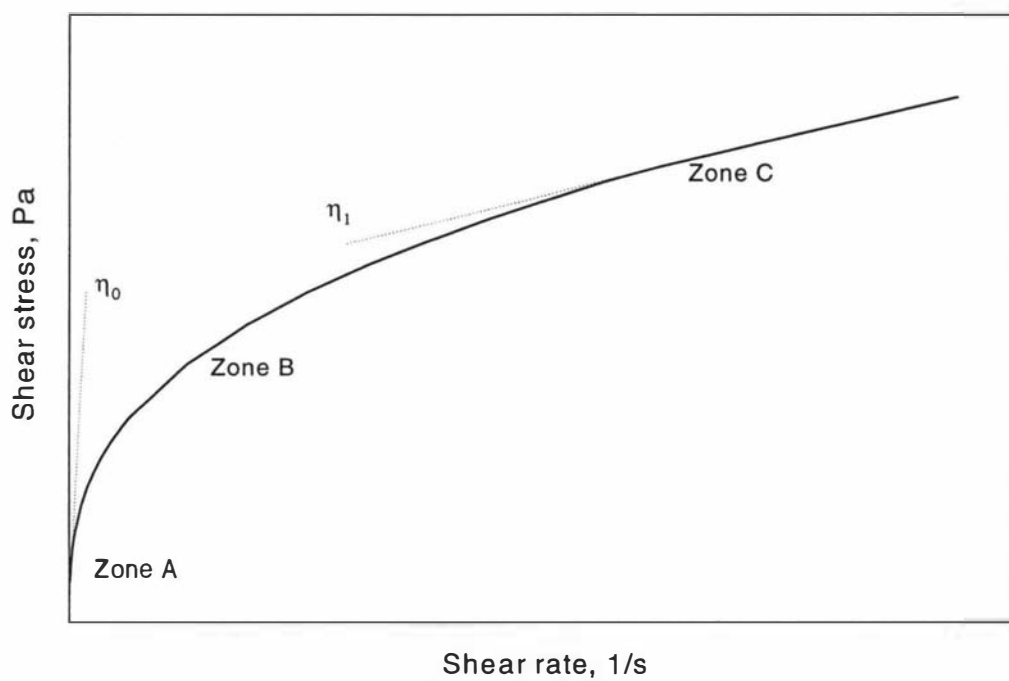
Figure 7.16 Complete flow curve of a pseudoplastic fluid.

7.2.3.2 Effect of Feed Rate at Constant Screw Speed

For this experiment the screw speed was set at 450rpm and three different feed rates were used 7.1kg/h, 9.8kg/h, and 12.1kg/h.

The torque increased as the feed rate was increased from 7.1kg/h to 12.1kg/h (Figure 7.17). SME was approximately constant when the feed rate increased from 7.1kg/h to 9.8kg/h and decreased as the feed rate increased to 12.1kg/h (Figure 7.18). At high feed rates, these results are in agreement with those of van Lengerich (1990) who found that low SME values are associated with a higher degree of fill. The values of SME reported in Figure 7.18 represent the mean of more than one thousand data points calculated with the values of torque, screw speed and feed rate, which have an error of less than 2% at 95% confidence. Therefore, the error in SME does not exceed 3.5%. During extrusion and rheological measurements, the melt produced from a batch of corn grits was continuously flowing through the SDV. Thus, each of these data points can be considered a true replicate.

Table 7.7 shows that the rheological parameters n and K varied slightly for the three feed rates. The degree of starch gelatinisation increased between about 87% to 90% as feed rate increased from 7.1kg/h to 9.8kg/h. No change in gelatinisation was observed when the feed rate further increased from 9.8kg/h to 12.1kg/h. The values of die pressure, also reported in Table 7.7, increased with increasing feed rate. These results would be indicating that the thermo-mechanical treatment varies with feed rate.

In this experiment, the screw speed was constant at 450 rpm, therefore, the shear rate inside the extruder barrel did not change when the feed rate increased. The shear rate was estimated by Equation (7.1) and found to be about 66 1/s. Figure 7.19 shows that at shear rates higher than 60 1/s, the melt produced at the highest feed rate (12.1 kg/h) had the lowest viscosity. The viscosities for the other two shear rates were similar. Thus, the rate of change in viscosity due to increase in feed rate from 7.1 to 9.8 kg/h is smaller (1.5 Pa.s/kg/h) than that for feed rates between 9.8 to 12.1/kg/h (3.0 Pa.s/kg/h). This explains why when feed rate increased from 7.1 to 9.8 kg/h, the torque increased at a higher rate resulting in a very small change in SME (Figure 7.18). However, when the feed rate was increased from 9.8 to 12.1 kg/h, there was a larger decrease in melt

viscosity. This is clearly illustrated in Figure 7.17 in where the torque increased at a smaller rate and in Figure 7.18 in where the SME ($\frac{Torque \times ScrewSpeed}{Feed\,Rate}$) decreased as the feed increased. It is worth noting that these effects could be much larger in an industrial machine where energy introduced into the melt could be significantly greater.

The above discussion clearly shows that at constant screw speed torque is related to feed rate and the values of SME do not follow a simple relationship when the feed rate is varied.

The decrease in melt viscosity observed when the feed rate increased can not be attributed to the shear thinning behaviour of the melt because the screw speed (shear rate) was constant. The similar values of degree of starch gelatinisation measured can not explain the decrease in melt viscosity when the feed rate was increased to 12.1 kg/h, suggesting that the melt was subjected to further starch degradation that could not be detected by the method used to determine the degree of starch gelatinisation.
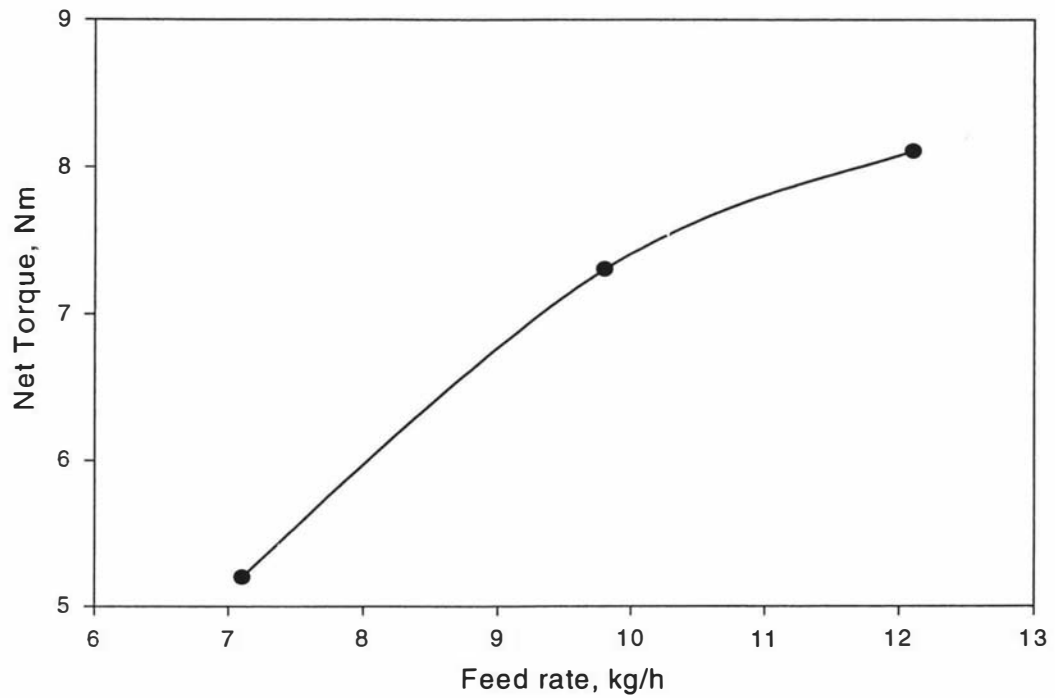
Figure 7.17 The effect of feed rate on torque at a constant screw speed 450rpm.
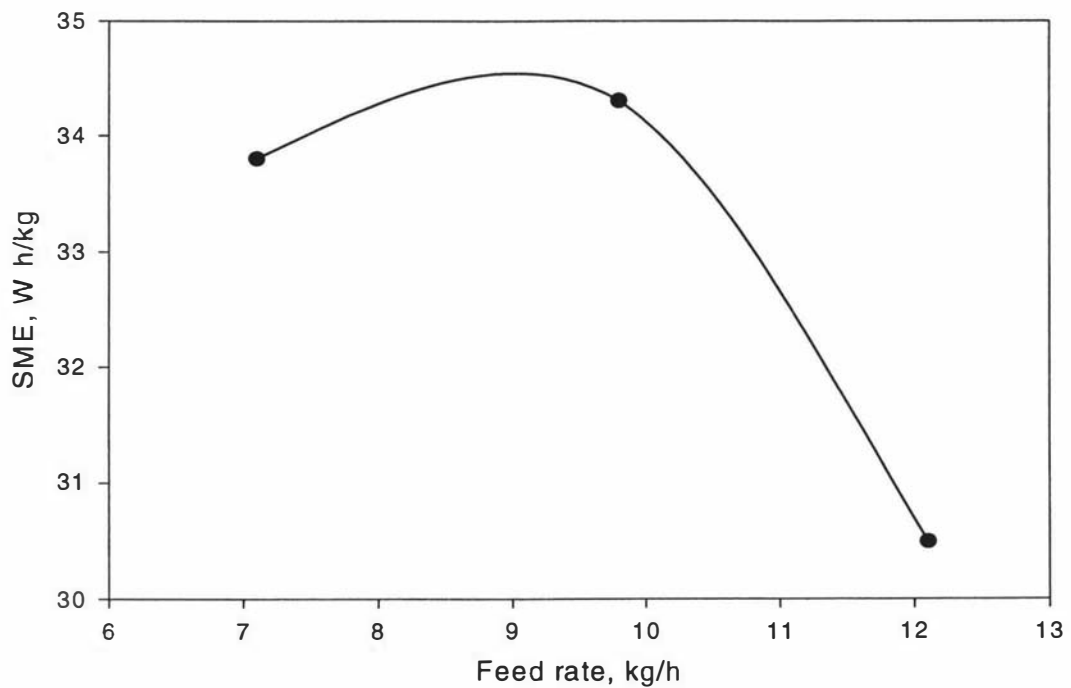
Figure 7.18 The effect of feed rate on SME at a constant screw speed 450rpm.

Table 7.7 The effect of feed rate on the rheological properties and the degree of starch gelatinisation at a constant screw speed 450rpm.

Feed rate	Viscosity		Die Pressure,	Degree of starch
kg/h	Power law index, n	Power law consistency, K	MPa	gelatinisation, %
7.1	0.42	2683.5	5.20	86.6 ± 1.5
9.8	0.45	2324.4	6.23	90.0 ± 1.0
12.1	0.40	2780.6	6.31	89.8 ± 2.2
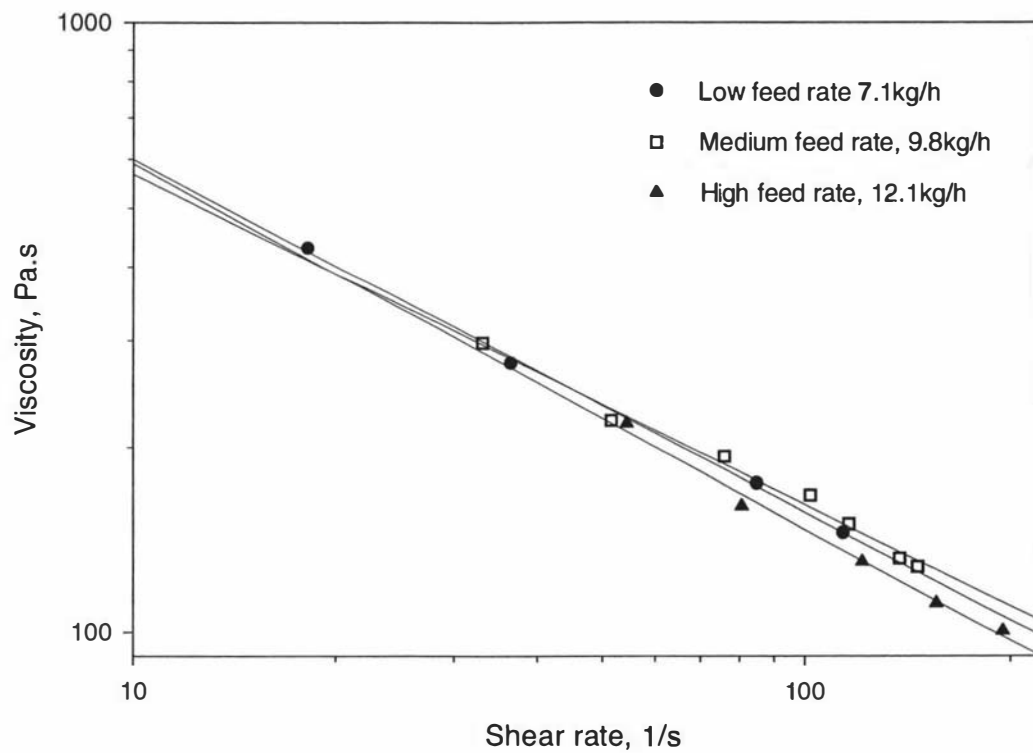
Figure 7.19 The effect of feed rate on the melt viscosity at a constant screw speed 450rpm.

7.2.3.3 Effect of Degree of Fill

It has been reported by van Lengerich (1990) that by keeping degree of fill constant and key operating conditions unchanged, notably die pressure and barrel temperature, the melt is subjected to the same thermo-mechanical treatment during the extrusion process. Therefore, various shear rates and shear stresses in the SDV could be obtained by simultaneously varying screw speed and feed rate. This approach was followed by van Lengerich to measure the viscosity of the melt on-line minimising the interaction between the extruder operation and the measurement system. An experiment was designed to verify if the assumption is correct. In this experiment, the screw speed and feed rate were simultaneously varied to keep the degree of fill constant. Table 7.8 summarises the conditions used.

The experiment consisted of two steps. In the first step, the viscosity was measured using van Lengerich's approach with the valve A of the new SDV fully open and the valve B fully shut (Chapter 6, Figure 6.4). Each feed rate determined a different shear rate in the SDV. Thus, the combination of four different feed rates and screw speeds yielded four different shear rates. The measured viscosity of the melt plotted as a function of shear rate is shown as a solid line in Figure 7.20.

In the second step, the viscosity of the melt was measured using the new SDV and the procedures developed in this study. For each feed rate and its corresponding screw speed (Table 7.8), the shear stress was measured at various shear rates by adjusting the openings of valves A and B. Measured viscosities are plotted as dotted lines in Figure 7.20. As indicated in the figure, there is a close agreement between the results obtained using the new SDV and those obtained from van Lengerich's approach.

It must be noted that it took approximately 2 hours to complete four viscosity measurements using the van Lengerich's approach. However, a similar set carried out using the new SDV took less than 50 minutes. Thus, the use of the new SDV developed in this research was more efficient and required less raw material.

The degrees of starch gelatinisation of the melts for the four operating conditions did not follow a particular trend and appear to be similar (85.9% to 89.6%, Table 7.8).

These data along with those shown in Figure 7.20 indicate that with a constant degree of fill, the melts produced had similar properties.

This experiment shows that the degree of starch gelatinisation and the melt viscosity remained unchanged whereas SME and torque increased with simultaneous increase of screw speed and feed rate (Figures 7.21 and 7.22). According to the definition of SME (Equation 6.6), the value of SME at constant degree of fill is proportional to the torque. Thus, increases in torque result in proportional increases of SME. This increase in SME, however, is accompanied by a decrease in the thermal energy input because by increasing screw speed the residence time decreases. As a consequence of the shortened residence time, the extrudate receives less thermal energy at a constant barrel temperature. The opposite changes on the melt produced by the mechanical and thermal energy could explain the small variations in melt viscosity and starch gelatinisation.

SME was used by many researchers (Kirby *et al.,* 1988; Senouci and Smith, 1988a; van Lengerich, 1990; Sokhey *et al.,* 1994) as an indication of the thermo-mechanical treatment undergone by the material during extrusion. It has also been reported that SME could be used to control the extrusion process (Forte, Personal communication, 1998). However, the results of the three experiments reported in this section clearly show that SME alone should not be used to predict the degree of thermo-mechanical modification of the extrudate melt. The thermal energy input should also be considered, in particular when the residence time distribution is varied. The thermo-mechanical treatment of the melt during the process is better quantified by parameters such as torque, die pressure and apparent viscosity rather than SME alone. Caution must be exercised in using SME as the key and only parameter to control the extrusion process.

Table 7.8 Screw speeds and feed rates used and corresponding rheological properties and degree of starch gelatinisation of the melt.

Settings	Viscosity		Degree of starch gelatinisation, %
	Power law index, n	Power law consistency, K	
Screw=200rpm, Feed=3.5kg/h, Water=1.0 l/h	0.38	3005.1	89.6 ± 0.9
Screw=300rpm, Feed=5.4 kg/h, Wr=1.5 l/h	0.41	2718.4	86.8 ± 2.2
Screw=400rpm, Feed=7.2 kg/h, Water=2.0 l/h	0.37	3106.0	89.1 ± 1.4
Screw=500rpm, Feed=9.3 kg/h, Water=2.5 l/h	0.31	3953.2	85.9 ± 0.8
van Lengerich's approach	0.38	2989.8	
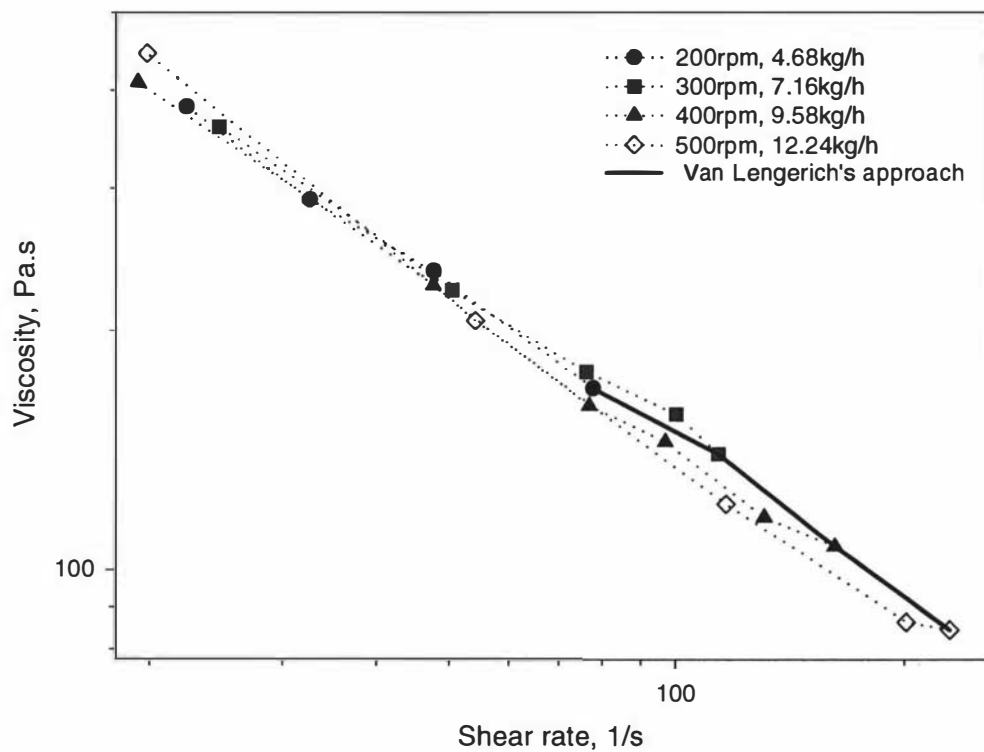
Figure 7.20 The effect of constant degree of fill on the melt viscosity, measured by the new SDV and by van Lengerich's approach.
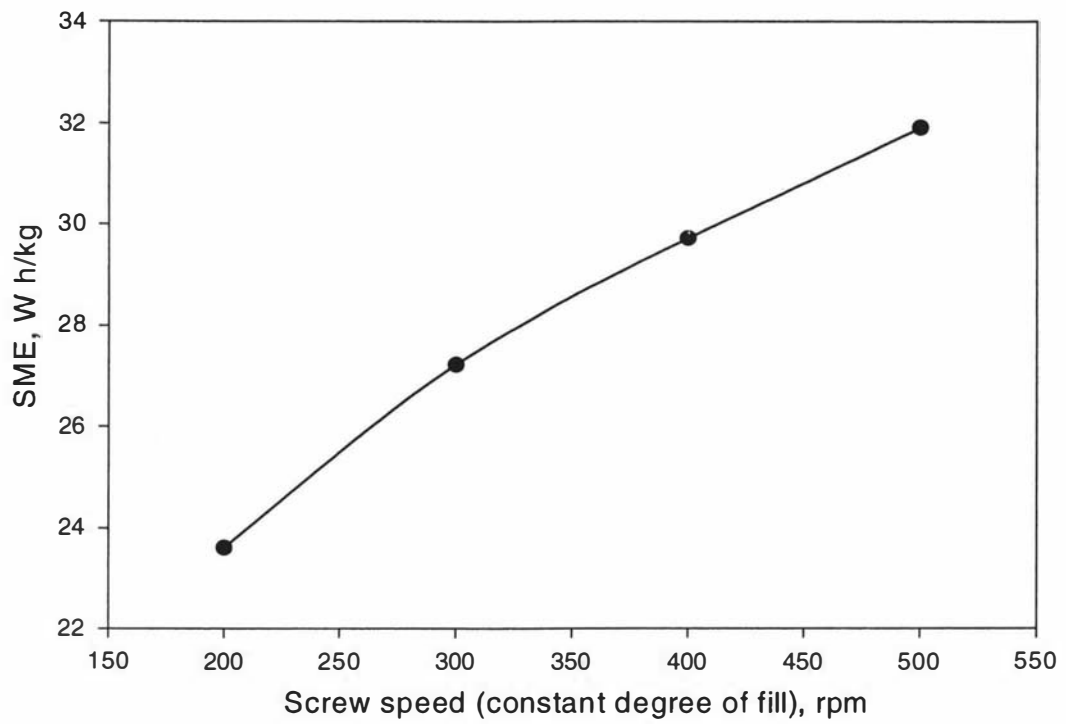
Figure 7.21 The effect of screw speed on SME at constant degree of fill.
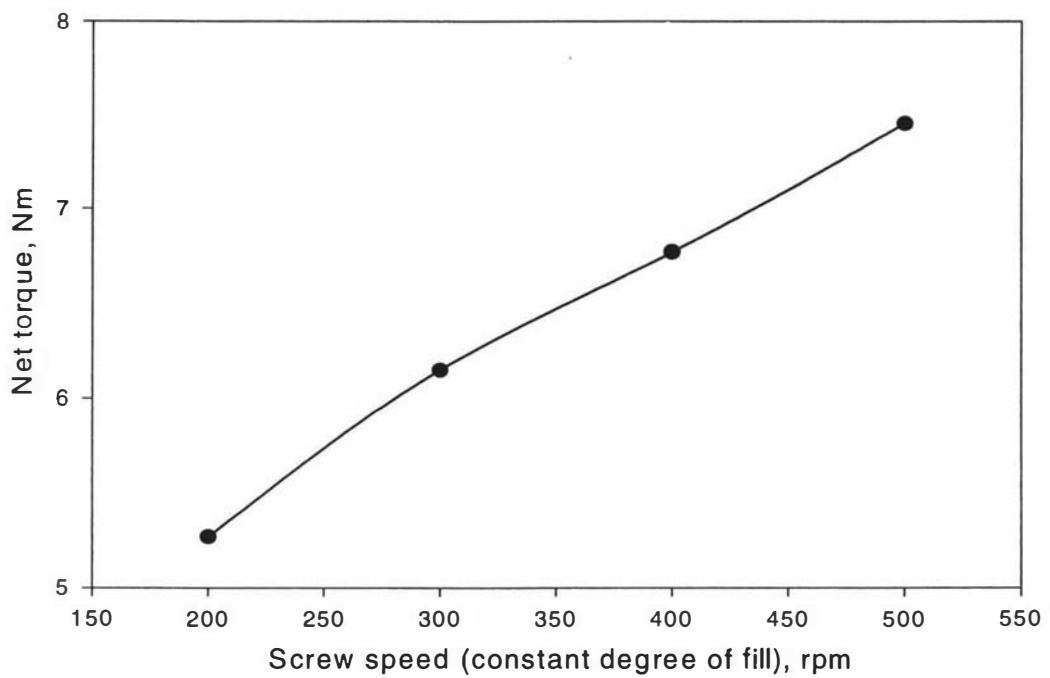
Figure 7.22 The effect of screw speed on torque at constant degree of fill.

