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**THE EFFECTS OF WATER ACTIVITY,
PARTICLE SIZE DISTRIBUTION AND
FREE FAT CONTENT ON FLOWABILITY OF
GRATED AND DRIED PARMESAN CHEESE**

***A THESIS PRESENTED IN PARTIAL FULFILMENT OF THE
REQUIREMENTS FOR THE DEGREE OF MASTER OF TECHNOLOGY
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ABSTRACT

A study was made of the effects of water activity, particle size distribution and free fat content on flowability of Grated and Dried Parmesan cheese made by Greenwood Valley Cheese Company (GVC) and by Kraft. The three parameters were modified to span a wide range around the original level of each.

Water activity (A_w) was decreased / increased through water sorption. A range of particle sizes for each of the samples was obtained by separating the samples into two fractions and then combining them together to different levels. Samples were sieved through a stack of sieves with different mesh sizes and particle size was expressed in terms of median particle size. A range of free fat levels was obtained by spraying samples with anhydrous milk fat (AMF). The response of flowability to changes in these parameters was assessed. Flowability was measured by using a rotating drum.

The results in the preliminary study showed that flowability increased with increasing moisture content from 18 to 22%, which is in contradiction to the normal expectation from the literature. Therefore, the hypothesis: flowability increases with increasing moisture content / A_w was proposed.

To test the validity of the hypothesis, several trials were conducted. The results in the first trial showed that flowability increased with increasing A_w from 0.66 to 0.79 for both GVC and Kraft products. The results also revealed a possible critical A_w between 0.79 to 0.83 at which the products could have their optimum flowability.

In the next trial, additional values in the A_w range between 0.79 to 0.83 were included. The results showed that flowability increased with increasing A_w from 0.67 to the critical A_w value and dropped down above this value. The critical A_w values for GVC products were determined at 0.80 ± 0.01 . Kraft's product did not show this increasing trend or the critical A_w value.

A series of commercial samples with different A_w levels from two of each of GVC products were chosen to test the hypothesis. Variation in water activity naturally occurred during processing due to the slight changes in drying conditions. The results obtained on these commercial samples showed flowability increased with increasing A_w from 0.69 to 0.77. Therefore, the hypothesis has been proven.

The study on the effect of particle size shows flowability also increased with increasing median particle size for both GVC and Kraft products. Of the methods chosen to plot cumulative undersize versus sieve size, linear regression is suggested rather than simply joining the points. This is because all the points are taken into account when linear regression is used.

The study conducted on the effect of free fat shows that flowability decreased with increasing free fat levels from 16.8 to 26.3%. The sieve analysis results on the samples with different free fat levels show that median particle size increased from 721 to 1476 μm with increasing free fat levels from 16.8 to 26.3%. This was presumably caused by particle aggregation. The results show that the positive effect of particle size on flowability could not counteract the negative effect of free fat. Further study is required to confirm the net effect of free fat and particle size.

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CHAPTER 1

INTRODUCTION

Parmesan is the name commonly used to describe a group of very hard cheese varieties that originated in the Po River valley of Italy. Locally, these cheese varieties are generally called Grana, or specially named after the city of manufacture (i.e. Parmesan comes from Parma, Reggino from Reggio, etc.). These cheeses are characterized by a granular texture, sharp flavour, hard body, very small eyes, long shelf life and excellent shipping properties (Sanders, 1953).

The traditional Italian manufacturing methods have been gradually and continually modified for commercial production as follows (Anonymous a, 1973):

- The cheese is manufactured by some producers on a continuous basis using pasteurised or heat treated and standardised milk.
 - The cheese is produced by using various starters in addition to whey starters.
 - The size of the cheeses generally ranges from 10 to 12.7 kg rather than the traditional 27 kg on average.
 - Ripening is controlled by mechanical refrigeration.
 - The cheese is coated with wax rather than oil or grease.
 - The cheese is cured for 10 to 14 months rather than 16 to 24 months in the traditional method and a shorter curing method with only 6 months is being trialed (Anonymous b, 1999).
-

Parmesan production in the United States is a growing segment of the cheese industry. For example, the production of Parmesan and similar cheese varieties increased from 30,750 tonnes in 1979 (Ferris, 1981) to about 68,000 tonnes in 1997 (Anonymous c, 1999). Kraft is a major manufacturer of Parmesan in the U.S. with production of about 27,200 tonnes annually, followed by Sartori at 12,700 tonnes per year (S. Dybing, personal communication).