7.3 Effect of the Measurement Temperature

The effect of the SDV temperature on the rheological properties and degree of starch gelatinisation of the melts was studied in this experiment.

The temperature of the SDV was set at four different temperatures 110°C, 120°C, 130°C, and 140°C. All other operating conditions were kept constant. The extruder barrels were set to temperatures of 60°C, 90°C, 110°C, 110°C for barrels #1 to #4 respectively. Normal grits were fed into the extruder at 7.5kg/h. Screw speed was set at 450rpm and the moisture content of the melt was 35% wwb.

The SDV and extruder were allowed to reach thermal equilibrium and steady state operating conditions before rheological measurements were taken. Because the viscosity of the melt varied due to changes in temperature, the die pressure and SME were different for experiments at different selected temperatures.

Figure 7.23 shows a plot of the melt viscosity as a function of shear rate for the four temperatures used. As indicated in the figure, the viscosity of the melt decreased when the SDV temperature increased from 110°C to 140°C. Table 7.9 shows that SME, torque, die pressure and thrust pressure all decreased as the temperature of the SDV increased.

The degree of starch gelatinisation of the melt sampled before the SDV decreased slightly from 81.8% to 77.1% when the SDV temperature increased from 110°C to 140°C (Table 7.10). Further starch gelatinisation occurred as the melt passed through the SDV. At the highest SDV temperature used (140°C), the melt sampled before the SDV showed the lowest degree of starch gelatinisation while samples collected at the exit of the SDV showed the highest.

These results indicate that the thermo-mechanical treatments of the melt varied with the SDV temperature as the new SDV constitutes a resistance to the melt flow during extrusion. At higher SDV temperatures, the lower viscosity of the melt resulted in a lower resistance to the melt flow, therefore, the die pressure and residence time decreased resulting in a lower degree of starch gelatinisation.

The results indicate that die temperature is a very important parameter in food extrusion. In commercial extrusion a shaping die is used in the place of the SDV. Variation in the temperature of the die affects melt viscosity and in turn causes variation in the extrusion and expansion processes resulting in large variation in product quality.
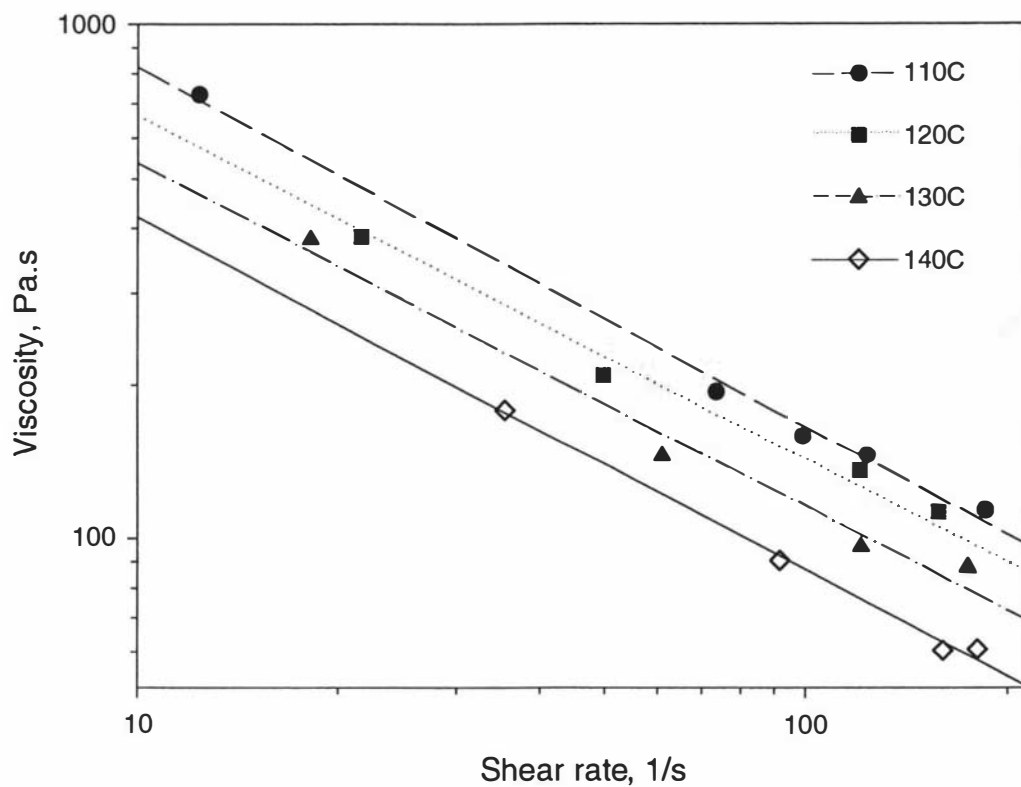
Figure 7.23 The effect of SDV temperature on the rheological properties of the melt.

Table 7.9 The effect of SDV temperature on the operating conditions of the extruder.

Temperature of SDV, °C	SME	Torque, Nm	P_{die}, MPa	P_{thrust}, MPa
110	37.5	14.7	6.1	5.5
120	33.9	13.9	5.6	5.0
130	25.2	12.2	4.8	4.4
140	19.9	11.1	3.6	3.3

Table 7.10 The effect of SDV temperature on degree of starch gelatinisation.

Temperature of SDV, °C	Before SDV	After SDV	% increased in the SDV
110	81.8 ± 2.4	84.9 ± 1.7	+3.1
120	81.5 ± 0.8	87.6 ± 1.7	+6.1
130	80.9 ± 0.8	92.1 ± 1.3	+11.2
140	77.1 ± 2.8	94.3 ± 1.2	+17.2

7.4 Effect of the Raw Material Properties

Manufacturers of extruded snack foods carefully specify the properties of the raw materials including grit size, oil, starch, protein and moisture contents. Variation in these variables can have large effects on the properties of the extrudates (Frame, 1994).

7.4.1 Amylose/Amylopectin Ratio

Two different commercial starches and a 1:1 mixture of both were used to investigate the effect of amylose/amylopectin ratio on the resulting rheological properties of the melt. The raw materials were:

i)   A high amylopectin starch, *Colflo67*, containing 99% amylopectin and 1% amylose.

ii) A high amylose starch, *Crispfilm*, containing 45% amylopectin and 55% amylose.

iii) A mixture of 50% *Crispfilm* and 50% *Colflo67* containing 72% amylopectin and 28% amylose.

For this experiment, the temperatures of the four extruder barrel sections were maintained at 60°C, 90°C, 120°C and 120°C. Screw speed was constant at 400rpm. Moisture content of the melt was 35% wwb and the SDV temperature 120°C. The feed rate for *Colflo67* was 4.88kg/h. The feed rate for *Crispfilm* had to be reduced to 2.77kg/h to keep the torque below the extruder shut-down limit, as the melt of the *Crispfilm* starch was far more viscous than that of the *Colflo67* starch. The feed rate for the mixture was 4.46kg/h.

Starch with a higher amylopectin content produced a melt with a lower viscosity than that with high amylose content (Figure 7.24). The amylopectin-rich *Colflo67* showed the lowest viscosity with the lowest power law consistency K (Table 7.11). The melt of *Colflo67* had a high power law index n (0.82) indicating that it had less shear thinning behaviour. Because the viscosity decreased with the increasing amount of amylopectin, the SME also decreased largely from 129.4 W·h/kg for *Crispfilm* to 40.9 W·h/kg for *Colflo67*.

The amylopectin-rich starch *Colflo67* exhibited a higher degree of starch gelatinisation than the amylose-rich starch *Crispfilm* (Table 7.12). The low SME and high degree of starch gelatinisation (96.5%) of *Colflo67* suggest that amylopectin is more easily gelatinised. The high viscosity, high SME (129.4 W·h/kg) and a relatively low (84.1%) degree of starch gelatinisation of *Crispfilm* indicated that the amylose component has a proportionally large effect on controlling the rheological properties of the melt and is more resistant to starch gelatinisation than amylopectin. This is further demonstrated by the plot of melt viscosity versus shear rate for the mixture showing that its rheological properties are close to the properties of *Crispfilm* (Figure 7.24).
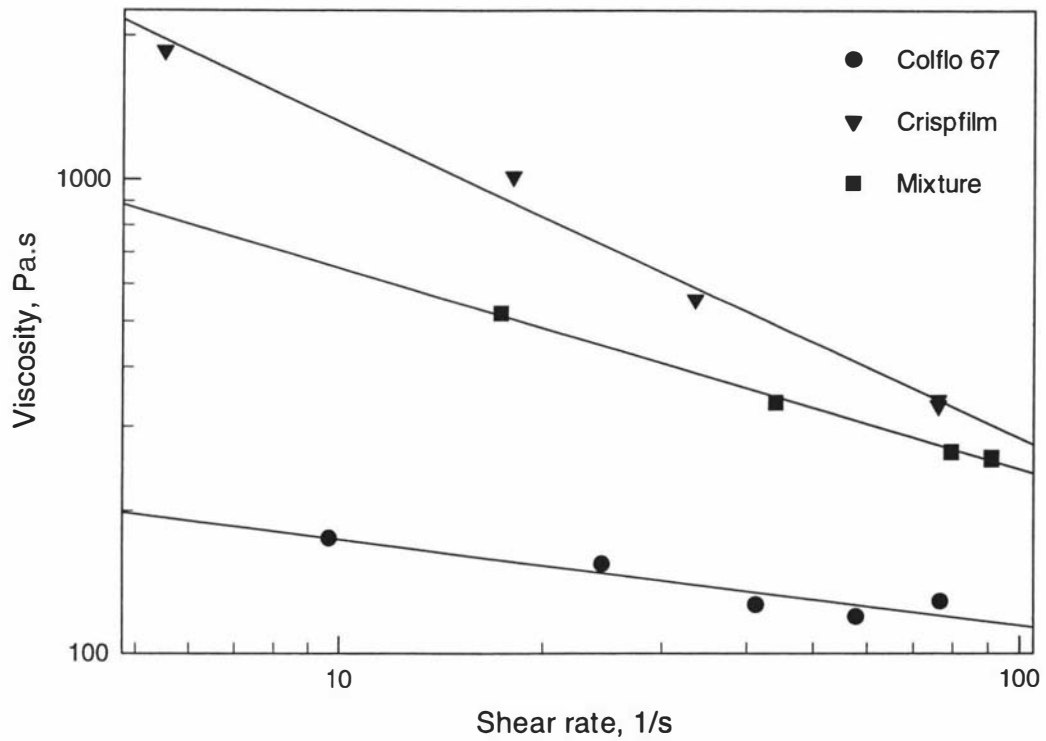
Figure 7.24 The effect of amylose/amylopectin ratio on the melt viscosity.

Table 7.11 The effect of amylose/amylopectin ratio on the rheological properties of the melt and SME.

	K	n	SME
Colflo67, 99% amylopectin + 1% amylose	279.3	0.82	40.9
Mixture: 72% amylopectin + 28% amylose	1943.8	0.58	86.7
Crispfilm, 45% amylopectin + 55% amylose	7262.6	0.33	129.4

Table 7.12 The effect of amylose/amylopectin ratio on the degree of starch gelatinisation.

	Before SDV %
Colflo67, 99% amylopectin + 1% amylose	96.5 ± 6.2
Mixture: 72% amylopectin + 28% amylose	84.2 ± 4.5
Crispfilm, 45% amylopectin + 55% amylose	84.1 ± 3.2

7.4.2 Grit Size

To generate a melt, raw materials need to be converted from a particulate structure to an amorphous molten phase. Thus, it has been considered that the grit size could have an important influence in this transition. The purpose of this trial was to investigate the effect of grit size on the rheological properties of the melt. Corn grits from the same hybrid with three different particle size distributions were used. They, based in its mean particle size, are designated *coarse*, *medium*, and *fine* grits.

A screw speed of 400rpm and a feed rate of about 7.9kg/h were used. The temperatures of the four barrels sections were maintained constant at 60°C, 90°C, 120°C, and 120°C. The SDV temperature was set at 120°C. Moisture content was set at 35% wwb.

Figure 7.25 shows that melts produced from fine grits had higher viscosities than those of melts produced from other grits. Table 7.13 shows that the fine grits had higher values of SME, torque, thrust pressure and die pressure whereas these values were similar for the other grit sizes. The value of the power law index n increased with grit size whereas the values of the consistency K was almost constant. These results indicate that the melt produced with the coarse grits had a higher shear thinning behaviour than that produced with the fine grits. Values of apparent viscosity calculated at a shear rate of 100 1/s decreased when the grit size increased.

The degree of gelatinisation of the extrudate collected before and after the slit die are shown in Table 7.14. Fine grits were almost completely gelatinised before passing through the SDV but gelatinisation of the medium and coarse grits was incomplete. After passing through the SDV, the degree of starch gelatinisation increased slightly for the medium and coarse grits.

The lower degree of starch gelatinisation in the melt produced with the coarse and medium grits indicates that the material did not receive a complete thermo-mechanical treatment during extrusion. To fully develop these melts, the structure of the grits needs to be completely degraded by the shear action and heat. At higher screw speeds, greater shear forces are developed resulting in greater thermo-mechanical modification. In twin screw extrusion and depending on the screw configuration, shear forces are usually very

high with considerable mixing and back flow. When large size grits are used, particularly at high moisture content, a portion of grits remain unaffected resulting in a lower degree of starch gelatinisation and lower viscosity of the melt.
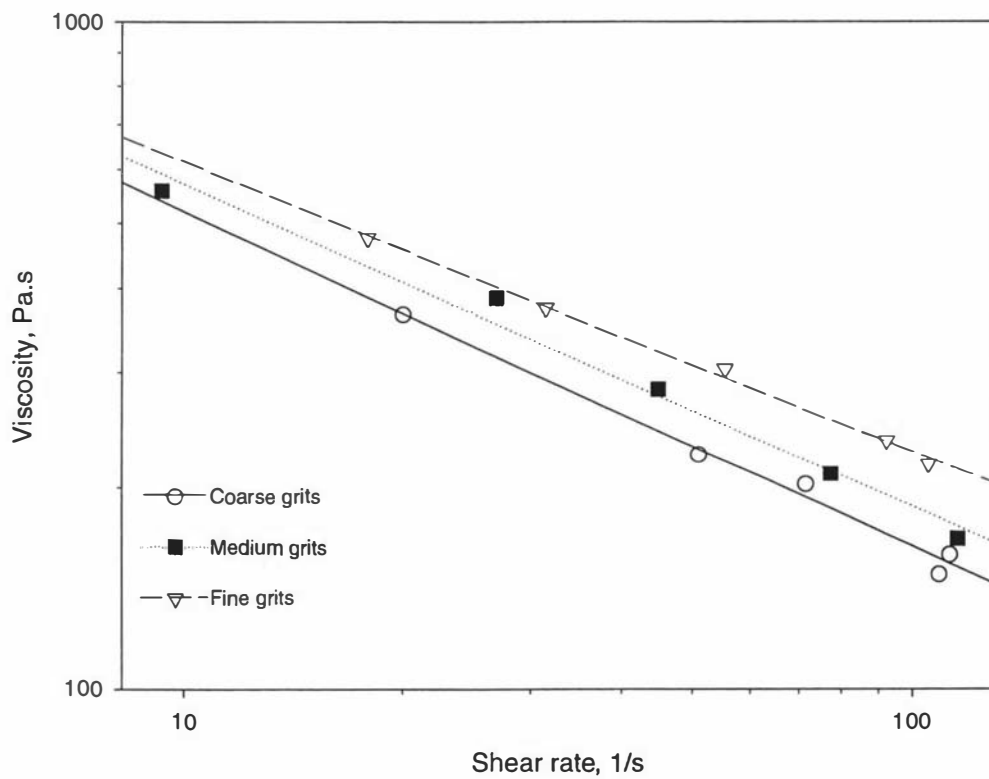
Figure 7.25 Viscosity of the melt produced from the grits of three sizes at 400rpm screw speed.

Table 7.13 Operating conditions for the three grits extruded.

Particle size range	n	K Pa.s^n	Viscosity Calculated at 100 1/s	SME W-h/kg	Torque, N-m	P_{thrust} MPa	P_{die} MPa	Throughput kg/h
Fine	0.57	1899.6	262.2	55.8	10.4	6.0	7.1	7.8
Medium	0.52	2002.2	219.5	35.2	6.8	4.9	4.9	8.1
Coarse	0.50	1910.6	191.1	36.3	6.6	4.9	4.6	7.7

Table 7.14 The effect of grit size on the degree of starch gelatinisation.

Grit Size	Before SDV, %	After SDV, %
Fine	99.3 ± 1.3	99.9 ± 0.6
Medium	91.5 ± 1.0	94.8 ± 1.3
Coarse	92.6 ± 2.4	90.2 ± 1.7

7.4.3 Effect of Hybrid

The effect of hybrid on the extrusion process was studied by using corn grits from 11 hybrids harvested in the 92-93 season and 17 hybrids harvested in the 94-95 season. The hybrids were carefully selected among those described in Chapters 3 and 4 in order to have a wide range of measured grain quality attributes such as SHT hardness and bulk density. The grits from the 92-93 season which were produced by grinding whole grains had oil contents (2.2-3.8%). The grits from the 94-95 season were produced by grinding degermed grains and the oil content of these grits was about 1.1%.

The temperature of the four extruder barrels were kept constant at 60°C, 90°C, 120°C, and 120°C. Moisture content was 35% wwb. The temperature of the SDV was set at 120°C and screw speed at 400rpm. Feed rate was set at 5.2kg/h for all tests.

The melts produced from hybrids grown in the same season showed similar rheological properties (Tables 7.15 and 7.16). However, the degree of starch gelatinisation of the melt sampled before the SDV was different among the hybrids, in particular, the two hybrids P3394 and P3585 were more easily gelatinised than P3751 and PF2xNZ56. P3585 and P3751 have a medium to soft endosperm type while that of PF2xNZ56 is hard. This suggests that the ease of gelatinisation during extrusion is not simply related to endosperm type.

For the 92-93 season grits, there was a moderate variation in the values of K and n but as shown later they were not linked to grain quality attributes. For the 94-95 season grits, similar moderate variations were found in all the parameters measured.

The experiments, however, showed important differences between seasons in SME and rheological properties of melts. Tables 7.15 and 7.16 show that grits from hybrids of the 94-95 season produced melts with a higher n and a lower K. In rheological terms, this means that melts from the 94-95 season were more Newtonian and less viscous than melts from the 92-93 season. Lower SME was also observed for grits of the 94-95 season. As shown in Table 7.17 Analysis of Variance (ANOVA) reveals that melts produced from grits of the two seasons had a similar degree of starch gelatinisation but significantly different SMEs and rheological properties. This difference could be

attributed to the two different methods used in the preparation of grits of the different seasons (see Chapter 6). Grits from the 92-93 season were produced from whole corn grains without degerming as a suitable degermer was not available at that time. Grits from the 94-95 season were produced from degermed grain. Thus, grits from the 92-93 season contained a higher proportion of pericarp (fibre) and oil than those of the 94-95 season. The high level of fibre increased the viscosity of the melt and thus SME. The higher oil content of the grits could also favour the production of amylose-lipid complexes with a resultant increase in viscosity (Mercier *et al.*, 1980; Launay, *et al.*, 1983).

Although variations in the rheological properties of the melt were relatively moderate within each season, it is of interest to see how these variables were affected by the grain quality. It is worth noting that the rheological behaviours are in reality means generated from many data points acquired as the melt passes through the SDV. This suggests that the COV values presented in Tables 7.15 and 7.16 may be overestimated and therefore differences among the hybrids could be more significant. For that reason a correlation matrix has been calculated for the rheological parameters presented in Tables 7.15 and 7.16 and the grain quality parameters discussed in Chapter 4. Tables 7.18 and 7.19 list the linear correlation coefficients between rheological and grain quality parameters for each season. R values greater than 0.60 are considered significant at 0.05 level of significance.

Since the objective of the section was to consider the effect of grain quality on rheological properties of the melt and extrusion operational parameters, the discussion will focus only on the correlation between these two groups of variables (un-shaded zones in tables 7.18 and 7.19). As discussed in Chapter 4, all the grain quality parameters measure grain hardness and are highly correlated. From correlation between the three grain hardness parameters for the 92-93 season and the extrusion parameters including degree of starch gelatinisation (Table 7.18), it can be seen that grain hardness is negatively correlated with the degree of starch gelatinisation.

Comparison of the 92-93 season and the 94-95 season correlations between grain quality, rheological parameters and the degree of starch gelatinisation show a very different pattern. Both bulk density (BD) and SHT milling energy (E) are positively

correlated with die pressure and thrust pressure. No other correlation is significant between other parameters.

The very large differences in the correlated variables over the two seasons provide further evidence for the importance of grain components including starch, fibre and proteins in determining the rheological properties of the melt during extrusion.