Parmesan cheeses are marketed in four basic forms: as pieces cut from larger wheels by local retailers in grated form, in shredded form, or as an ingredient of another food (Anonymous a, 1973). Parmesan is normally consumed as a table cheese in very small quantities, so virtually all Parmesan is grated before it is consumed. Most of the Parmesan purchased by consumers is either grated, dehydrated and packaged or shredded and packaged at the higher moisture level (Anonymous a, 1973). After being grated and dehydrated, the so-called Grated and Dried Parmesan or Grated Parmesan is packed into consumer sized canisters (eg. 227g). Dehydration reduces the moisture content of the product to 12 – 18%, an average 17.7% as shown in Table 1.1.

Table 1.1 The compositions of Hard and Grated and Dried Parmesan

	Hard (mature) Parmesan (%)	Grated and Dried Parmesan (%)
Moisture	29.2	17.7
Total solids	70.8	82.3
Fat	25.8	30.0
Total protein	35.8	41.6
Carbohydrates (by difference)	3.2	3.7
Ash (Includes added salt)	6.0	7.0

(Kosikowski and Mistry, 1997:69)

Drying to these moisture contents greatly enhances the shelf life, allowing Grated Parmesan to be widely marketed in canisters (e.g. 227g) for use on soups, salads, pizza and pasta. At 12 to 18% moisture there is a problem of clumping or agglomeration of the grated cheese product, which hinders consumers' acceptability. Clumping is easily observed in the canister and constitutes a serious defect. Aggregates range from small clumps to large lumps. The small clumps may be disrupted and eliminated when shaking the canister. However, some lumps aggregate into hard pieces that cannot be broken apart with acute shaking. These lumps hinder the flowability of product out of the canister during shaking. Therefore, flowability is an indirect measure of clumping. Also, flowability is a major criterion by which consumers determine product quality. Hence, flowability constitutes a vital product characteristic, which is even more important than clumping.

The overall objective of this study is to determine the fundamental factors affecting flowability in Grated and Dried Parmesan cheese. The production of Grated Parmesan cheese creates a food powder. Therefore, the theories presented in the literature review should describe the factors that control flowability in this powder product. Three major factors known to significantly affect flowability in food powders include the water activity (A_w) of the particles, the particle size distribution and the amount of free fat present on the particle surface. This is a fundamental study on the flowability of Grated and Dried Parmesan cheese. The study was divided into three sections to specifically focus upon the following factors:

- Determining the effect of A_w on flowability
- Investigating the effect of particle size distribution on flowability
- Illustrating the influence of free fat on flowability

Other properties such as porosity of powders and storage conditions are not included in this study.

CHAPTER 2

LITERATURE REVIEW

2.1 Mechanism of Flowability

Flowability is a relatively complex phenomenon that involves simultaneous and complementary internal and external interactions. The internal factors that influence flowability include particle density, bulk density, particle shape, particle size and size distribution, moisture content, fat content and static charge (White et al., 1967). External factors that affect flowability include temperature, relative humidity and storage time (Provent et al., 1993). For cohesive powders, the system geometry (the bin angle and aperture diameter) plays a decisive role in establishing the flow regime, i.e., mass or funnel flow, and its rate and stability (Peleg, 1983).

The ability of a powder to flow depends on the relative movement of a bulk of particles among neighbouring particles or along the surface of the container wall (Peleg, 1977). Differences between the properties of solids and liquids allow significantly different mechanisms to control the flow of these phases. Specific differences (according to Jenike, 1967) include:

- Solids can transfer shearing stress under static conditions, which produces a static angle of friction greater than zero and allows solids to form dome piles.
 - Many solids can possess cohesive strength following the application of pressure, thereby retaining a shape under load and forming a stable dome.
 - Solids can possess shearing stress being consolidated independently of the rate of shear and dependent on the mean pressure acting within the solids.
-

Therefore, the treatment of a powder's gravitational flowability must be based upon theories of solid mechanics and not upon hydrodynamics (Peleg, 1983). The forces involved are gravitational forces, friction, cohesion (interparticle attraction) and adhesion (particle-wall attraction) (Retsina and Coucoulas, 1988).

2.1.1 Cohesion / cohesiveness and flowability

The forces involved in the gradational flowability allow the classification of non-cohesive (free-flowing) powders and cohesive powders (Peleg, 1977). Peleg (1977) defined non-cohesive (free-flowing) powders as powders in which interparticle forces (cohesion) were negligible and the major obstruction to flow was the internal friction. Alternatively, interparticle forces played a significant role in the powder bed mechanical behaviour in cohesive powders.

Both cohesion and cohesiveness quoted by Buma (1971c) from Brown and Richard (1970) is defined as "the sticking of the components of a bulk solid to one another and is conveniently assessed as the resistance of a powder to shear at zero compressive normal load". According to Peleg (1977), cohesiveness reduces the flowability or stops the flowability altogether. This is supported by other researchers such as Pilpel (1970), Retsina and Coucoulas (1988) and Schubert (1987) Greater cohesiveness reduces the flowability of a powder (Pilpel, 1970).

2.1.2 Mechanism of flowability

The various mechanisms by which particles are attracted to each other and are interlocked together have been discussed by Pietsch (1969) and Schubert (1981). Schubert (1987) described these theories for food powder systems, which are illustrated, in Figure 2.1.

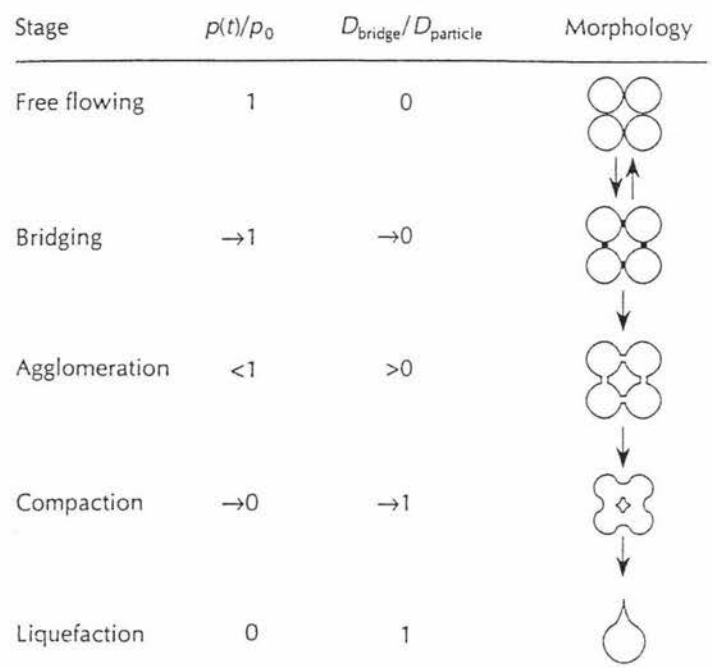


Figure 2.1 Mechanisms of adhesion between solid particles and a solid plate in a gaseous environment
(Schubert, 1987:9)

2.1.2.1 Formation of solid bridges

Sintering, chemical bonding or crystallisation may promote the formation of solid bridges between particles. The crystallisation of amorphous particle compounds frequently produces an undesirable stickiness in the food powders (Schubert, 1987). This crystallisation of amorphous particles has been widely studied as a function of temperature, time and moisture content (Jouppila and Ross, 1994a; 1994b; Lloyd et al., 1996; Schenz, 1995; Slade and Levine, 1991). Caking and clumping may be defined differently. Caking is caused by amorphous sugar crystallisation in food powders, producing hard rocky pieces rather than soft clumps (Aguilera et al., 1995; Brennan et al., 1971; Chuy and Labuza, 1994; Downton et al., 1982; Flink, 1983; and Jouppila and Roos, 1994a; 1994b). Lactose crystallisation is the main cause of caking in milk powder products such as whole milk powder, whey powder and infant formula during processing, handling and storage (Levine and Slade, 1986). Lactose crystallisation is not a significant problem in Grated and Dried Parmesan cheese because this product contains virtually no lactose (Kosikowski and Mistry, 1997). Therefore, the ability of lactose crystallisation to promote caking is not discussed in this review.