Tables 7.18 and 7.19 also show that the correlation coefficients between n (or K) and grain bulk density, grain hardness and H/S ratio are all less than 0.43. Therefore, these results agree with the findings drawn from Tables 7.16 and 7.17 that the effect of hybrids on the rheological properties of the melt was negligible. It is known that variation in raw materials causes large variations in product quality during the production of snack foods using single screw extruders (personal communication with Bluebird Foods Ltd. NZ). In this work, however, no such variation was found. The versatility of twin screw extruders and their capacity to handle materials of different properties could be the reason for the small effect of hybrids on the extrusion process. The strong shear and mixing action provided by the twin screw rotation may overpower the effect of differences in properties of the grits such as hardness and bulk density.

Table 7.15 The rheological properties, SME, and degree of starch gelatinisation for 11 hybrids from the 92-93 season.

Hybrid Name	Power law consistency K	Power law index n	Apparent viscosity at 100 1/s	SME	Before SDV, %	After SDV, %
PF1xAS3-94	8339.3	0.20	218	47.7	92.8 ± 1.2	97.4 ± 2.5
PF2xNZ56	7012.5	0.26	213	47.1	85.8 ± 1.2	91.1 ± 7.6
PF1xBS22-39	5854.0	0.29	224	49.1	90.0 ± 5.0	93.3 ± 3.4
PF1xNZS1-141	7189.5	0.24	205	46.7	89.0 ± 1.4	94.6 ± 1.1
D1275xM378-80	7291.9	0.24	214	48.0	94.5 ± 1.8	94.9 ± 0.4
N190xNZ2	7757.5	0.23	229	55.9	93.7 ± 2.5	95.5 ± 1.9
WGI207xM378-80	9602.6	0.21	257	52.3	93.3 ± 1.7	95.2 ± 2.1
A82-8xMBS847	6906.1	0.25	219	49.7	90.3 ± 4.2	91.3 ± 1.9
P3751	6566.6	0.28	237	56.5	88.9 ± 3.4	95.4 ± 4.4
P3394	5733.0	0.30	232	51.1	97.4 ± 1.8	94.5 ± 4.3
P3585	6608.1	0.27	227	48.0	95.6 ± 1.9	96.6 ± 2.2
Mean	7179	0.25	225	49.9	91.9	94.5
COV, %	15.4	12.3	6.3	7.7	3.7	2.1

Table 7.16 The rheological properties, SME, and degree of starch gelatinisation for 17 hybrids from the 94-95 season.

Hybrid names	Power law consistency K	Power law index n	Apparent viscosity at 100 1/s	SME	Before SDV, %	After SDV, %
P3751	2468.0	0.44	187	38.4	96.7 ± 3.4	97.3 ± 4.0
FURIO	2267.6	0.47	198	35.3	97.9 ± 1.3	99.4 ± 0.3
NZS3-523-1-1-1 × T1	3376.6	0.37	188	37.2	93.2 ± 3.8	93.4 ± 1.9
NZ84 × Mo17Ht	3208.1	0.38	181	35.8	91.5 ± 3.3	92.4 ± 1.4
Hmv-124-2 × T1	3341.9	0.37	183	35.3	96.6 ± 3.1	95.7 ± 1.6
A665 × H99	3460.1	0.36	185	36.0	93.0 ± 3.5	92.3 ± 1.4
NZ45 × T1	2871.5	0.40	181	36.8	92.0 ± 2.9	96.0 ± 2.4
M396-14-1-1-1-1-1 × A665	2183.5	0.48	195	33.1	95.3 ± 2.5	94.2 ± 3.0
157-R-3-9-1 ×A82-8	2512.3	0.43	186	35.3	94.7 ± 4.0	94.8 ± 3.9
N192 × Mo17Ht	2358.2	0.44	182	34.0	93.7 ± 6.0	93.6 ± 7.1
NZ84 × A82-8	2822.8	0.41	186	34.6	93.6 ± 3.8	93.6 ± 4.2
NZ84 × E1386	2539.1	0.44	190	35.3	88.9 ± 6.2	92.6 ± 2.7
A665 × NZ77	3608.4	0.34	173	33.6	91.5 ± 3.8	93.1 ± 2.7
D1260 × NZ84	2868.1	0.39	173	36.5	97.5 ± 1.4	98.6 ± 1.4
A82-8 × NZ43	2581.8	0.40	163	33.4	96.3 ± 1.7	95.0 ± 0.2
E1386 × NZ84B	2735.0	0.41	181	34.5	97.3 ± 1.3	97.9 ± 2.4
Mo17Ht ×NZ84	2582.8	0.41	171	32.9	95.6 ± 1.8	96.9 ± 1.6
Mean	*2810*	*0.41*	*183*	*35.18*	*94.4*	*95.1*
COV, %	*15.7*	*9.3*	*4.9*	*4.4*	*2.7*	*2.4*

Table 7.17 The ANOVA test for samples from the 92-93 and 94-95 seasons. Critical F value at 0.05 level of significance is 4.4.

	Power law consistency K	Power law index n	Apparent viscosity at 100 1/s	SME	Before SDV %	After SDV %
Computed F values	145.5	112.5	69.4	131.4	4.1	0.1

Table 7.18 Correlation coefficients between operating variables, rheological properties, degree of starch gelatinisation, and grain quality of the 11 hybrids from the 92-93 season.

	BD	E	H/S	n	K	SME	NetTorq	P_{die}	P_{thrust}	Gel%
BD	1.00									
E	0.92	1.00								
H/S	0.90	0.92	1.00							
n	-0.43	-0.36	-0.28	1.00						
K	0.20	0.12	0.02	-0.88	1.00					
SME	-0.42	-0.45	-0.45	0.24	0.09	1.00				
NetTorq	-0.45	-0.49	-0.50	0.26	0.09	1.00	1.00			
P_{die}	-0.39	-0.38	-0.43	0.33	0.01	0.91	0.92	1.00		
P_{thrust}	-0.40	-0.38	-0.46	0.28	0.04	0.94	0.95	0.98	1.00	
Gel%	-0.38	-0.63	-0.50	0.22	0.03	0.27	0.30	0.08	0.07	1.00

Note: BD: Bulk density
        E   Milling energy
        H/S   Ratio of hard to soft endosperm
        Visc   Viscosity calculated at shear rate of 100 1/s
        n Power law flow index
        K Power law consistency
        SME   Specific Mechanical Energy
        NetTorq   Torque
        P_{die} Die pressure
        P_{thrust} Screw thrust pressure
        Gel%   Degree of starch gelatinisation before the SDV

Table 7.19 Correlation coefficients between operating variables, rheological properties, degree of starch gelatinisation, and grain quality of the 17 hybrids from the 94-95 season.

	BD	E	n	K	SME	NetTorq	P_{die}	P_{thrust}	Gel%
BD	1.00								
E	0.63	1.00							
n	0.04	0.02	1.00						
K	-0.00	0.05	-0.99	1.00					
SME	-0.04	0.10	-0.41	0.40	1.00				
NetTorq	0.26	0.38	-0.22	0.24	0.83	1.00			
P_{die}	0.68	0.73	-0.28	0.32	0.56	0.82	1.00		
P_{thrust}	0.74	0.73	-0.18	0.22	0.45	0.75	0.98	1.00	
Gel%	-0.35	-0.27	0.33	-0.27	-0.00	-0.09	-0.38	-0.47	1.00

Note: BD: Bulk density
         E   Milling energy
         Visc   Viscosity calculated at shear rate of 100 1/s
         n   Power law flow index
         K   Power law consistency
         SME   Specific Mechanical Energy
         NetTorq   Torque
         P_{die} Die pressure
         P_{thrust} Screw thrust pressure
         Gel%   Degree of starch gelatinisation before the SDV

7.5 Summary

The new Slit-Die-Viscometer was useful for measuring the viscosity of the melt on line. The experimental procedure set for operating the SDV was simple and efficient.

One of the advantages of using the new SDV is that the measurement becomes independent of the extruder operation. For each of the conditions under which the extruder is operated, the rheological measurements represent a true flow curve of the melt because variations in the product thermo-mechanical treatment are minimised during the measurements.

The properties of the melt depend on its thermo-mechanical history which in turn is related to the operating conditions of the extruder and the raw materials.

The melt produced by extrusion of corn grits follows a power law rheological behaviour. The viscosity of the melt decreased with increasing moisture content. SME, torque and die pressure also decreased with moisture content.

The degree of starch gelatinisation increased when the barrel temperature increased from 90°C to 130°C. For temperatures higher than 130°C, most of the starch was gelatinised. The increase in barrel temperature, however, resulted in small changes in the apparent viscosity of the melt with a maximum around 130°C.

At constant feed rate, SME increased and torque decreased when screw speed increased due to the shear thinning behaviour of the melt.

At constant screw speed, the torque increased and SME decreased with increasing feed rate. The decrease in SME was due to the change in rheological properties of the melt resulting in a decrease in apparent viscosity at the high feed rate.

The apparent viscosity of the melt did not change significantly when the screw speed was varied at constant feed rate or vice versa. Melt viscosity measured using the new SDV agreed with the results obtained using the van Lengerich's approach, which uses a

conventional SDV, provided the ratio between the feed rate and screw speed is maintained constant.

A higher degree of fill always corresponded to a lower SME. However, SME should be used with caution either to predict the degree of thermo-mechanical treatment of the product or as the key and only variable for process control.

The apparent viscosity of the melt decreased considerably when the SDV temperature increased from 110°C to 140°C. The temperature of the SDV affects not only the rheological measurements but also the extruder operating conditions, indicating that the die temperature is a very important factor in the performance of the food extrusion process.

Starch with a high amylopectin content produced a melt with a lower viscosity than that produced with a high amylose starch. Amylose is a key component in controlling the rheological properties of the melt and is resistant to starch degradation.

Melts produced with fine grits had higher viscosities than those produced with medium and coarse grits at a screw speed of 400rpm. Fine grits required more mechanical energy input (larger SME value) and produced higher die and thrust pressures than medium and larger grits. Gelatinisation of fine grits extruded at screw speed of 400 rpm was almost complete.

The effect of hybrids of the same season on the melt rheological properties was negligible. The rheological properties of melts produced from degermed grain from the 94-95 season were less viscous than those produced from whole grain from the 92-93 season.

Chapter 8

Achievements, Overall Conclusions and Recommendations

8.1 Major Achievements

i)   A modified Stenvert Hardness Test (SHT) using milling energy as the key hardness index was developed. The test is now fully computerised providing quick and accurate measurements of grain hardness.

ii)   A roller mill test was developed to estimate the order of the breaking force applied during the roller milling of corn grains.

iii)   A computerised data acquisition system for both the modified SHT and the roller mill test was developed. This system includes a computer program for data acquisition, analysis and reporting.

iv)   The modified SHT along with the data acquisition system is being used as a standard tool in New Zealand to assess corn grain quality for research and commercial applications.

v)   A new on-line Slit-Die-Viscometer (SDV) for extrusion was developed to measure melt viscosity with minimum interaction between the extruder and the measuring system.

vi)   A computerised data acquisition and control system was developed for the operation of the new SDV and the extruder.

8.2 Summary of Conclusions

- The modified Stenvert Hardness Test (SHT) is a quick and simple method that can be used to measure and compare the endosperm hardness of different maize hybrids.

- The simplicity, accuracy and the small amount of sample required for the modified SHT suggest that this test is a convenient tool to carry out routine quality control of corn.

- At similar moisture content, SHT milling energy E can effectively distinguish differences in hardness among hybrids.

- The hard to soft endosperm ratio of maize hybrids were correlated with grain hardness measured by the modified SHT and with bulk density.

- All measurements of kernel hardness should be based on a mechanical milling test such as that described here and not a visual assessment of the ratio of hard to soft endosperm area.

- Corn kernel hardness can be altered by choosing the kernel properties of the hybrid parents.

- Grain moisture content has a large effect on bulk density, hardness, and the yield of grit during milling. Corn grain hardness and milling characteristics should be determined at the same moisture content for all samples.

- The roller milling experiments provided valuable information on the corn dry milling process. Grit recovery and breaking force were related to corn grain hardness.

- The new on-line Slit-Die-Viscometer (SDV) was useful for measuring the viscosity of the melt on line. The experimental procedure for operating the SDV was simple and efficient.

- The measurement of the viscosity using the new SDV becomes independent of the extruder operation. For each of the conditions under which the extruder was operated, the rheological measurements represent a true flow curve of the melt.

- The properties of the melt depend on its thermo-mechanical treatment which in turn is closely related to the operating conditions of the extruder and the characteristics of the raw materials.

- The melt produced by extrusion of corn grit followed a power law rheological behaviour.

- The viscosity of the melt decreased with moisture content. The increase in moisture content also decreased SME, screw torque and die pressure.

- The degree of starch gelatinisation increased when the barrel temperature increased. For temperatures higher than 130°C, most of the starch was fully gelatinised.

- The increase in barrel temperature resulted in small changes in the melt apparent viscosity with a maximum around 130°C.

- At constant feed rate, SME increased and torque decreased when screw speed increased resulting in a decrease of the apparent viscosity of the melt in the extruder. This effect was due to the shear thinning behaviour of the melt when the screw speed increased.

- At constant screw speed, the torque increased and SME decreased with increasing feed rate. The decrease in SME was due to the change in rheological properties of the melt resulting in a decrease in apparent viscosity at the high feed rate.

- A higher degree of fill always corresponded to a lower SME.

- The effect of screw speed at constant feed rate on the melt viscosity was less noticeable.

- Viscosity measured using the new SDV had a good agreement with the results obtained using the van Lengerich's approach.

- SME should be used with caution either to predict the degree of thermo-mechanical treatment of the product or as the only variable for process control.

- The viscosity of the melt decreased when the SDV temperature increased.

- The temperature of the SDV affects not only the rheological measurement but also the extruder operating conditions, indicating that the die temperature is a very important factor in the performance of the food extrusion process.

- Starch with a high amylopectin content produced a melt with a lower viscosity than that produced with a high amylose starch. Amylose is a key component in controlling the rheological properties of the melt and is resistant to starch degradation.

- Melts produced with fine grits had higher viscosities than those produced with medium and coarse grits at a screw speed of 400rpm. Fine grits required more mechanical energy input (larger SME value) and produced higher die and thrust pressures than medium and larger grits. Gelatinisation of fine grits extruded at screw speed of 400 rpm was complete.

- The effect of hybrids from the same season on the rheological properties of the melt was negligible. The rheological properties of melts produced from degermed grain from the 94-95 season were less viscous than those produced from whole grain from the 92-93 season.

8.3 Recommendation for Future Work

The main objectives of this work were (1) the developments of methods to determine physical characteristics of New Zealand corn hybrids and (2) to determine the rheological properties of extrudate melts produced with New Zealand corn grits, (3) the investigation of the effect of the extruder operating conditions and properties of the raw materials on the rheological properties of the melts and (4) the establishment of the correlation between corn grain quality, rheological properties of the melts and starch degradation. As far as the author of this work is concerned, these objectives were achieved. Recommendations for future research based on this work are:

- Studies of the effects of micro structure of corn kernel and endosperm on grain hardness;

- The development of a specialised corn roller mill to investigate the dry milling characteristics of corn grains of different hybrids at different moisture contents.

- The investigation of the effect of size and hardness of grits on the single screw extrusion;

- The establishment of the relationship between the physical and sensory characteristic of the expanded snack product and the rheological properties of the melts;

- The development of quantitative analytical methods to study the effect of thermal-mechanical energy on starch degradation during extrusion and its relationship with the rheological properties of the melts.

- Studies using a pilot scale extruder to determine if results obtained in this work can be applied to larger extruders. This is essential for scale-up and the development of a control system for industrial scale extruders.

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Appendix A

Data Acquisition System for Milling

A data acquisition system was developed for the modified Stenvert Hardness Test and the Roller Milling Test.

A general description of the data acquisition system for the milling tests was given in Chapter 3. However, the key component of the computer program was not discussed in that chapter. This section describes details of the program developed.

The computer program was written using the QuickBasic language. It consists of functions such as data logging, processing, displaying, plotting and storing. Apart from these main functions, the program has many user friendly features such as automatic file name generation, duplicated file name warning, automatic data logging trigger, automatic data logging termination, mill-screen blockage detection and warning, build-in timer for measuring SHT resistance time, data re-plotting, data variation warning, user name recording and background music. The program having more than 3000 lines is too long to be included in this thesis. A flow chart of the program is shown in Figures A1 to A5. A copy of the computer program can be obtained by request to the author of this thesis.
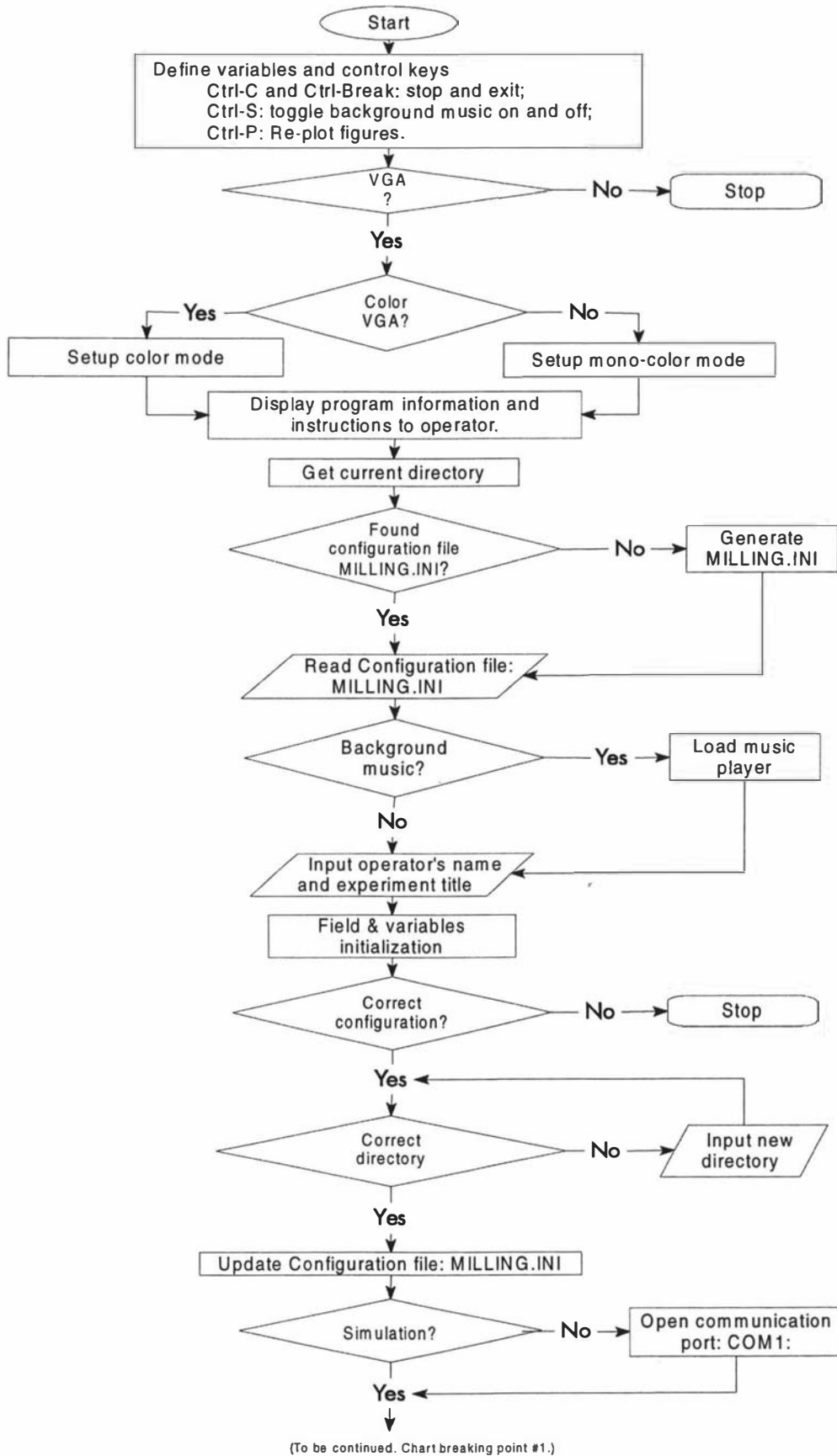
Figure A. 1 Flow chart of the data acquisition program for the milling tests. Part I.

Figure A. 2 Flow chart of the data acquisition program for the milling tests. Part II.

Figure A. 3 Flow chart of the data acquisition program for the milling tests. Part III.
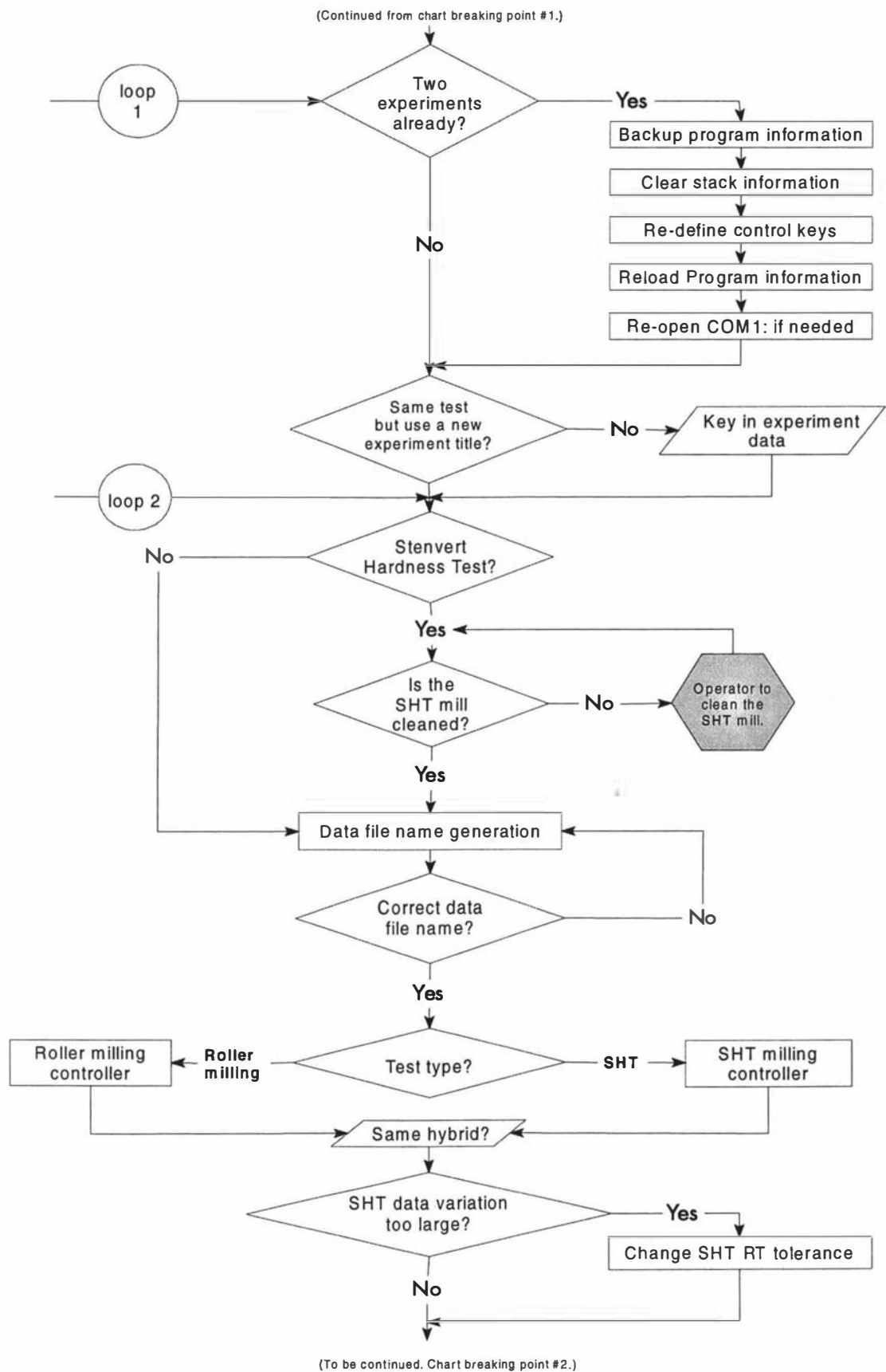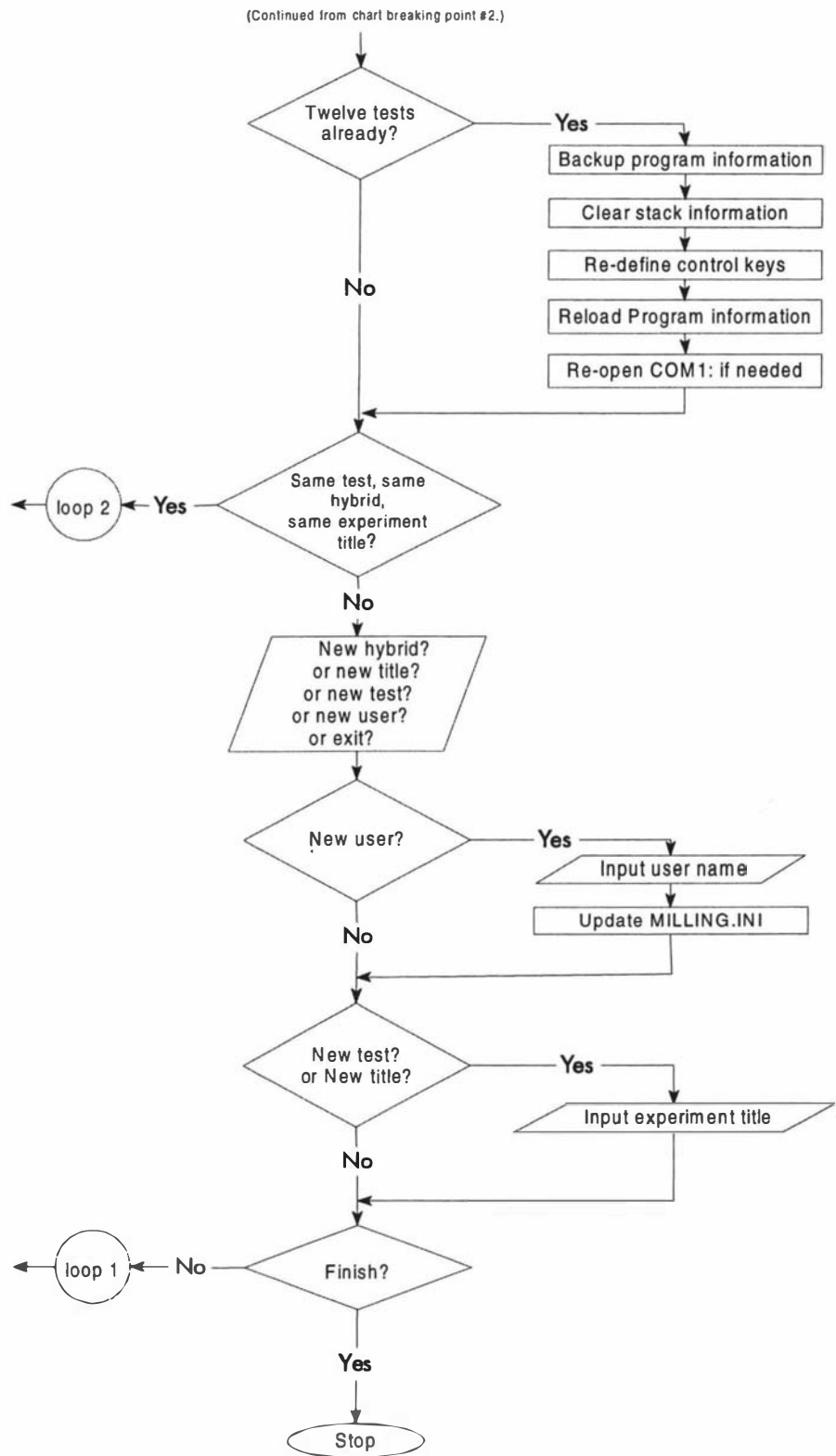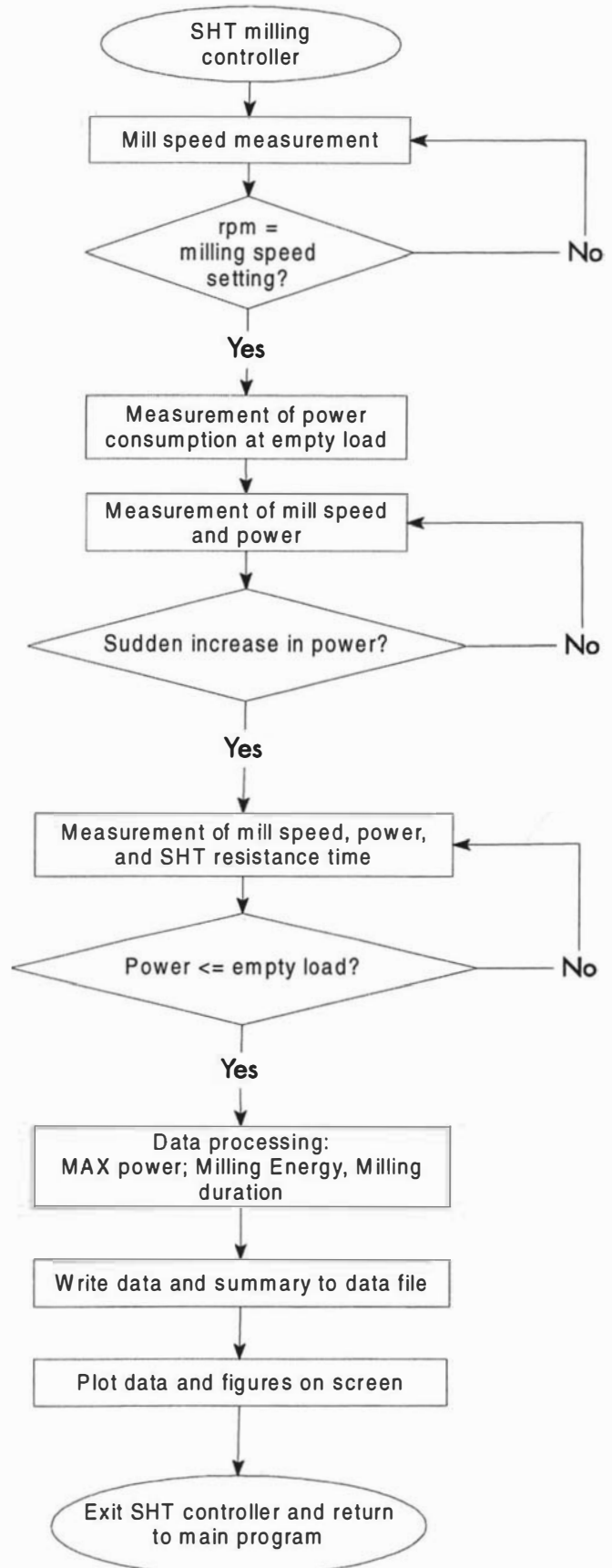
Figure A. 4 Flow chart of the data acquisition program for the milling tests. Part IV.
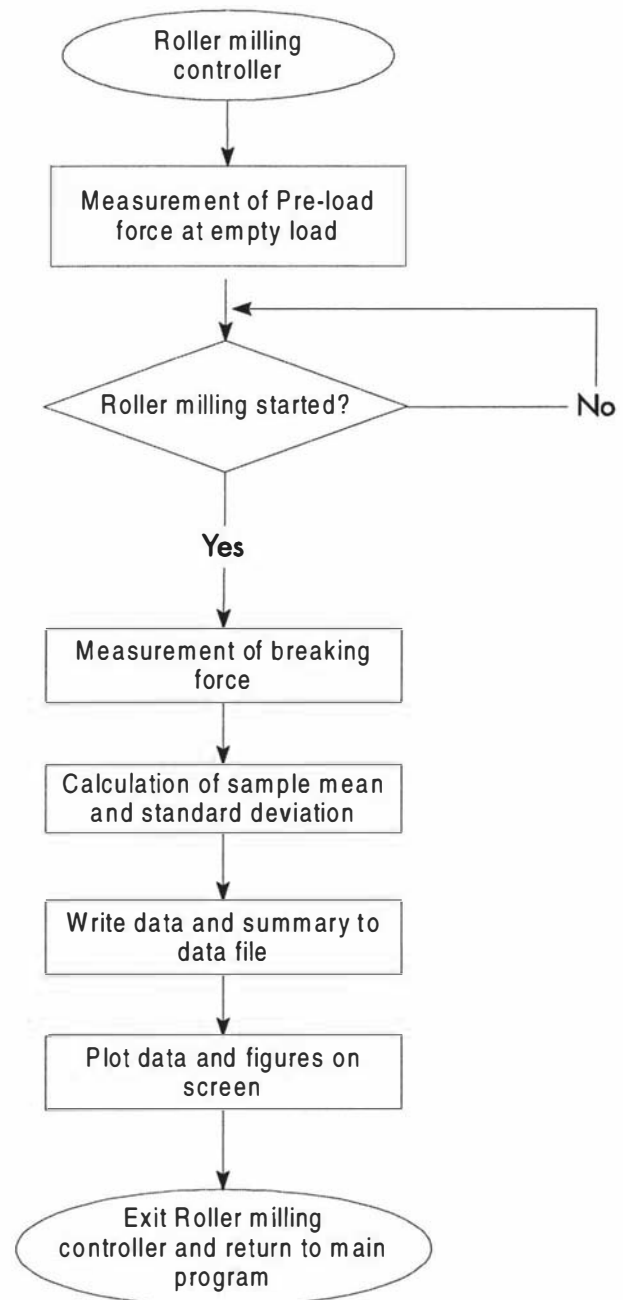
Figure A. 5 Flow chart of the data acquisition program for the milling tests. Part V.

Appendix B

Theory of Slit Viscometry

In order to calculate the melt viscosity the flow rate and pressure profile along the slit in the Slit-Die-Viscometer must be converted to shear rate and shear stress respectively. The formulas for obtaining shear rate and shear stress for SDV can be derived by using a two-dimensional slit flow model as shown below:

The assumptions used to solve the fluid mechanics of this problem are:
- The flow is two dimensional as shown in Figure B1;
- There is no slip on the slit wall;
- The rheological model of the melt can be described as a power law model $\eta = K\dot{\gamma}^{n-1}$.
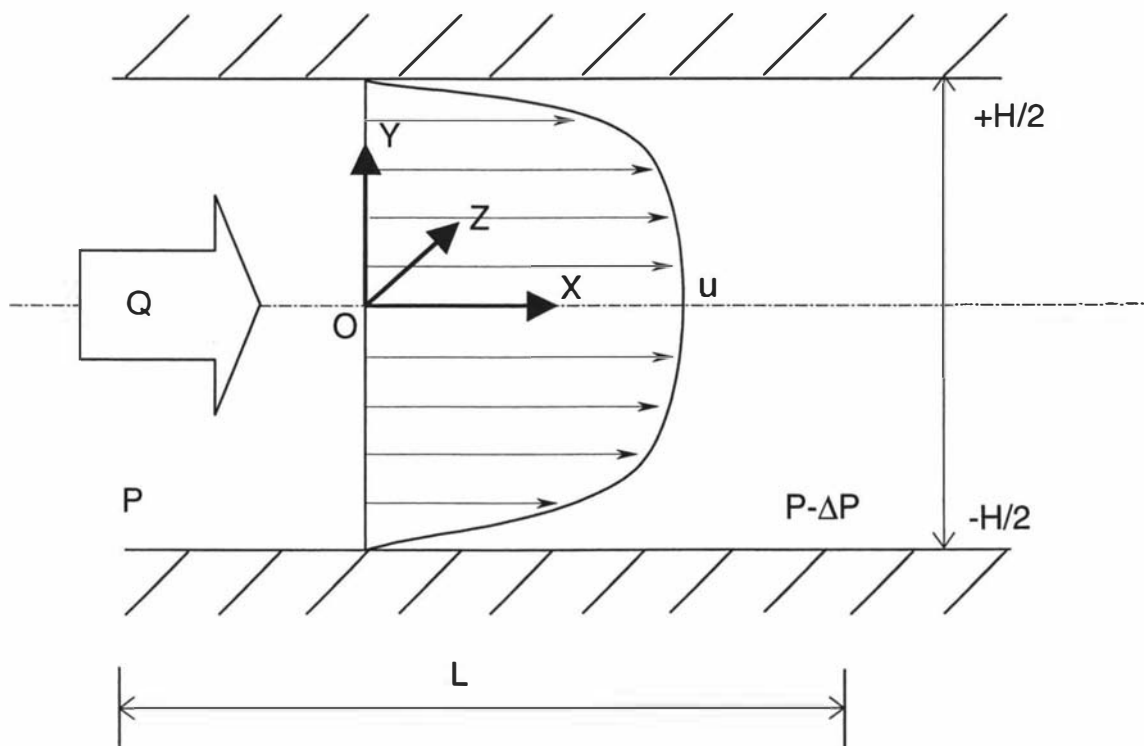

Figure B. 1 Two-dimensional slit flow model.

The volumetric flow rate of the slit can be calculated by

$$Q = \int_{-\frac{H}{2}}^{\frac{H}{2}} u \cdot W \cdot dy = 2W \int_{0}^{\frac{H}{2}} u \cdot dy \tag{B.1}$$

where H is slit height; W is the slit width; u is the flow velocity; and Q is the volumetric flow rate. Integration (B.1) by parts gives:

$$\frac{Q}{2} = W \cdot u \cdot y \Big|_{0}^{\frac{H}{2}} - W \cdot \int_{0}^{\frac{H}{2}} y \cdot \frac{du}{dy} \cdot dy \tag{B.2}$$

The first term on the RHS of equation (B.2) is zero because of the assumed non-slip condition, i.e., $u=0$ at $y=H/2$. Thus

$$\frac{Q}{2} = -W \cdot \int_{0}^{\frac{H}{2}} y \cdot \frac{du}{dy} \cdot dy \tag{B.3}$$

An expression for the shear rate du/dy can be obtained from the power law model as:

$$\frac{du}{dy} = -\left(\frac{\tau}{K}\right)^{\frac{1}{n}} \tag{B.4}$$

By replacing equation (B.4) into equation (B.3), Equation (B.5) is obtained:

$$\frac{Q}{2} = W \int_{0}^{\frac{H}{2}} y \cdot \left(\frac{\tau}{K}\right)^{\frac{1}{n}} \cdot dy \tag{B.5}$$

The shear stress and the pressure gradient along the slit channel are related as:

$$\tau = y \cdot \left(\frac{\partial P}{\partial x}\right) \tag{B.6}$$

By substituting y in equation (B.5) by equation (B.6):

$$\frac{Q}{2} = W \int_{0}^{\tau_w} \frac{\tau^{(1+\frac{1}{n})}}{\left(\frac{\partial P}{\partial x}\right)^2 \cdot K^{\frac{1}{n}}} \cdot d\tau \tag{B.7}$$

τ_w is shear stress at the channel wall. Integration of equation (B.7) yields:

$$\frac{Q}{2} = \frac{W}{\left(\frac{\partial P}{\partial x}\right)^2 K^{\frac{1}{n}}} \cdot \frac{n}{2n+1} \cdot \tau_w^{\frac{2n+1}{n}} \tag{B.8}$$

At $y=H/2$:

$$\tau = \tau_w = \frac{H}{2}\left(\frac{\partial P}{\partial x}\right) \tag{B.9}$$

so that equation (B.8) becomes:

$$\tau_w = \frac{H}{2}\left(\frac{\partial P}{\partial x}\right) = K\left(\frac{2n+1}{3n}\right)^n\left(\frac{6Q}{W \cdot H^2}\right)^n = K' \cdot (\dot{\gamma})^n \tag{B.10}$$

Thus:

$$K' = K\left(\frac{2n+1}{3n}\right)^n \tag{B.11}$$

$$\dot{\gamma} = \frac{6Q}{W \cdot H^2} \tag{B.12}$$

By measuring Q, W, H, and $\frac{\partial P}{\partial x}$, the power law index n and consistency index K can be determined. By algebraic manipulation B.10 yields:

$$\frac{\partial P}{\partial x} = \frac{2K}{H}\left(\frac{2n+1}{3n}\right)^n\left(\frac{6}{W \cdot H^2}\right)^n Q^n \tag{B.13}$$

or

$$\frac{\partial P}{\partial x} = C\left(\frac{6Q}{W \cdot H^2}\right)^n \tag{B.14}$$

where C is a constant given by:

$$C = \frac{2K}{H}\left(\frac{2n+1}{3n}\right)^n \tag{B.15}$$

By taking the logarithm of both sides, Equation (B.14) becomes

$$\log\left(\frac{\partial P}{\partial x}\right) = \log C + n\log\left(\frac{6Q}{W \cdot H^2}\right) \tag{B.16}$$

therefore the index n can be determined by the slop of the plot of $\log\left(\frac{\partial P}{\partial x}\right)$ vs $\log\left(\frac{6Q}{W \cdot H^2}\right)$. Once n is known, the wall shear stress and the shear rate can be obtained from equations (B.10) and (B.12). It must be noted that rather than determine the local gradient $\frac{\partial P}{\partial x}$, an overall gradient $\frac{\Delta P}{L}$ is used provided the pressure profile is linear.

Appendix C

Data Acquisition System for Extrusion Test

A data acquisition system was developed for the new designed on-line Slit-Die-Viscometer.

It consists of the SDV developed for this study, the extruder control panel, two PC boards with inputs and outputs, a computer and a computer program specially written for this system. Figure C1 shows a schematic of the data acquisition system.

The data acquisition computer program contains about 4970 lines and is too lengthy to be listed in this thesis. The computer program, however, is available by request to the author of this thesis. Figures C2 and C3 illustrate a simplified flow chart of the computer program. In this program, there are many features built in to help the operator in the extrusion experiments. There is an on-line data processor to filter the noise of the data caused by the heaters and the extruder control units. Moisture content of the extrudate is calculated and displayed on the computer screen. A specially designed on-line calculator can be used to calculate settings of screw speed, feed rate and water rate according to the related throughput and moisture level, or vice verse. Pressure distribution on the SDV is displayed on-line. The SDV exit pressure is calculated by extrapolating the profile up to the SDV exit point. The pressure profile of the SDV is also monitored and if the pressure is too high, a warning is displayed on the computer screen and an alarm is set off. In this case, the extruder operating condition should be changed to reduce the pressure. The pressure distribution along the SDV and the throughput of the SDV are used to calculate the shear rate and shear stress. After a few measurements by varying the flow rate in the SDV to obtain different shear rates and stresses, the power law index n and consistency K are calculated. The procedures of calculation of n and K are listed in Appendix B.
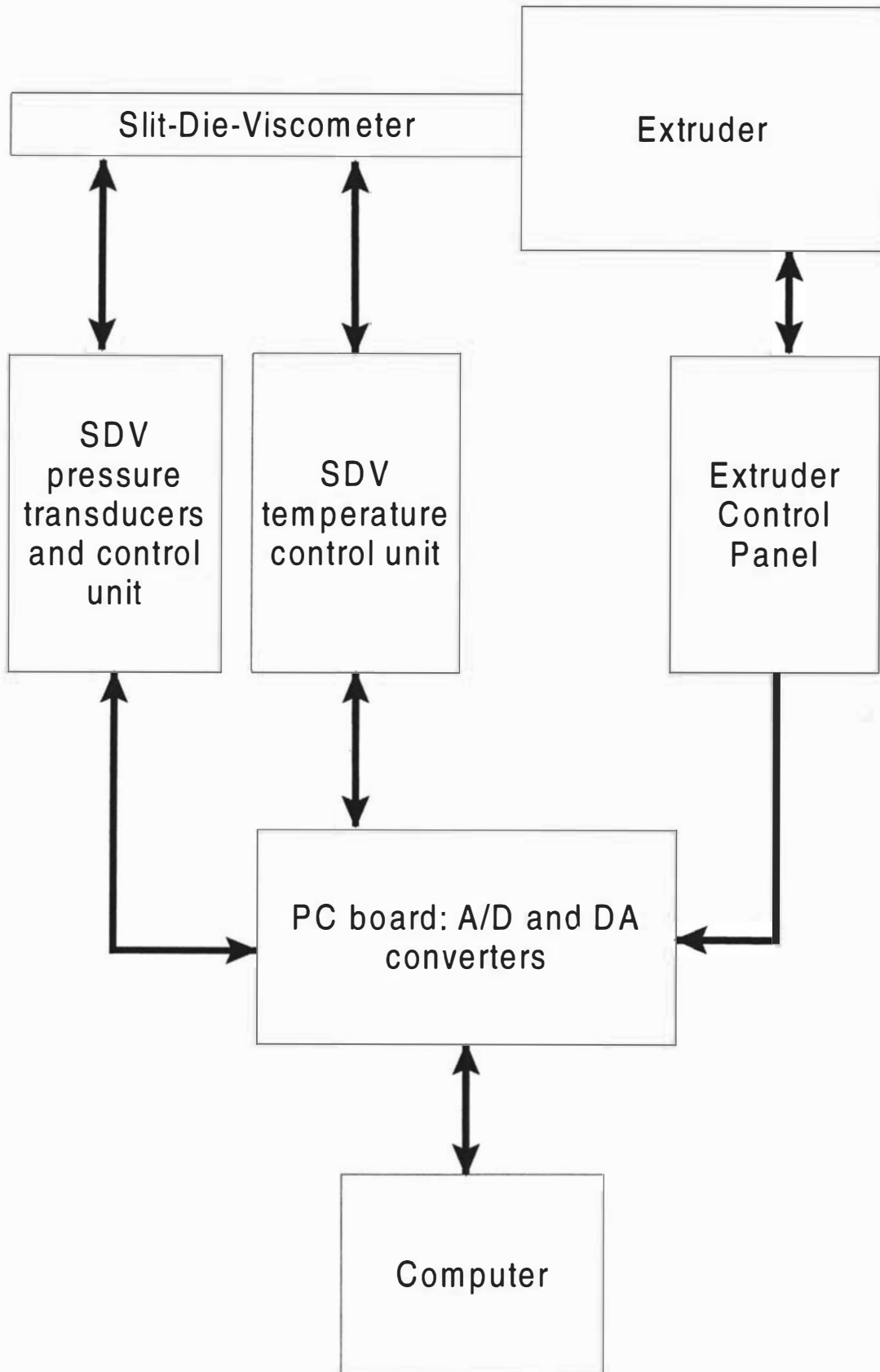
Figure C.1 Schematic diagram of the data acquisition program for extrusion tests.
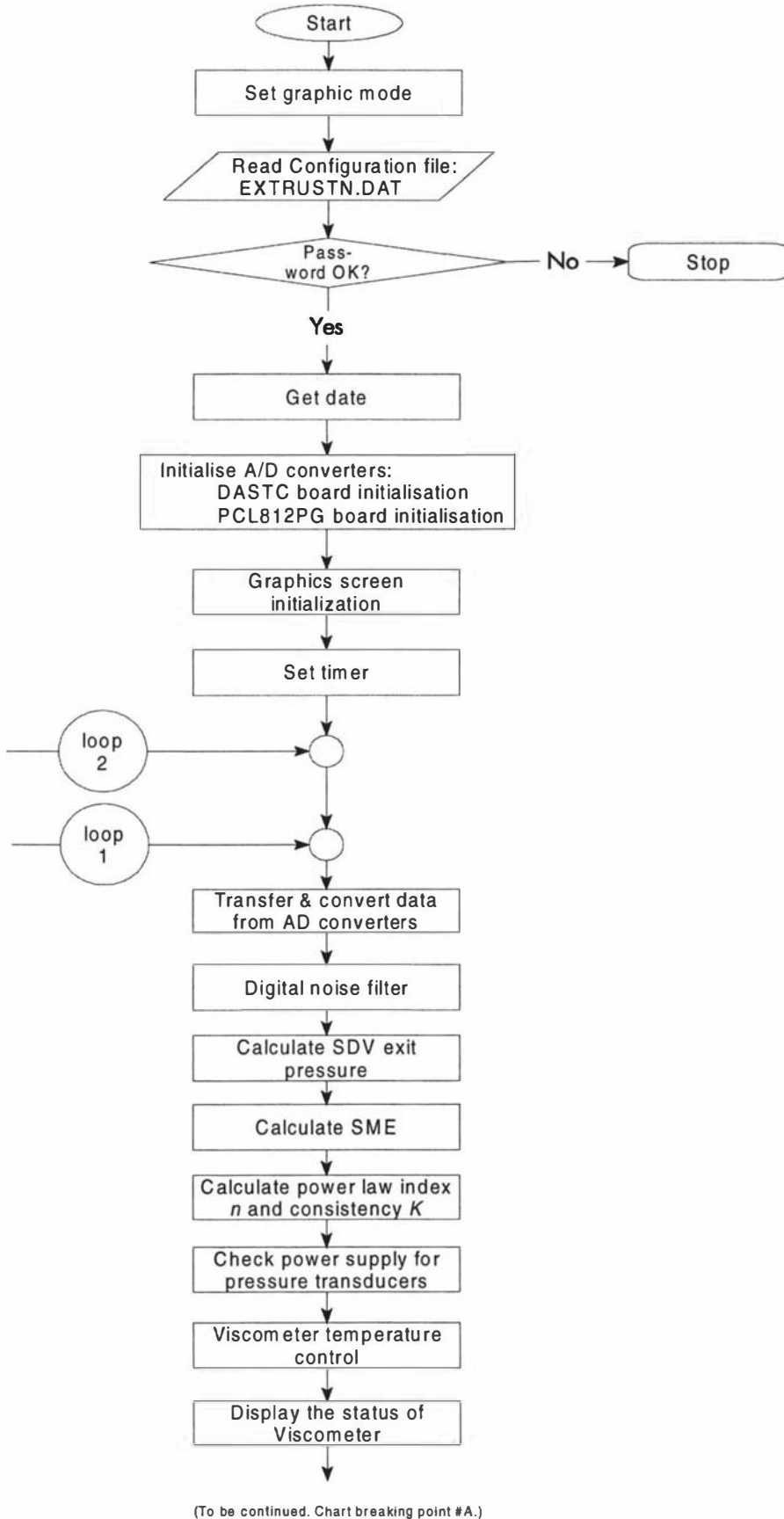
Figure C.2 Flow chart for the data acquisition program developed for the extrusion tests. Part I.

(Continued from chart breaking point #A.)
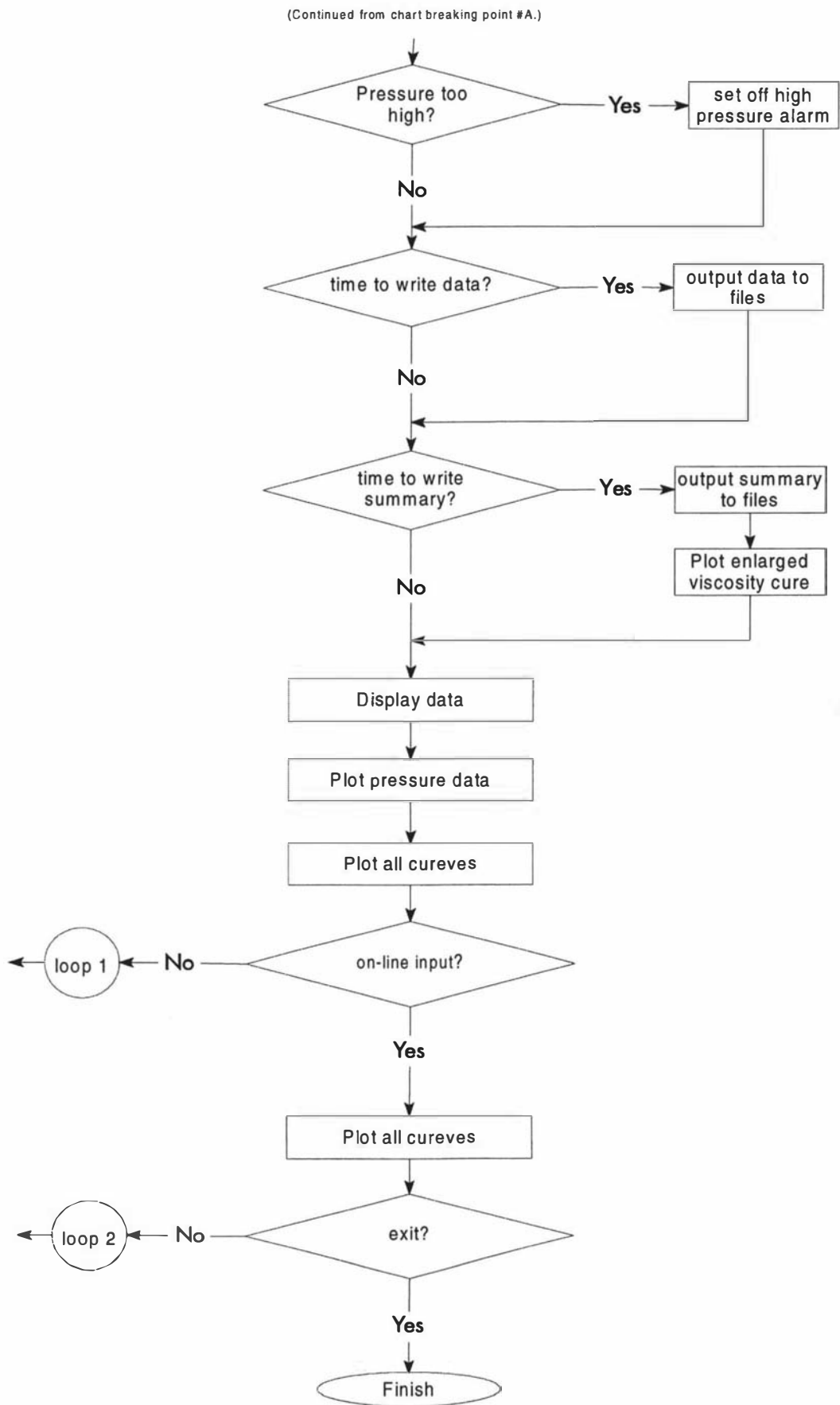
Figure C.3 Flow chart for the data acquisition program developed for the extrusion tests. Part II.

Appendix D

Engineering Drawings for the New Slit-Die-Viscometer
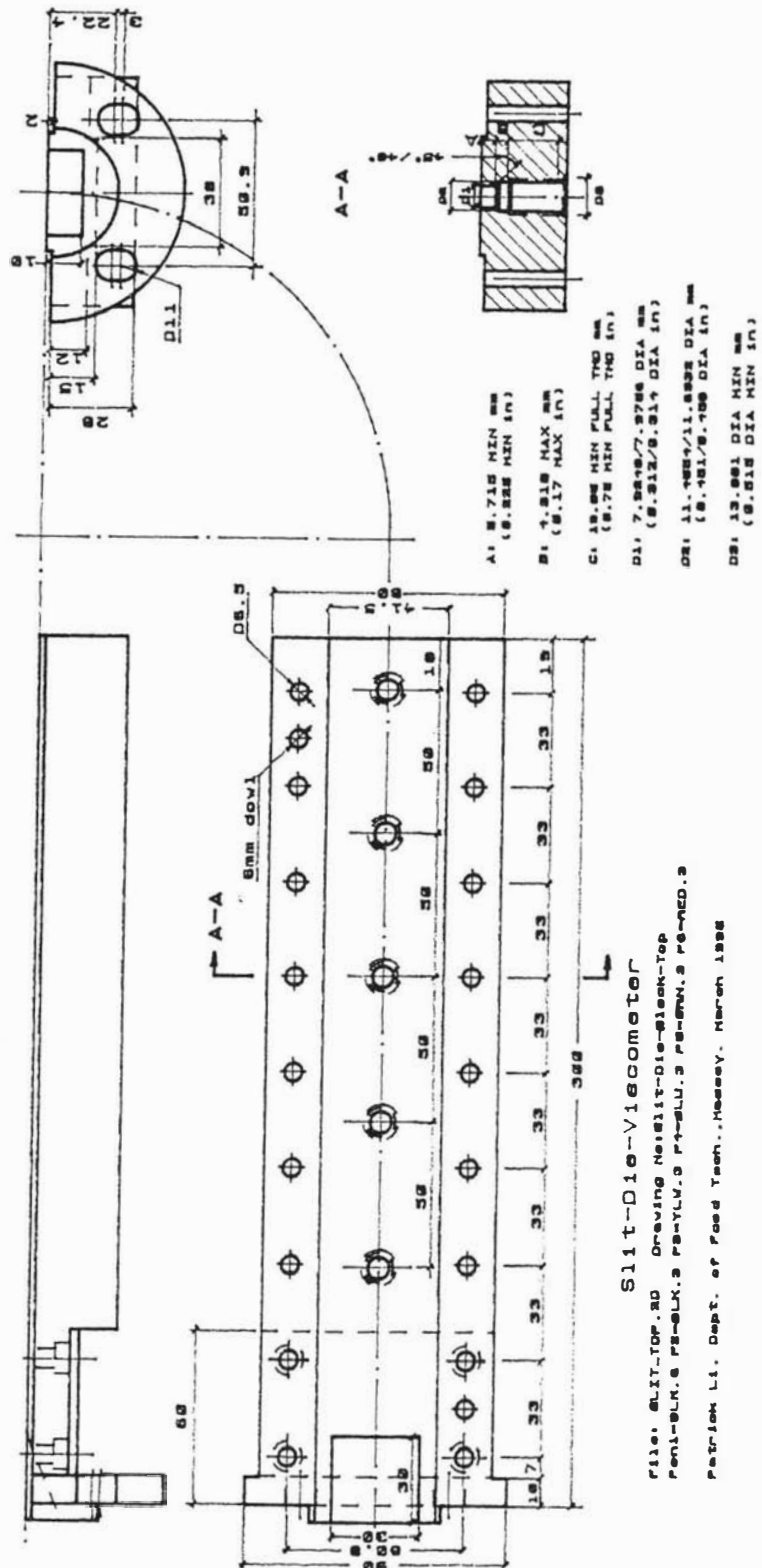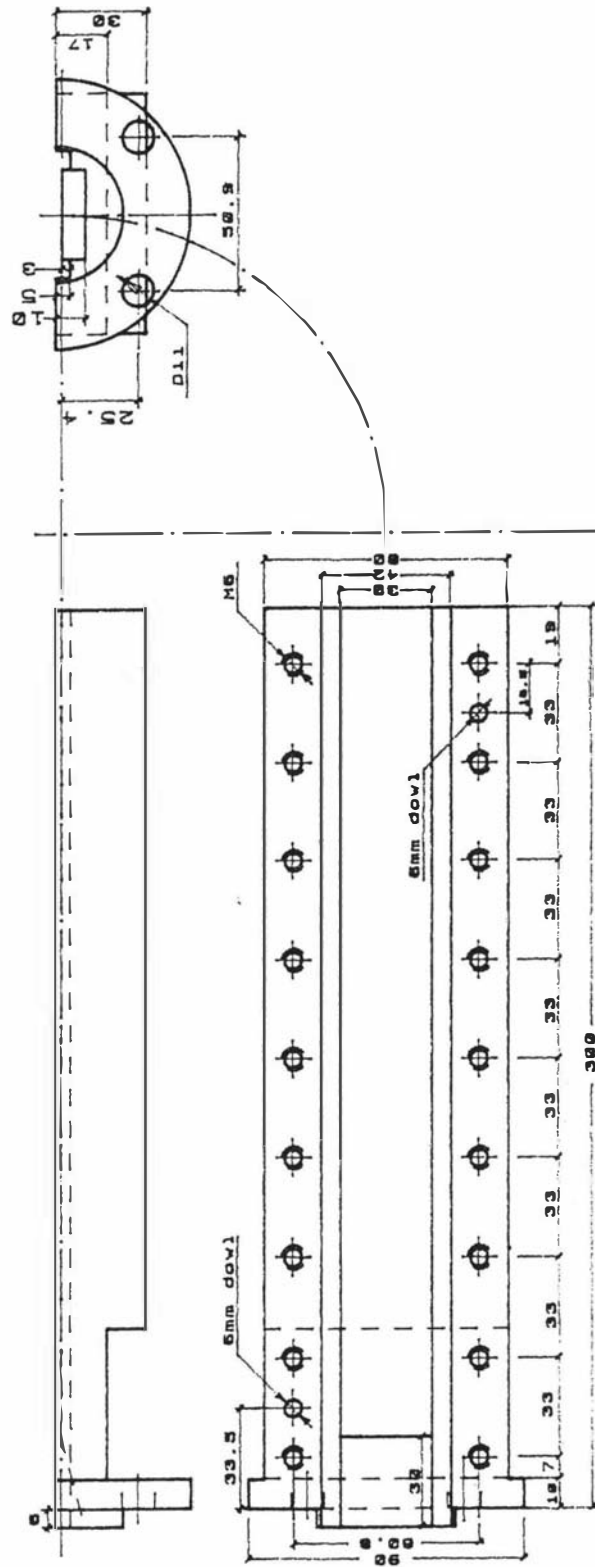
Slit-Die-Viscometer

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????

Patrick Li, Dept. of Food Tech., Massey. March 1995
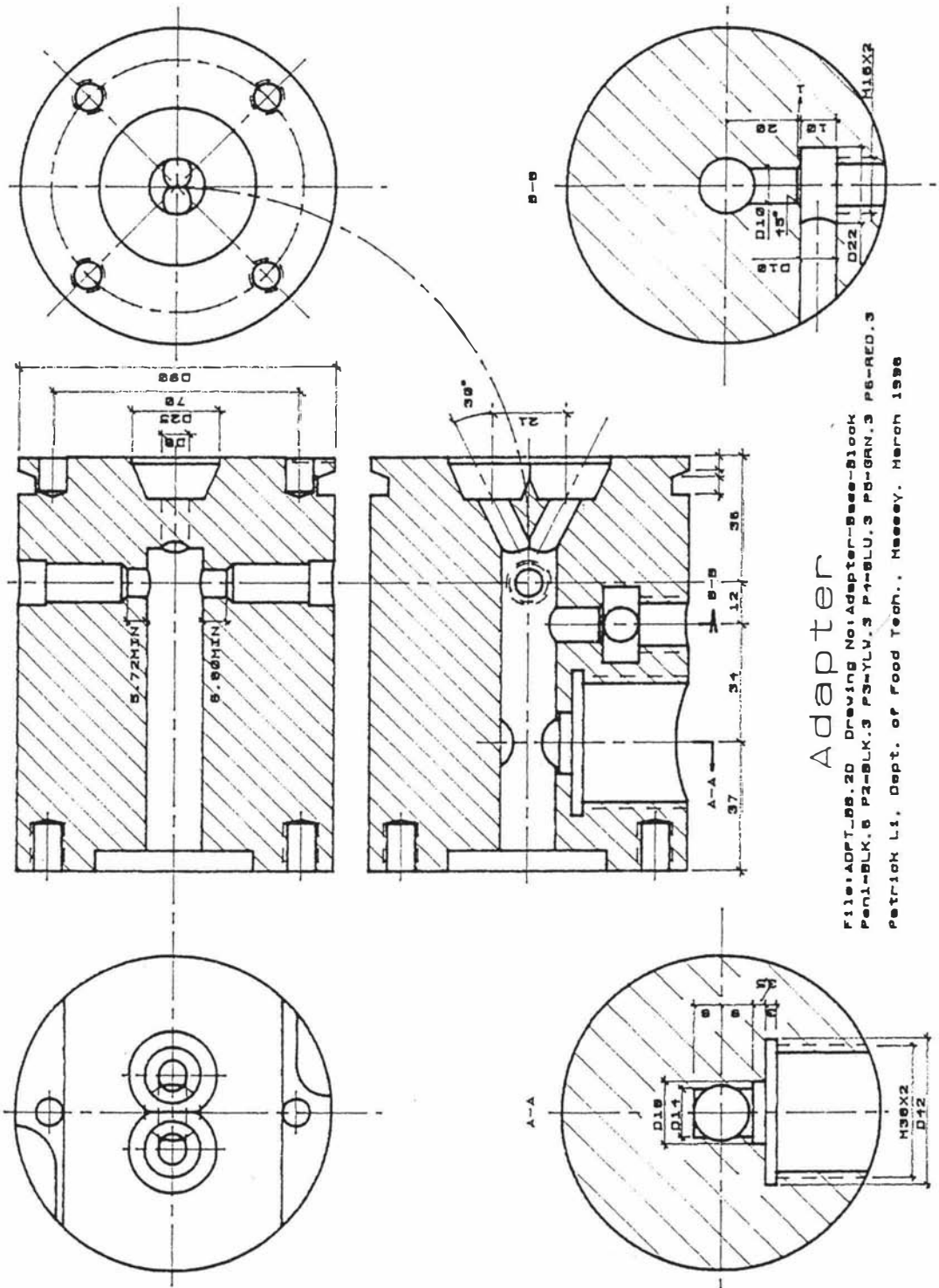
Adapter

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Patrick Li, Dept. of Food Tech., Massey. March 1998
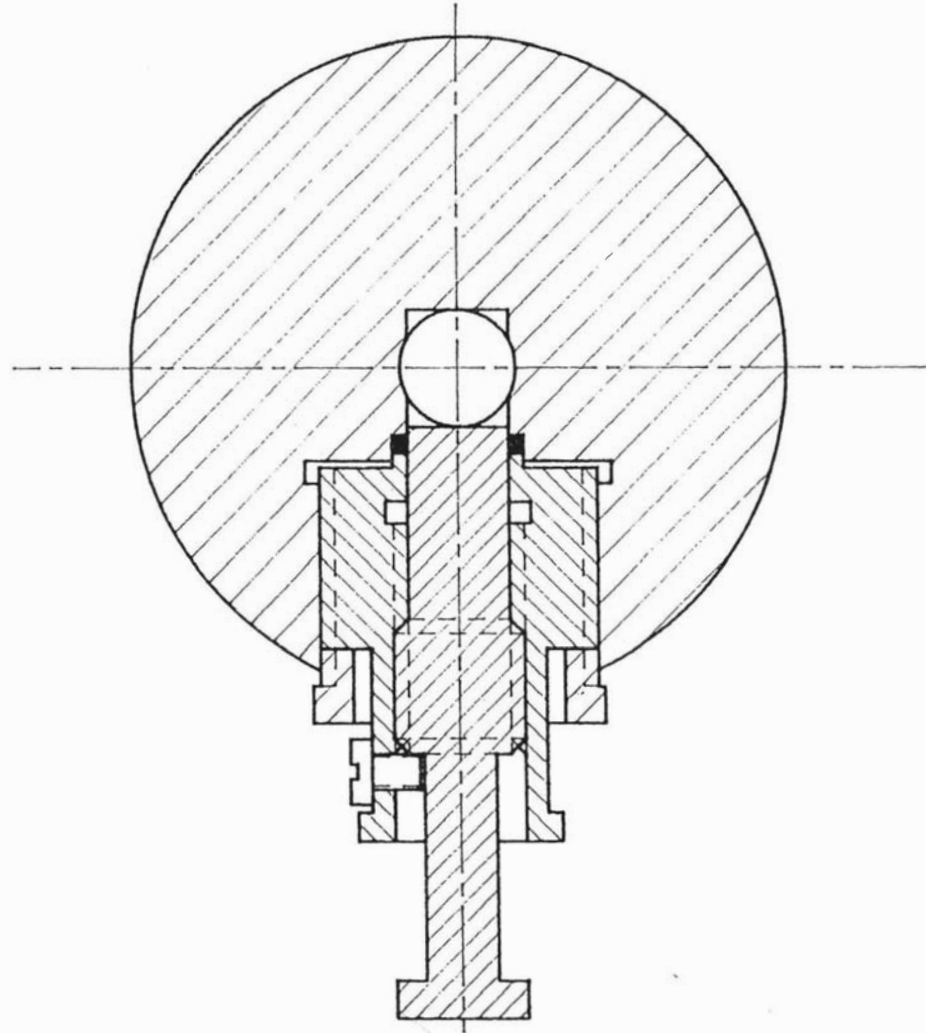
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Patrick Li,Dept. of Food Tech. Massey. March 1996
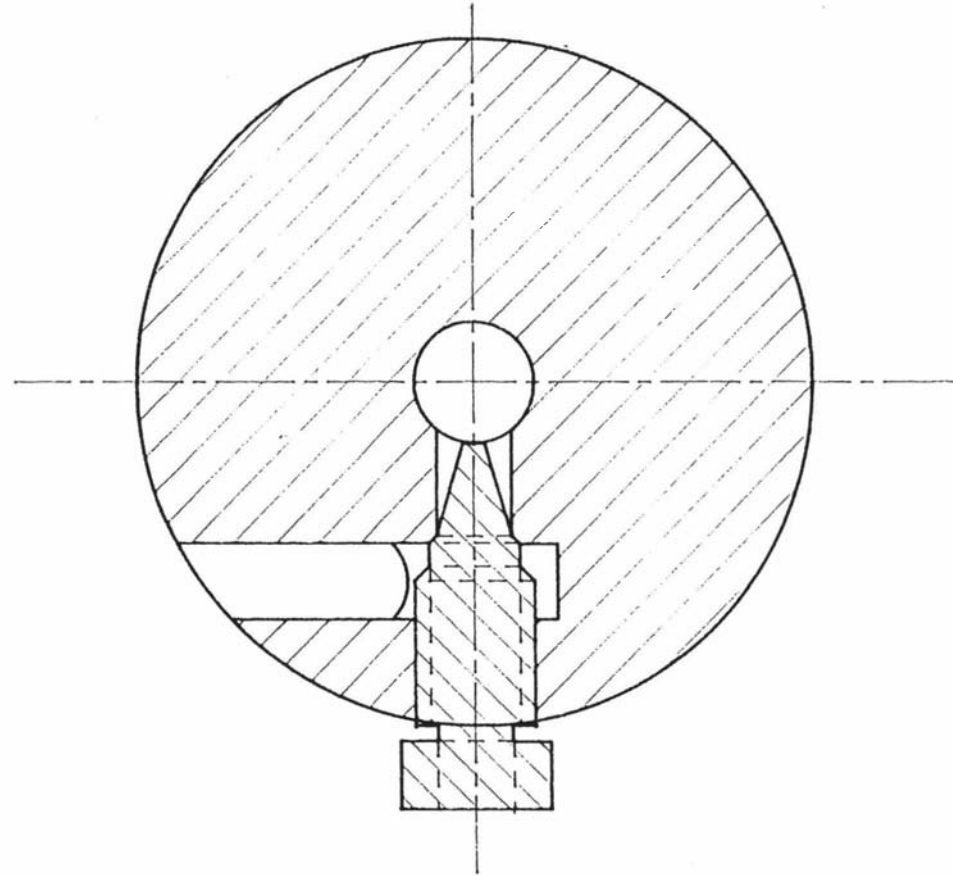
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Patrick Li.Dept. of Food Tech., Massey. March 1996
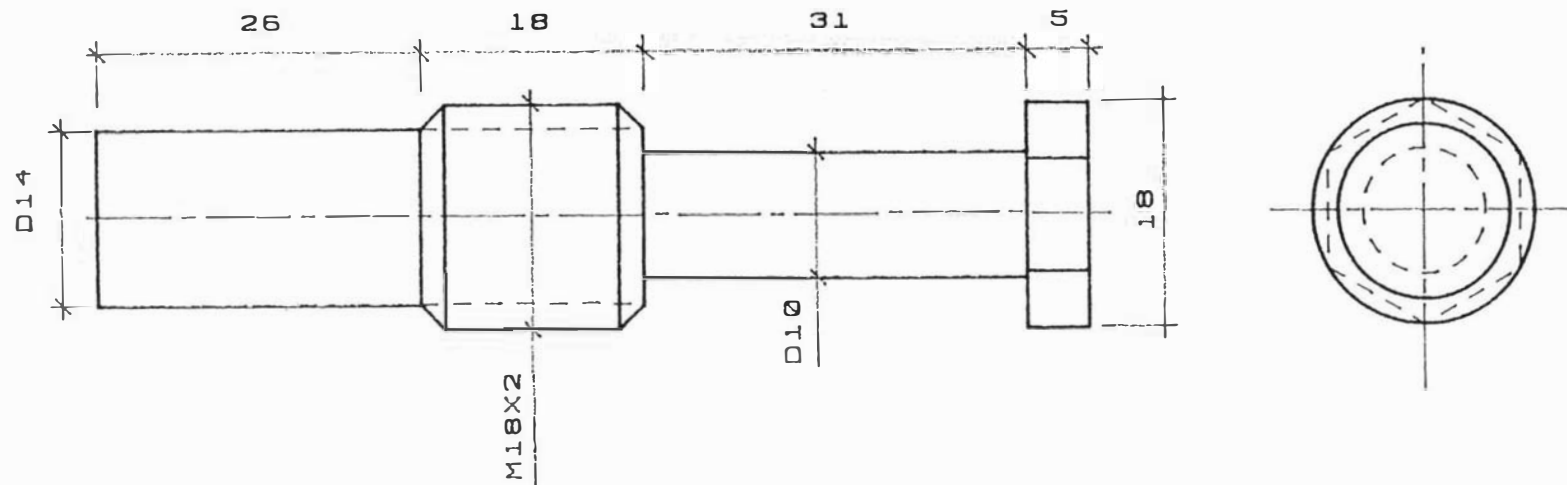
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Patrick Li. Dept. of Food Tech., Massey. March 1996
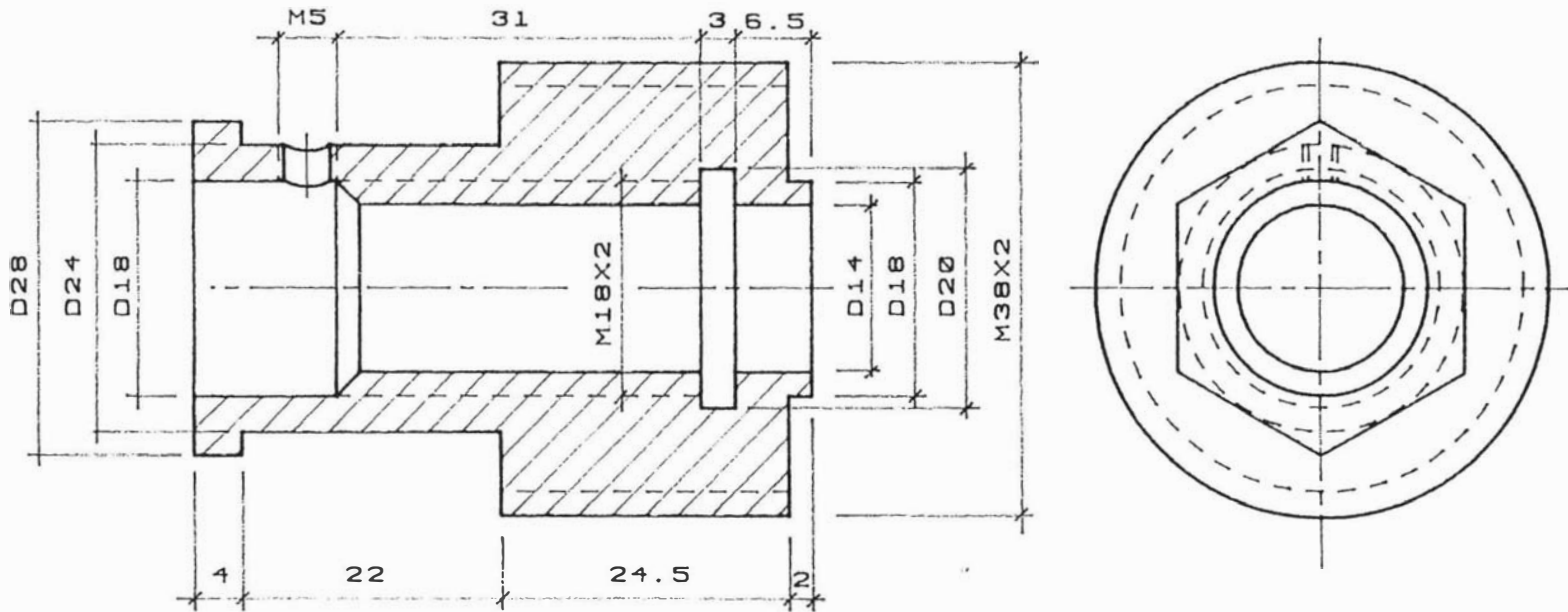
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Patrick Li, Dept. of Food Tech., Massey, March 1996
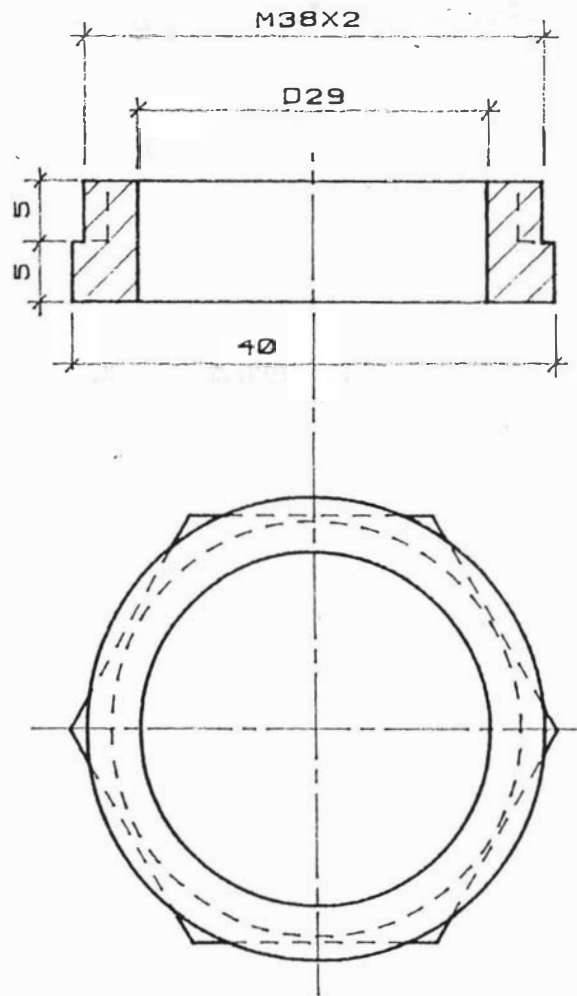
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Patrick Li. Dept. of Food Tech. Massey. March 1996
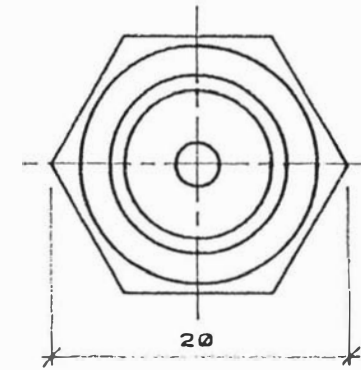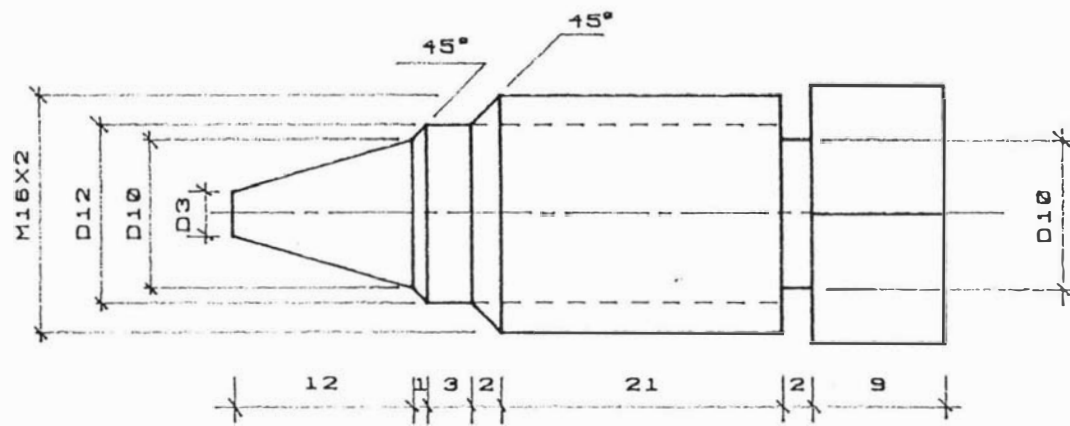
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Patrick Li Dept. of Food Tech..Massey. March 1996

Appendix E

Peer Reviewed Publications

- Li, P. X.-P., Campanella, O. H., Kirkpatrick, K. J. and Hardacre, A. K. 1997. A design of a new on-line Slit-Die-Viscometer to use in food extrusion. In Jowitt R. Ed. *Engineering & Food at ICEF 7*. Part I, International Association for Engineering and Food, Sheffield Academic Press, U.K, A153-A156.

- Li, P. X.-P., Campanella, O. H., Hardacre, A. K. and Kirkpatrick, K. J. 1997. Study of Rheological Characteristics of Corn Extrudate Using a Modified On-Line Slit-Die-Viscometer. *The Food Technologist*, Vol 27(1), 22-25.

- Li, P. X.-P., Hardacre, A. K. Campanella, O. H. and Kirkpatrick, K. J. 1996. Determination of Endosperm Characteristics of 38 Corn Hybrids Using the Stenvert Hardness Test. *Cereal Chemistry*. 73(4): 466-471.

A design of a new on-line slit-die-viscometer to use in food extrusion

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1. SYNOPSIS

Viscosities of extrudate melts during extrusion were measured using a modified on-line-Slit-DieViscometer (SDV). The new design consists of an adaptor inserted between the extruder and the die which diverts the extruder output. By varying the rate of the diverted flow the flow rates and shear in the viscometer can be varied without changing the extruder die pressure and the torque. The new viscometer was used to determine the rheological properties of two corn grits samples with different particle size distributions at two different screw speeds.

2. INTRODUCTION

Important properties which define the quality of extruded products such as expansion, pore-size distribution, and pore structure, are closely related to the viscosity of the extrudate or extrudate melt at the extruder die. The melt viscosity is also important when modelling the extrusion process as it affects the temperature and pressure.

Extruder-fed slit die rheormetry has been used extensively in food extrusion because it allows direct measurements of the pressure gradient in a slit die attached to the extruder by mounting pressure transducers on the slit surface. In order to vary the shear rate, the classical approach is to modify the extruder throughput (2,3). However, Vergnes et al. (1) showed that changes in the extruder throughput can change the extruder operating conditions, resulting in different thermomechanical treatment of the material and thus different final products. Therefore, the flow curves obtained do not represent the flow behavior of a single product. In order to circumvent this drawback, van Lengerich (4) defined 'specific throughput' as the ratio between the extruder throughput and the screw speed and suggested that by maintaining a constant specific throughput, flow rates can be varied while the product receives the same thermomechanical treatment. This method, however, leads to very long experimental procedures.

Padmanabhan and Bhattacharya (5) used a side stream valve placed at the last section of the extruder to control the flow rate and thus the shear rate in the viscometer. The flow curves obtained were significantly different from those previously obtained varying the screw speed or the throughput. However, there was no indication whether the pressure before the slit die viscometer was kept constant during the experiments. Vergnes et al. (1) described an on-line slit die viscometer design based on the principle of twin channels. Using a balance of feed rate between the two channels the flow rate and shear rate in one of the channels was modified with a piston valve without changing the operating conditions in the extruder. However, the die pressure was not monitored during the experiments and in order to maintain the same operating conditions the valve position. which depends on the power law index n, had to be calculated before the experiment. For the calculation the authors assumed that the valve position is not a strong function of n provided a proper ratio between the valve and the slit lengths is chosen and n is greater than 0.4. However, maize extrudates can have power indices in the range 0.2 to 0.5 making the above assumption inaccurate. An obstruction of the flow in the slit area can also disturb the flow pattern.

The objective of this work was to design and test the effectiveness of a new on-line viscometer to determine the rheological properties of corn grit extrudates. The object of the design was to provide for varying the shear rates in the slit die without affecting conditions in the extruder.

3. MATERIALS AND METHODS

3.1. Materials
Corn grits from a single hybrid but of two different size distributions were obtained from Seedbank Ltd. with moisture content of about 14.1% w/w, density of 1430 kg/m3 (measured with a picnometer). Figure 1 shows the particle size distribution of the grits. A simple additive rule (1) was applied to calculate the density of the extrudate on line.

3.2. The Modified Slit-Die-Viscometer (SDV) and the extruder
The modified SDV is shown in Fig. 2. It consists of a slit die block with dimensions of 2x30x250mm Five combined pressure and temperature transducers (Dynisco TPT463) are flush mounted on one side of the slit. Heating elements controlled by a computer are employed to maintain the temperature in the viscometer. Before the slit viscometer, an adaptor is fitted which allows the diversion of flows. By carefully adjusting the openings of valves A and B to maintain a constant extruder die pressure, extruder operating conditions remained unchanged, whereas the flow rate in the SDV varied and different shear rates were obtained. The die pressure was monitored by a pressure transducer (Dynisco PI415). The design is relatively simple and has the advantage that any typical SDV can be easily converted without changing the original design. Compared with Vergnes's design (1), this design has the advantage that the position of the valves A and B is independent of n. The modified SDV was attached to a Clextral BC21 twin screw corotating extruder. The screw configuration combined forward screw elements with different pitches, mixing paddles and a pair of reverse elements.
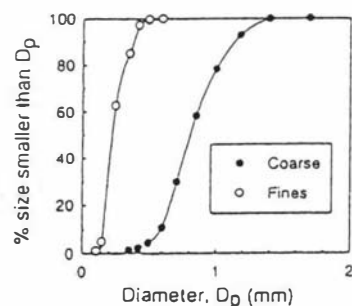
Fig 1. Corn grits size distributions
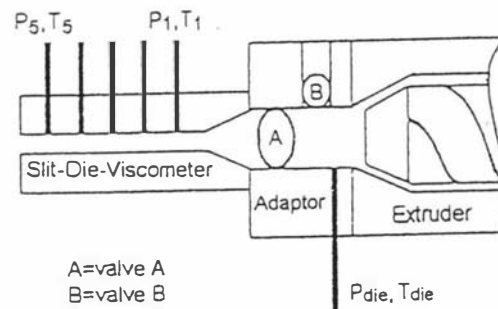
Fig 2. Diagram of the modified slit-die-viscometer.

3.3. Experimental Approach
The extruder and SDV were first allowed to reach thermal equilibrium at 120•C. The first and second barrel sections were set at 60•C and 90•C respectively. Corn grit and water were metered into the extruder. Initially valve A (Fig. 2) was fully-open and valve B fully-shut. The SME was calculated from the following equation:

where SME is in W-h/kg, *ScrewSpeed* is in *rpm*, Torque and InitialTorque in *N-m*, and *Throughput* in *kg/h*. InitialTorque is the torque required to rotate the extruder screws with empty load, which is a function of screw speed.

Degree of gelatinisation of the extrudate was determined as described by Wootton et al (7).

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4. RESULTS AND DISCUSSION

Typical flow curves for the fine grits extruded at two different screw speeds are shown in Fig. 3. It is evident that the melts follow a power-law rheological model and that melts with slightly lower viscosities can be produced at higher screw speeds. Fig. 4 shows apparent viscosity vs shear rate curves for two different grits samples at 400 rpm. The figure indicates a crossover at a shear rate of about 8 s~. For shear rates larger than this, the viscosity of the coarse melt is less than that of the fines melt. Gomez an* Aguilera (6) noted that the extrusion process can modify the structure of native starch by the action of shear and temperature. For a given screw profile, shear can be increased by changing the extruder screw speed. However an increase in screw speed also reduces the product residence time and therefore the extent of starch modification. Measured and calculated values for all the data collected in this study are given in Table 1 which effectively summarises the differences between the screw speeds and grits types.

Particle size range	Speed rpm	n	k $Pa.s^n$	SME W-h/kg	(Torque - InitialTorque) N-m	P_{omot} MPa	P_{die} MPa	Throughput kg/h
Fine	280	0.56	2.089	47.9	12.6	6.4	7.5	7.7
Fine	400	0.56	1.971	54.7	10.5	6.0	7.1	7.7
Coarse	280	0.44	2.657	29.6	7.7	5.3	4.8	7.8
Coarse	400	0.44	2.400	36.3	6.7	4.9	4.1	7.8

Table 1. Summary of the variables measured and calculated during the extrusion trial.

The effect of particle size on the extruder performance is clearly illustrated in Fig.4 and shows that for the same feed rate, temperature profile, screw profile and moisture content the fine grits required a larger mechanical energy input (larger SME values) than the coarse grits. The larger mechanical energy applied to the fine grits resulted in a melt which was less viscous and which showed less shear-thinning as indicated by the smaller values of k and larger values of n. This indicates that using the same operating conditions, greater modification of the extrudate can be achieved if finer grits are used. Table 1 also illustrates the effect of screw speed on the viscosity of the product. Increasing screw speed for a given feed results in a less-viscous melt. It is important to note in Table 1 that die (Pdie) and thrust (P~h~) pressure were greater for the fine than the coarse grits. The difference in die and thrust pressure could be explained by the fact that coarse grits melts exhibit more shear thinning (smaller n values) than fine grits melts, so, at the high shear rates present at the extruder die this melt has a lower viscosity and less pressure is generated.
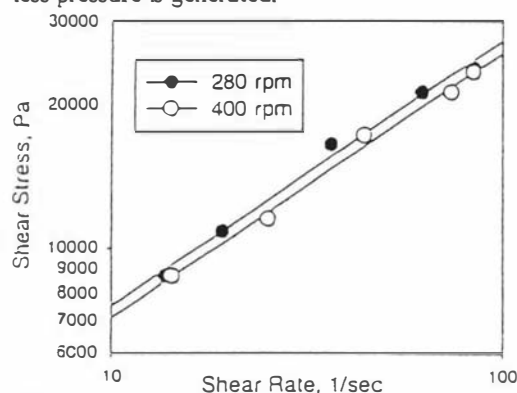

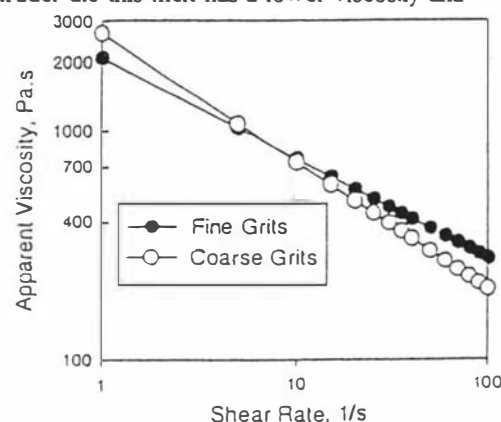Fig 3. Shear rate vs shear stress for the fine grits at two different screw speeds.

Fig 4. Apparent viscosity vs shear rate curve

In order to confirm that the rheological properties of the melt were affected by the extruder screw speed and the grit size, the degree of gelatinisation of the extrudate collected before and after the slit die was determined, Table 2. Fine grits were almost completely gelatinised prior to passing through the SDV but gelatinisation of the coarse grits was incomplete. The increased screw speed increased gelatinisation of the coarse grits. These results are in agreement with the viscosity measurements. The table also shows that for the coarse grits and at the lower screw speed of 280 rpm the product after the slit die is slightly more gelatinised that the product before the die indicating that some cooking occurs when the melt pass through the slit die.

%	Fine, 280rpm	Fine, 400rpm	Coarse, 280rpm	Coarse, 400rpm
Before Slit-Die	98.0	99.0	89.2	92.5
After Slit-Die	99.0	100.0	93.9	93.0

Table 2. Measurement of degree of gelatinization of the extruded samples.

5. CONCLUSIONS

This experimental approach can standardise the effects of thermomechanical treatment history on the rheological measurements. The design was used to study the effect of particle size and screw speed on the rheological properties of corn grits. The study showed that the rheological constants are related to the gelatinisation which in turn depends on the particle size of the raw material and the screw speed of the extruder.

6. ACKNOWLEDGMENTS

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(6) Gomez, M.H. and Aguilera, J. M. 1983. Change in the starch fraction during extrusion cooking of corn. *J. Food Sci.*, 49:4043, 63.
(7) Wootton, M., Weeden, D. and Munk, N. 1971. A rapid method for the estimation of starch gelatinisation in processed foods. *Food Technol. Aust.* 23:612-615.

Study of
Rheological Characteristics
of Corn Extrudate
using a
Modified On-Line Slit-Die-Viscometer

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(2) Crop & Food Research Ltd., Palmerston North, New Zealand.

ABSTRACT:

A new design of an on-line Slit-Die-Viscometer (SDV) is described which incorporates a side stream valve allowing flow rates and shear in the viscometer to be varied without altering die pressure. Using this configuration the effects of thermomechanical treatment history on the rheological measurements can be eliminated. Results obtained from this system for the rheological flow behaviour of the melt produced during the extrusion of corn meal was found to follow the simple power law. Viscosity of the melt was affected by extruder barrel temperature. The viscosity of the melt increased with temperature to about 140°C after which the viscosity decreased. Other extruder operating parameters also altered the viscosity of the melt.

INTRODUCTION

The viscosity of the melt in the extruder at a defined temperature and pressure critically affects the properties of the extrudate and the quality of the product such as expansion rate or bulk density, die swell and product curvature. Knowledge of viscosity of the extrudate melt is also very important when modelling or designing the extrusion process as it affects the temperature and pressure developed in the extruder. In general, a power-law equation can be used to describe the rheological behaviour of the extrudate melt:

$$\tau = K\dot{\gamma}^n$$

where τ is shear stress, Pa (Pascal); K is consistency index, Pa·s^n; $\dot{\gamma}$ is shear rate, 1/s; and n is power law index.

The rheological characteristics of extrudate melt must be measured on-line (Bhattacharya and Padmanabhan 1991, 1994) because commonly used techniques such as rotational rheometry, are unworkable. Extruder-fed slit die rheometry has been used extensively because it allows direct measurements of the pressure gradient developed in the die by mounting pressure transducers on the slit surface. The classical approach used to vary flow rates through the slit die viscometer (SDV) and therefore apply different shear rates, was to modify extruder throughput (Senouci and Smith 1988, Lai and Kokini 1990). As explained by Vergnes et al (1993), changing the extruder throughput changes the thermomechanical treatment of the material and leads to different final products. Flow curves obtained using this type of approach do not represent flow behaviour of a single product and Padmanabhan and Bhattacharya (1991) reported negative values of power law index.

For the twin-screw extruder, Van Lengerich (1991) suggested that if specific throughput (the ratio between the extruder throughput and the screw speed) is kept constant, the degree of fill in the extruder also remains constant and therefore the product undergoes the same thermomechanical history. Hence it is possible to vary the throughput to obtain different flow rates at the SDV. However, this method leads to very long experimental procedures.

Recently Padmanabhan and Bhattacharya (1993), Bhattacharya and Padmanabhan (1994) evaluated the use of a SDV and a side stream valve to measure the shear viscosity and the first normal stress difference. A side stream valve was placed at the last section of the extruder to control the flow rate through the die. They found that the flow curves obtained using this technique are significantly different from those obtained from varying the screw speed and throughput. Their use of a side stream valve to vary the flow rate of the SDV reduced the effects of the thermomechanical history on the rheological measurements. Bhattacharya and Padmanabhan (1994) monitored the melt pressure before the SDV but it was not shown whether the pressure was maintained constant. From their schematic diagram of the experiment, the side stream valve was positioned before the die. Therefore, varying the side stream valve opening altered both the pressure at the extruder die and the flow rate at the SDV. Pressure changes at the die changed the extruder operating conditions and varied the thermomechanical treatment of the extrudate.

A study by Vergnes et al (1993) described an on-line SDV based on the principle of twin channels. Using a balance of feed rate between the two channels the flow rate and shear rate in one of the channels could be modified without changing the flow conditions in the extruder. Each channel was provided at its entrance with a piston valve, which could be moved up and down in order to partially obstruct the flow section. Because the die pressure of the extruder was not monitored, to maintain the same operating condition the relationship of the two valve openings had to be calculated before the experiment. They found that the relationship between the two valve openings is dependent on the power law index n and on the ratio between the lengths of the valve and the slit die. They also pointed out that the valve opening is not very dependent on the power law index n when a proper ratio between the valve and slit lengths is chosen and when n is greater than 0.4. However, maize extrudate melt can have power law indexes in the range 0.2 to 0.5 which makes the above assumption inaccurate. Specific Mechanical Energy (SME) was the only indicator used by Vergnes et al (1993) to determine whether the product was subjected to the same thermomechanical treatment process.

Studies on the rheological flow behaviour of corn meal melts independent of the operating conditions are limited. The objective of this work was to design an on-line viscometer to measure the rheological properties of extruded corn grits which did not affect the thermomechanical treatment of the extrudate.

MATERIALS AND METHODS

Materials

Corn grits with moisture content of 14.1% w/w and bulk density 1430kg/m^3 were supplied by Seedbank NZ. The particle size distribution of the grits is shown in Table 1. A simple additive rule

was applied to calculate the density of the extrudate on line (Vergnes et al 1993).

<0.2 mm	<1 mm	<1.18 mm	<1.4 mm	<1.7 mm	>1.7 mm
Nil	20-35%	40-45%	20-35%	0-5%	Nil

Table 1. Particle size (mm) distribution of corn meal (w/w).

The Modified Slit-Die-Viscometer(SDV)

The modified SDV used in this study is shown in Figure 1. It consists of a normal slit die block which is used to measure the shear rate and shear stress and an adapter. Five pressure transducers and thermocouples (Dynisco TPT463, with a range from 500psi to 3000psi) are flush mounted on one side of the slit. The slit is 2mm in height, 30mm in width and 250mm in length. Computer controlled heating elements are employed to maintain the viscometer die at a constant temperature. Ahead of the viscometer an adapter with two valves diverts part of the flow (Figure 1). By varying the openings of valve A and B, the extrudate flow can be divided into two streams. The exit pressure of the extruder (Pdie) is kept constant during the experiment and is monitored by a pressure transducer (Dynisco PT415, 5000psi). This design is relatively simple and has the advantage that a classic SDV can be easily converted into a modified SDV without modifying the die structure. Because the exit pressure of the extruder is closely monitored and kept constant, the degree of fill is also kept constant. Thus the material in the extruder barrel undergoes the same thermomechanical treatment while varying shear rate in the SDV. Compared to Vergnes et al's (1993) design, this design has the advantage that the positions of valves A and B are not dependent on the power law index n.
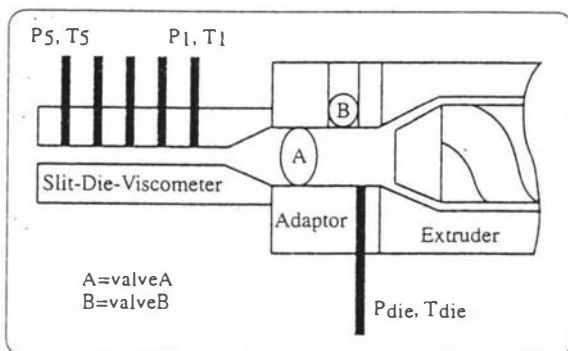

Figure 1. Schematic Diagram of the Modified SDV.

The extruder

The modified SDV was attached to a Clextral BC21 twin screw co-rotating extruder. The screw diameter is 25mm. The extruder barrels are equipped with an independent temperature control unit for each section. Material was fed into the extruder by a volumetric feeder and water was directly injected into the mixing section of the extruder with a dosing pump. The screw configuration combined different pitches on the forward screw elements, mixing paddles and a pair of reverse elements. The screw rotating speed, feed rate and water rate were controlled and the screw torque and thrust pressure were measured. The SME was defined as the amount of mechanical energy input to each unit of the extrudate melt mass. It was calculated from the screw torque, screw speed and throughput by:

$$SME = \frac{\pi \times \omega \times T}{30 \times Q}$$

Where *SME* is Specific Mechanical Energy in w~h/kg, ω is screw speed in rpm, *T* is screw torque in *N-m*, and *Q* is extruder throughput in *kg/h*.

Data acquisition and control system

A computerised data acquisition and control system was developed for this study. All extrusion and SDV parameters were controlled, measured and stored by this system. A data processor was employed to perform real time noise filtering, error handling, statistical analysis, and on-screen data plotting. The data displayed included SDV pressure profile, linear and log-log graphing of shear rate and shear stress curves.

With real time on-screen plotting, steady states of the extrusion process could be easily visualised thus increasing the confidence and efficiency of operating the equipment and the accuracy of the measurements.

Experimental Approach

In all experiments, the extruder and SDV were allowed to reach thermal equilibrium prior to data collection. At the beginning of an experiment, valve A of the adapter was open and valve B shut. The extruder was allowed to reach stable operating conditions. By carefully adjusting the openings of valves A and B to maintain a constant extruder exit pressure and constant SME, shear rate in the SDV could be varied while operating conditions in the extruder remained unchanged. Once stable operation was reached data were recorded for a few minutes with between 100 and 1000 readings for each parameter. These readings were then averaged to obtain the data points presented.

The temperatures of the extruder barrels, screw speed and throughput were chosen as input variables. For each experiment one parameter only was varied, screw configuration and all other parameters remained constant.

RESULTS AND DISCUSSION

1. Effect of barrel temperature

The temperature of the extruder barrels was controlled in 10°C steps between 100°C and 160°C, the temperature of the SDV remained constant at 120°C to eliminate its influence on the viscosity. The screw speed (450 rpm), feed rate of grits (9.7kg/h), water feed rate (2 l/h), and consequently moisture content of the melt (35%w/w) were kept constant.

At all temperatures, the melt followed the power law behaviour (Figure 2). For each temperature plots of log wall shear stress and log apparent shear rate were approximately linear ($R^2 \geqslant 0.93$).

The viscosity of the melt was greatest at about 140°C as was the value of the power law consistency K. At higher temperatures the viscosity decreased, it is expected that at such high temperatures starch dextrinization occurred. When the temperature of the
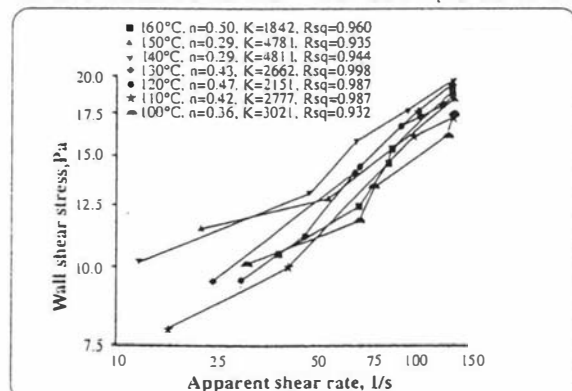

Figure 2. Effect of extruder barrel temperature on the viscosity as a log-log plot.

extruder barrels was altered, the SME, torque, die block pressure and thrust pressure also altered albeit within a small range (data not shown).

2. Effect of Screw speed and throughput

In these experiments the temperatures of the SDV and extruder barrels were kept at 120°C. The moisture content of the extrudate was controlled at 35% w/w.

Screw speed had little effect on the rheological properties of the extrudate at a constant feed rate. The flow curves generated for three different screw speeds in Figure 3 were very similar. The screw speed had little impacts on the torque, the thrust pressure or the die pressure (Figure 4). However SME decreased when the screw speed was changed from 300 rpm to 400 rpm then increased with increasing speed. As the screw speed increased, more materials were pushed through the extruder within the same period of time thus the degree of fill decreased. This led to the reduction of torque and thrust pressure observed. The viscosity of the material was also reduced because of the increased shear, resulting
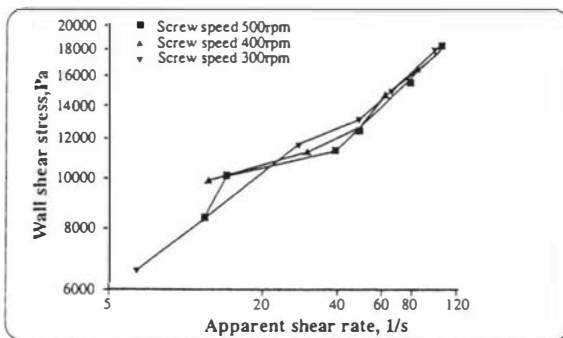
Figure 3. Effect of screw speed (constant feed) on the viscosity as a log-log plot.
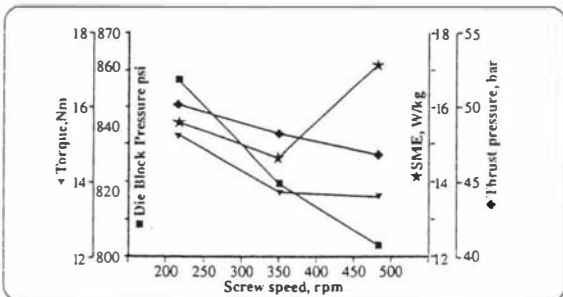
Figure 4. Effect of screw speed (constant feed) on the extruder parameters.
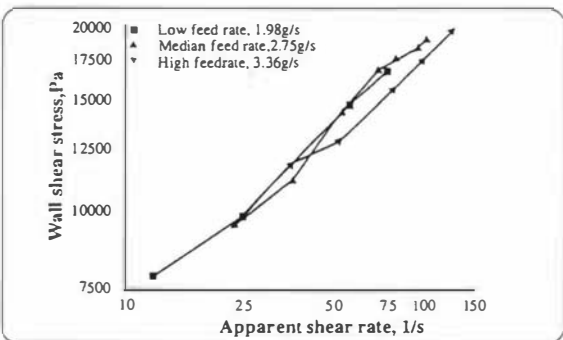
Figure 5. Effect of throughput (constant screw speed) on the viscosity as a log-log plot.
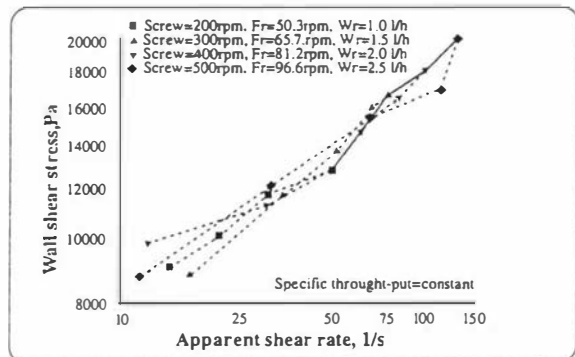
Figure 6. Effect of throughput on the viscosity as a log-log plot.

a less pressure build up in the die. Because the torque scarcely varied between 400 rpm and 500 rpm, it follows from the definition of SME that a large increase in screw speed must lead to an increase in SME.

With a constant screw speed and small throughput ($\leq$ 2.75g/s) the melts demonstrated similar rheological behaviour (Figure 5). When the throughput was further increased the viscosity of the melt appeared to vary indicating that the 'degree of fill in the extruder was changing.

These results clearly showed that screw speed and throughput of the extruder had less effect on the viscosity of the extrudate melt than barrel temperature did over the range of conditions tested. Further more, if a constant specific throughput was maintained the products had very similar rheological behaviours (Figure 6). This indicated that the materials experienced a similar thermomechanical treatment. It is shown in Figure 7 that as throughput increased, the die-pressure, SME, torque, and thrust pressure increased significantly, even when specific throughput was constant.
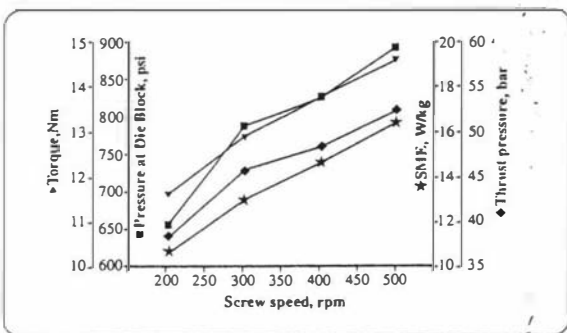
Figure 7. Effect of throughput on the extruder parameters.

CONCLUSIONS

Using this new experimental approach, the effects of thermomechanical treatment history on the rheological measurements can be eliminated.

The viscosity of the melt increased with temperature and reached its greatest viscosity at about 140°C this is probably due to the increased proportion of the starch gelatinising and melting sooner as temperatures increase. Above 140°C dextrinisation of the starch reduces the viscosity of the melt.

Compared with temperature, the rate of product flow through the extruder and screw speed had less effect on the viscosity of the melt.

With a constant specific throughput, the effect of thermomechanical treatment history on the rheological measurements was reduced, but SME was affected significantly.

ACKNOWLEDGMENTS

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GRAIN QUALITY

Determination of Endosperm Characteristics of 38 Corn Hybrids Using the Stenvert Hardness Test

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ABSTRACT

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Grain hardness characteristics for a range of corn hybrids were measured using a modified Stenvert Hardness Test (SHT). A computer-based data logging and analysis system was constructed to obtain the milling time and transient power consumption during the milling process, from which milling energy and peak power demand were calculated. In addition, the time taken to mill 17 ml of flour (resistance time) and the height of the milled 20-g sample in the collection tube were recorded. The SHT measurements were correlated with the ratio of hard to soft endosperm and the bulk density of the grain. Thirty eight hybrid corn cultivars were tested. It was found that the testing system was simple and easy to use and error variation was low. Results indicate that significant variation in

the measured parameters occurred among the hybrids. SHT parameters were highly correlated with the ratio of hard to soft endosperm. Statistical analysis using analysis of variance showed that significant differences occurred among the hybrids for the variables measured. Principal component analysis showed that the milling energy and resistance time were the most effective SHT parameters for assessing grain hardness. Both parameters were highly correlated with the ratio of hard to soft endosperm and the bulk density of the grain. Cluster analysis indicated that endosperm characteristics were genetically controlled and associated with germplasm groups.

In the food industry, corn is milled to produce a range of flours and grits that can be further processed for snacks, breakfast cereals, and other cooked or extruded products. Corn grains are viscoelastic in nature and are heterogeneous in structure. Their mechanical properties change with time, temperature, moisture content, chemical composition, and microstructure within the kernel. Grain hardness is related to the protein content and kernel physical properties including kernel density, bulk density, and the ratio of hard to soft endosperm (Watson 1987). Although not widely used by the industry, classification of grain using properties such as milling characteristics and the ratio of hard to soft endosperm have been reported in the literature (Kirleis and Stroshine 1990, Watson 1987). Classification based on these properties could reduce product wastage by varying processing parameters according to grain quality.

A mature corn kernel consists of three major regions: the germ comprising the embryo and the scutellum; the endosperm; and a protective seed coat comprising the aleurone layer and pericarp. The endosperm constitutes 80–85% of the kernel dry weight and is 86–89% starch by weight. Two types of endosperm, floury and hard, are usually present, depending on the structure and amount of the protein matrix that surrounds the starch granules. In the hard endosperm, the protein matrix is thicker and remains intact on drying, binding the starch granules tightly together in a strong structure with a translucent glassy appearance. During drying, the floury endosperm collapses, tearing the thin protein matrix, resulting in loosely bound starch granules (Watson 1987). The endosperm often contains voids and is structurally quite weak (Duvick 1961). The outer region of the kernel tends to comprise hard endosperm while the inner region tends to comprise soft endosperm. In popcorns and flint types of maize, almost all the endosperm is of the hard type, while in the most floury cultivars, almost no hard endosperm is present. Cultivars for the food industry contain a mixture of both types. Generally, a higher proportion of hard endosperm is preferred for milling.

Breeding for improved grain hardness is possible by introgressing flint lines into germplasm with other desirable characteristics such as high yield. Grain hardness can also be increased by improving nitrogen fertilization (Hamilton 1951). The kernel properties vary with hybrid types, planting, environmental variables such as soil type, water, fertility, and climate, and management practices during and after harvesting (Watson 1987).

Despite the importance of corn hardness in industrial applications and the number of studies that have been published on the subject, there is no generally accepted standard for evaluation of corn milling characteristics. Different measurement systems have been applied to investigate the relationship between kernel hardness and other grain variables such as moisture content, drying conditions, the proportion of hard endosperm, and hybrid cultivar. The ratio of cross-sectional area of hard to soft endosperm has been measured (Hamilton 1951, Kirleis 1984) and while it represents the proportions of hard and soft endosperm, it does not necessarily measure endosperm hardness. Jindal and Mohsenin (1978) used a pendulum impact tester to determine dynamic hardness of corn grain and found it to be a function of moisture content. They also found it difficult to obtain a true measure of the energy absorbed by a specimen. As for measurements of the hard and soft endosperm areas, variation among kernels was high, and only one kernel could be tested at a time. The technique was time-consuming and impractical for a large number of samples.

Tran et al (1981) modified and used a Strong-Scott barley pearler to determine corn grain resistance to abrasion and to estimate grain hardness. The grinding energy and grinding index were found to be linear and inversely related to moisture content. Lawton and Faubion (1989) used a tangential abrasive dehulling device to measure kernel hardness. Their results also showed that the moisture content had a large effect on hardness and that care should be taken to develop a repeatable testing method. Significant differences were found between a pop and a dent corn. However, these devices only measure surface properties of the grain. For a representative estimate of grain hardness, the hardness of the entire kernel should be measured.

The Stenvert Hardness Test (SHT) has been reported as a useful approach for measurement of kernel hardness (Pomeranz et al 1985; 1986a,b; Pomeranz and Czuchajowska 1987). In this technique, 20 g of corn kernels at a specific moisture content are milled in a Culatti micro hammer mill. Three parameters were

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chosen to define the index of hardness: resistance time (the time required to mill 17 ml of meal); the height of the meal in the collection tube; and the weight ratio of coarse to fine particles in the resulting meal. They found that for three pairs of isogenic maize lines (dent and flint) and three yellow dent hybrids at 12% moisture, resistance time was highly correlated with kernel density and the proportion of coarse particles in the meal. Kirleis and Stroshine (1990) showed that the milling evaluation factor (MEF), a numerical index highly correlated with flaking grit yields, varied significantly among three corn hybrids of varying endosperm hardness. Furthermore, MEF was highly correlated with bulk density, kernel density, and Stenvert hardness measured as resistance time. Kernel hardness decreased as the grain drying temperature was increased between 27 and 93°C. Paulsen and Hill (1985) also showed increases in the yield of large flaking grits by selecting hybrids with low breakage susceptibility and high bulk density.

Mestres et al (1991) summarized the dry-milling properties of 18 corn landraces from Africa using a range of techniques and concluded that the ratio of hard to soft endosperm (vitreousness) was correlated with kernel density but not with protein content or dry milling properties including semolina recovery.

To the best of our knowledge, there is little information on the use of the micro hammer mill or any other techniques to systematically study the endosperm properties and milling characteristics of a wide range of corn hybrids. However, it does appear from the literature that the proportion of grits, and hence grain quality for processing, is correlated with the results of SHT and the ratio of hard to soft endosperm.

The objectives of this study were to systematically evaluate a number of parameters obtained from the SHT as a method of rapidly and reliably estimating grain hardness and to determine whether this is correlated with the ratio of hard to soft endosperm and bulk density for a wide range of hybrid cultivars. It was also aimed at determining whether variation for all the measured parameters occurred in commercially available hybrids and hybrids from New Zealand's maize breeding program.

MATERIALS AND METHODS

Corn

Thirty-five corn hybrids were machine-harvested from a research farm in the Manawatu region of New Zealand in June

TABLE I
Experiment Data for all Hybrids, Ranked for Bulk Density

Sample[a]	Sample Name	Origin[b]	Moisture Content (%)	Bulk Density (kg/hl)	Peak Power (watts)	Mill Time (sec)	Total Energy (J)	Sample Height (mm)	Resistance Time (sec)	Hard/Soft Endosperm Ratio
20	PFI×BS22-78	EF×HT	10.95	80.0	169	28.9	2,391	98.0	8.9	5.0
5	PFI×AS3-94	EF×HT	10.55	79.7	178	27.0	2,408	96.7	10.6	3.6
9	PFI×BS22-39a	EF×HT	10.10	79.2	180	26.4	2,223	94.7	7.9	4.5
27	PFI×BS22-39b	EF×HT	10.38	78.5	175	24.3	2,029	97.3	8.3	5.0
31	P3162	US Com Hard	11.20	78.2	172	26.4	2,373	100.0	8.5	7.4
6	PFI×NZS1-141	EF×HT	10.70	77.9	171	28.1	2,253	100.0	9.3	4.1
28	Hmv5313×PFI	HD×EF	9.85	77.9	156	30.4	2,802	100.0	9.8	3.9
21	PF2×NZ40	EF×HT	10.55	77.5	176	29.3	2,397	95.7	11.6	4.3
30	D1275×M378-80	ED×HT	11.25	77.3	179	25.7	1,936	100.0	7.9	1.8
1	PF2×NZ56	EF×HT	10.25	76.7	182'	26.7	2,258	94.3	10.8	3.2
3	PF2×NZ56	EF×HT	10.65	76.7	175	28.7	2,251	96.7	9.2	2.4
23	PFI×NZS3-32	EF×HT	10.50	76.5	161	28.1	2,302	98.3	8.0	3.1
38	Dea	E.Com Flint	12.40	76.2	163	30.8	2,660	100.0	9.8	4.0
2	A676×NZ56	US×HT	10.20	75.7	173	25.2	1,962	99.3	8.1	2.8
29	N190×NZ2	US×HT	10.90	75.6	175	24.9	1,909	98.7	8.6	2.5
22	D1275×NZ84	ED×HT	10.90	75.3	177	24.5	1,738	98.0	7.6	1.8
10	WGI207×BS22-22	US×US(NZ)	11.18	75.3	176	22.0	1,784	100.3	7.4	1.6
4	E1873×NZ3	EF×HT	10.30	75.2	178	25.8	2,113	97.0	8.6	2.2
12	WGI207×M396-14	US×HT	10.85	74.7	178	24.6	1,989	95.7	9.1	2.3
35	P3902	US Com Med-Hard	11.05	74.5	177	25.7	1,922	102.0	7.7	2.4
14	A665×M396-14	US×HT	11.15	74.4	176	23.7	1,848	100.7	7.1	1.8
13	WGI29×PFI	US×EF	9.65	74.2	169	26.3	1,956	96.3	8.7	3.0
8	P3787	US Com Med.	10.90	73.5	174	23.7	1,751	102.0	6.8	0.9
11	WGI207×M378-80	US×HT	10.50	73.4	173	24.8	1,863	98.3	8.7	1.7
19	A82-8×NZ84	US×HT	10.10	73.1	167	24.8	1,739	108.7	6.6	2.0
26	D1260×BS22-22	ED×HT	10.40	72.4	169	26.5	1,802	103.3	7.0	1.5
25	Furio	E Com Dent	10.25	72.2	162	26.5	1,778	95.3	7.0	2.0
33	P3751b	US Com Med.	10.45	71.2	174	26.2	1,793	99.3	6.4	1.7
36	PAC42	US Com Med.	10.35	70.9	166	25.6	1,723	107.3	6.8	1.4
16	P3514	US Com Hard	10.55	70.8	167	25.9	1,671	102.7	6.2	0.7
24	A82-8×MBS847	US×US	10.40	70.4	170	22.2	1,608	99.7	6.5	1.3
7	MBS847×NZS3-59	EF×HT	10.15	70.2	152	30.4	1,750	104.0	7.2	1.4
34	P3901	US Com Med.	10.00	70.2	163	23.9	1,830	104.3	7.6	1.3
17	P3515	US Com Hard	10.15	69.6	162	25.3	1,625	104.0	5.6	0.7
18	P3751a	US Com Med.	10.40	69.2	167	22.0	1,726	102.3	7.5	0.6
15	P3394	US Com Med.	10.35	69.1	168	24.2	1,607	103.0	6.0	0.9
32	P3585	US Com Med.	9.60	69.0	173	21.6	1,539	101.7	5.9	0.8
37	PX74	US Com Soft	10.60	68.6	171	25.5	1,332	103.3	4.2	0.2
	Test Statistic(F value)				3.8	3.4	21.6	2.0	18.2	34.9
	Probability > F				0.0001	0.0001	0.0001	0.0067	0.0001	0.0001
	Coefficient of variation (%)				3.5	8.4	6.1	4.2	7.9	18.7

[a] Samples 31 and 37 were produced in Gisborne region of New Zealand, sample 38 was produced in a southern New Zealand site; all other samples were produced in Manawatu region of New Zealand.

[b] EF = European flints, HD = Hungarian dents, ED = European dents, HT = highland tropical sources, US = United States Com Belt Dent, Com = commercial. All the US commercial hybrids are dent types, while the European commercial hybrids are either flint or dent types.

1993. To this set, samples of DeKalb brand PX74 and Pioneer brand P3162 produced in the Gisborne region of New Zealand were added along with a sample of Pioneer brand Dea produced in a southern New Zealand site. Immediately following harvest, the grain was slowly dried to ≈14% moisture and stored in a cool room at 7°C and 30% rh until required. During storage, the grain equilibrated with the air in the cool room resulting in a mean moisture content (MC) of 10.5% (range 9.6 to 12.4%) when tested. Before the testing, all samples were equilibrated to room temperature (25°C) in heavy paper bags.

The corn hybrids used are listed in Table I. They represented germplasm from several diverse sources. The highland tropical (HT) source originates from the CIMMYT (International Center for Maize and Wheat Improvement) maize breeding program and in this article refers to lines containing both highland tropical and corn belt dent germplasm (Eagles and Hardacre 1985). This material generally confers medium hardness properties to the endosperm. The lines of U.S. origins used can be of soft or medium hard endosperm, while the European dents (ED) and Hungarian dents (HD) are of medium hardness. The European flints (EF) have very hard endosperm. The commercial hybrids are of unknown origin although it is suspected that those labeled soft share similar parentage.

Analytical Methods

Moisture content and bulk density. Moisture content and bulk density (test weight) of the stored and dried corn samples were determined by Dickey-John GAC2000 grain analysis meter using a supplied program. Calibration for moisture was checked using oven drying techniques. Bulk density was recorded as kg/hectoliter and moisture as percent wet weight.

Hard to soft endosperm ratios. The hard to soft endosperm ratios in the samples were estimated by sectioning the kernels and measuring the areas of hard and soft endosperm presented at the cut surface (Kirleis et al 1984). Dried kernels were sectioned just above the top of the embryo region using a pair of secateurs. The hard to soft endosperm ratios for 10 kernels of each hybrid were calculated by measuring the average width and depth of the cut surface and the soft endosperm region using a pair of vernier calipers. From the approximate areas measured, the ratio of hard to soft endosperm was calculated. The method was time consuming and not practical for a very large number of samples, although it is the most direct measurement of the proportion of hard endosperm available. For this reason, it was used as the measurement of kernel hardness to which the other indirect measurements were compared.

Milling. The Stenvert Hardness Test was based on the method described by Stenvert (1974) and Pomeranz et al (1985). A 20-g sample of grain was ground using a Glen Creston micro hammer mill fitted with a 2-mm aperture particle screen. The mill speed was set to 7,500 rpm at empty but slowed substantially under load. This speed was considerably higher than recommended, but as the unit used was not fitted with a tachometer and as none was available at the time, a convenient setting was used. In future work, a purpose-built mill, Glen Creston micro hammer mill No5 equipped with a tachometer, will be used. The mill used in this work was equipped with a computerized data logging system to log the instantaneous electric power consumption during the milling test. From these data the transient peak energy and milling energy were determined.

Before collecting data, the mill was switched on and allowed to warm up by milling a set of five dummy grain samples. The set of test samples was then milled and the data logged. Data acquisition began automatically as soon as the power load increased above the unloaded power demand and continued until power consumption decreased to within 0.3 watts of the initial unloaded condition. In addition to these two measurements, resistance time, the time taken to mill 17 ml of meal, and the meal height in the collection tube at the completion of milling the 20-g grain sample were recorded. All data except the transient power curves were analyzed statistically using three to five replicates depending on the variable measured. Analysis of variance using the SAS procedure GLM (SAS 1988) generated F values and coefficients of variation (CV) appropriate for determining differences among the hybrids. The multivariate analysis of variance procedure for principal component analysis (SAS Princom) and cluster analysis (SAS Cluster) were applied to all data. Multivariate techniques were used to determine which of the variables, when considered together, contributed to variability among the hybrids, and how these variables, considered together, separated the hybrids into groups.

RESULTS AND DISCUSSION

The transient power consumption for two hybrids with either predominantly hard (P3162) or soft (PX74) endosperm during the SHT are presented in Figure 1. After the sample was dropped into
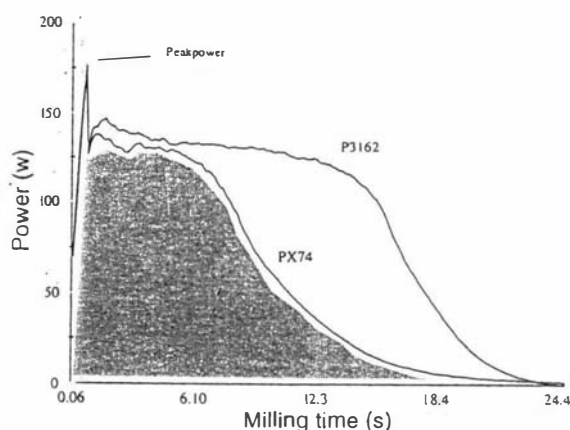
Fig. 1. True transient power changes of two typical hybrids.

TABLE II
Correlation Matrix Using Stenvert Hardness Test Parameters[a] and Moisture Content (MC)

	MC	BD	PeakP	MT	E	H	RT	H/S
MC	1.00							
BD	0.33	1.00						
PeakP	0.18	0.32	1.00					
MT	0.13	0.36	−0.26	1.00				
E	0.25	0.79	0.01	0.63	1.00			
H	−0.05	−0.43	−0.31	−0.17	−0.34	1.00		
RT	0.18	0.73	0.23	0.40	0.79	−0.50	1.00	
H/S	0.23	0.81	0.11	0.38	0.74	−0.32	0.62	1.00

[a] BD = bulk density, Peak P = peak power, MT = milling time, E = milling energy, H = height, RT resistance time, H/S = ratio of hard to soft endosperm.

the grinding chamber, the power consumption of the mill climbed rapidly to its peak value as a result of the sudden load. As the milling process progressed, more grits and flour were swept out through the particle screen, and the power consumption decreased. After 20–40 sec of milling, the grinding chamber was empty and the test was automatically terminated when the power consumption by the mill decreased to within 0.3 watts of the initial unloaded level. The duration of the test was highly dependent on the time required to eliminate the last of the grain from the milling chamber, although this had little impact on the milling energy. It is clear that the softer kernels of PX74 were more rapidly and easily reduced to a size that could pass through the screen than were the harder P3162 kernels. This resulted in higher total power consumption for milling the P3162 when compared to the PX74. This can be seen graphically from the area underneath the plots. Peak power consumption did not differ between the two hybrids.

CV within hybrids were low for most variables (Table I). For the hard to soft ratio, the CV was 18.7%, suggesting that for this variable, differences among hybrids will be harder to resolve. From the F values (Table I) and their probabilities at the 5% level, it is evident that significant differences occurred among the hybrids for all variables. F values were lower for sample height, peak power, and milling duration as were CV, suggesting that overall variability for these variables was low and that they may be less useful in separating the hybrids for milling characteristics. As moisture content and bulk density measurements were unreplicated for these variables due to small available sample sizes, statistical tests could not be conducted. However, experience with these measurements has proven that errors are inherently very low (CV < 4%) and therefore, differences in bulk density >3kg/hl are probably significant. The reason for differences in moisture content are unknown as all samples were equilibrated under the same conditions. We suggest that differences in endosperm chemistry may be involved. Although, moisture content has a significant

effect on hardness measurements (Tran et al 1981) the differences found (Table I) are probably too small to have a significant effect.

From the data in Table I, it can be seen that some trends exist. Higher bulk densities are associated with hybrids with higher milling energies (E), greater resistance times (RT), and higher hard to soft endosperm ratios. Rankings on bulk density of entries that are repeated but sourced from different areas of the field, P3751 and PF1×BS22-39, are similar, as are other measured parameters for these pairs of hybrids.

Since one of the objectives of this work was to determine whether the ratio of hard to soft endosperm was correlated with indirect but faster measurement techniques for grain hardness, considerable emphasis was placed on SHT parameters and bulk density which have high correlation coefficients with the ratio of hard to soft endosperm areas (H/S).

The Stenvert properties, milling energy and resistance time were highly correlated with H/S (Table II); therefore these properties are likely to be good estimators of grain hardness. However, H/S had a higher correlation coefficient with milling energy (E) (0.74) than with resistance time (0.62) suggesting that E was a better predictor of the proportion of hard endosperm. In previous publications (Pomeranz et al 1985; 1986a,b), only resistance time was measured. Bulk density was also highly correlated with milling energy (0.79), resistance time (0.73), and H/S (0.81). It is therefore a simple estimator of grain hardness. Correlation coefficients for peak power, milling time and the height of the meal in the collection tube with H/S were ≤0.5 and are therefore less useful estimators of grain hardness.

A plot of E against H/S reveals some anomalies (Fig. 2) that also occurred for resistance time and bulk density with H/S (plots not presented). The hybrids, P3162, PF1×BS22-78, and both entries of PF1×BS22-39 had high H/S ratios, but their milling energies, bulk densities, and resistance times were lower than expected. Deviations from the expected correlation may be due to these hybrids having softer kernel texture. This may be caused by differences in the properties of endosperm from different hybrids. An alternative explanation is that the proportion of hard endosperm at the measured section of the kernel is not an accurate estimate of the volumes of hard and soft endosperm. If this is true, we suggest that the SHT is a more accurate assessment of kernel hardness than the ratio of hard to soft endosperm.

SAS PCA is a multivariate technique used to reduce the number of variables in a dataset (SAS 1988). Therefore, this technique produces new variables called principle components (PC) from correlated variables that account for the total variation in the data. Typically, most of the variation is accounted for by the first one or two PC. The eigenvectors generated by PCA indicate the contribution of the original variables to the variation accounted for by each of the PC. Due to the high correlation among some of the variables obtained in this study, it was considered appropriate to apply PCA to combine the correlated variables to more accurately express a trait, in this case kernel hardness.

PCA revealed that 52% of the variability in the data could be accounted for by the first principal component (PC1) and only 22% by PC2. PC2 and the other principal components are therefore ignored. From PCA, bulk density, milling energy, and resistance time had the larger eigenvectors and are, therefore, the ma-
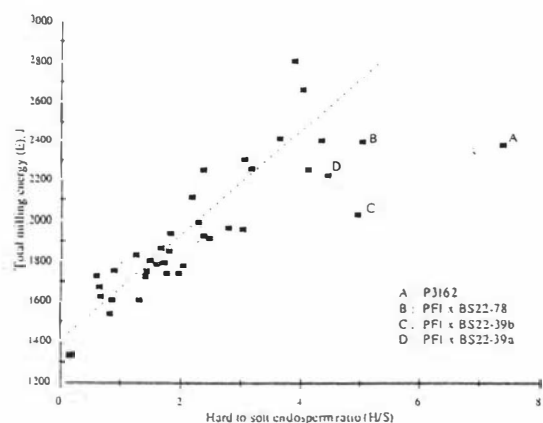

Fig. 2. Relation between the hard to soft endosperm ratio and the milling energy of the 38 hybrids. (Points represent 38 hybrids listed in Table II.)

TABLE III
Correlation Matrix of Stenvert Hardness Test Parameters[a] and Principal Component (PC1)

	PC1	BD	E	RT	H/S
PC1	1.00000				
BD	0.91387	1.00000			
E	0.91218	0.78920	1.00000		
RT	0.86538	0.73254	0.78775	1.00000	
H/S	0.83847	0.80877	0.74099	0.61823	1.00000

[a] BD = bulk density, E = milling energy, RT resistance time, H/S = ratio of hard to soft endosperm.

TABLE IV
Rankings of Principal Component PC1 and Other Parameters[a]

Sample Names	PC1	Ranking				
		PC1	BD	E	RT	H/S
PF2×NZ40	3.38	1	8	4	1	5
Dea	3.07	2	13	2	5	7
PF1×AS3-94	2.99	3	2	3	3	9
PF2×NZ56	2.59	4	10	8	2	10
PF1×BS22-78	2.54	5	1	5	9	2
Hmv5313×PF1	2.39	6	7	1	4	8
PF2×NZ56	2.10	7	11	10	7	15
PF1×BS22-39a	1.82	8	3	11	18	4
PF1×NZS1-141	1.76	9	6	9	6	6
P3162	1.71	10	5	6	14	1
PF1×NZS3-32	1.09	11	12	7	17	11
WGI207×M396-14	1.06	12	19	14	8	17
E1873×NZ3	0.97	13	18	12	13	18
E1386×BS22-39b	0.97	14	4	13	15	3
D1275×M378-80	0.83	15	9	17	19	21
N190×NZ2	0.56	16	15	19	12	14
WGI29×PF1	0.34	17	22	16	10	12
A676×NZ56	0.18	18	14	15	16	13
WGI207×M378-80	0.06	19	24	20	11	25
D1275×NZ84	0.05	20	16	30	21	23
P3902	0.02	21	20	18	20	16
A665×M396-14	−0.43	22	21	21	26	22
WGI207×BS22-22	−0.51	23	17	25	24	26
Furio	−0.55	24	27	26	28	19
P3751b	−1.01	25	28	24	33	24
P3787	−1.09	26	23	27	29	32
D1260×BS22-22	−1.15	27	26	23	27	27
MBS847×NZS3-59	−1.52	28	32	28	25	28
P3514	−1.83	29	30	33	34	36
P3901	−2.01	30	33	22	22	31
A82-8×MBS847	−2.06	31	31	35	32	30
P3751a	−2.15	32	35	31	23	37
PAC42	−2.23	33	29	32	30	29
A82-8×NZ84	−2.27	34	25	29	31	20
P3394	−2.56	35	36	36	35	33
P3515	−2.76	36	34	34	37	35
P3585	−3.08	37	37	37	36	34
PX74	−3.28	38	38	38	38	38

[a] BD = bulk density, E = milling energy, RT resistance time, H/S = ratio of hard to soft endosperm.

jor contributors to the variability accounted for by PC1. Moisture content, milling time, peak power, and sample height contributed less to the variation accounted for by PC1.

Table III shows that PC1 is highly correlated with bulk density, milling energy, resistance time, and the ratio of hard to soft endosperm. In all cases, the correlation of these variables with PC1 are higher than the correlation of the variables with each other. It can therefore be concluded that PC1 is a better estimate of grain hardness than the variables considered separately. The rankings associated with PC1 in Table IV are considered to be the best estimators of kernel hardness.

Cluster analysis (SAS 1988) was then applied to separate the hybrids into groups based on all the variables (analysis not presented). Two major groups were revealed, one with hard endosperm, which includes the first 12 hybrids in Table IV, and a softer group comprising the remaining 26 hybrids. The group with hard endosperm comprises hybrids which are known to contain a high proportion of flint germplasm, either from the inbreds PF1 and PF2 or of unknown origin for the commercial hybrids P3162 and DEA. It is interesting to note that most of the hybrids occurring in this group are based on crosses between flint and highland tropical germplasm. The crosses of highland tropical germplasm with nonflint lines are intermediate in hardness, suggesting that highland tropical germplasm may be a useful source of grain hardness. These hybrid combinations also have good grain yield potential in the cool temperate New Zealand climate (Eagles and Hardacre 1985). The hybrids with softer grain often contain the inbreds MBS847 and A82-8 as one, or are the commercial hybrids

which are commonly used for grain production in New Zealand. It therefore appears possible to improve kernel hardness by choosing the kernel properties of the parents of the hybrids.

CONCLUSIONS

From the data presented here, it is possible to see that significant and quite large differences in the hard to soft endosperm ratio in the maize hybrids studied was closely correlated with grain hardness measured by the modified Stenvert Hardness Test and bulk density. The SHT is, therefore, proven to be a quick and simple method of comparing the endosperm hardness of diverse maize hybrids at constant moisture content. In addition, the test has the benefit of measuring a large number of kernels in each test so providing a better estimate than tests based on single kernels.

The presence of outliers in the correlation between milling energy and the ratio of hard to soft endosperm suggests that all measurements of kernel hardness should be based on a mechanical milling test such as that described here and not on a visual assessment of the ratio of hard to soft endosperm area.

In New Zealand, commercially available hybrids are capable of high yields. However, the grain tends to be soft and less suitable for milling for grit production. The challenge has been to discover germplasm combinations that result in commercially acceptable yields with good milling quality. Table IV shows that hybrids in which one of the parents was from the flint group, or to a lesser extent of highland tropical origins, had higher values for PC1 and had a higher proportion of hard endosperm.

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