2.1.2.2 Liquid bridges

Liquid bridges are bonds formed between particles by liquids. The formation of liquid bridges depend upon the surface tension, the wetting angle, the adsorption characteristics of the powder and upon the amount and viscosity of the binding liquid (Retsina and Coucoulas, 1987). Schubert (1981, 1987) described the contribution of liquid bridges to mechanical strength. Pietsch (1969) found that the formation of liquid bridges at the coordination points between individual particles accounted for setting or agglomeration of particulate matter during storage and material handling. Peleg (1977) stated that liquid bridges could be formed when there is a liquid phase on the particle surface, promoting the formation of liquid bridges by the following factors:

- Moisture absorption (hygroscopic materials),
- Fusion (e.g., of fatty components),
- Chemical reactions that liberate liquid (e.g., browning),
- Excessive liquidation of ingredient (e.g., flavouring oils),
- Moisture liberation during the crystallisation of amorphous compounds.

2.1.2.3 Capillary liquid formed in completely or partly filled pores of the agglomerates

Isolated liquid bridges are formed only when there is relatively small quantities of liquid in the agglomerate. As the liquid content is increased, a transition zone is reached in which the liquid bridges and the pores filled with liquid merge to saturate the system (Schubert, 1987).

2.1.2.4 Van der Waals' and electrostatic forces

Van der Waal's and electrostatic forces are two major factors that may create adhesions without material bridges (Schubert, 1987). Van der Waals forces have an effective range of up to about 100Å (Peleg, 1977). At a larger distances, the Van der Waal's forces become significantly weaker (Schubert, 1981). Their effect may only become significant in very fine food powders not only because of the number of contact points between particles, but also because the surface energy per unit weight increases with the size reduction of the powder (Peleg, 1977). White et al. (1967) showed that various food powders did not flow when their particle size was less 120 µm, despite the fact that

these particles were dry, thereby showing the contribution of such short-range forces to clumping in food powders (Peleg, 1977).

Particles with opposing charges invoke electrostatic adhesion. Excess charges may be already present or may arise only when the particles come into contact due to differences in the values of the electron work function (Schubert, 1987). Although individually these forces are much weaker than other bonding mechanisms, they play an important role in dry state initial adhesion. Furthermore, they often form a first stage to granule growth by holding particles in flocculation and allowing other stronger bonds to form (Retsina and Coucoulas, 1988).

2.1.2.5 Mechanical Interlocking

Particles maintaining irregular or fibrous shapes can become mechanically interlocked (Mohsenin, 1986). Vibration or pressure may orientate such particles into physical aggregation or clumps. However, this does not appear to be a dominant factor affecting the flowability of food powders (Peleg, 1977).

2.1.2.6 Functions of flow conditioners

Flow conditioners are finely divided solids that increase the ability of a powder to flow and/or inhibit the tendency of this powder to cake (Peleg and Hollenbach, 1984). The mechanisms by which conditioners affect flowability include:

- Physical separation of particles, either by interruption of liquid bridging for cohesive powders or by lubrication for free-flow powders;
- Competition for adsorbed water with the host powder;
- Cancellation of electrostatic charges and molecular forces (Peleg and Hollenbach, 1984).

2.2 Factors Affecting Flowability

2.2.1 Particle shape, particle size and particle size distribution

2.2.1.1 Particle shape

Particle shape affects many secondary physical properties relevant to powder handling such as bulk density and failure properties (Svarovsky, 1990). Retsina and Coucoulas

(1988) pointed out that particle shape has a pronounced effect on the flowability: for example, particles with a fibrous, highly assymetric shape can lock together and inhibit free flow. The irregular shapes and rough surfaces of particles increase the number of contact points between adjacent particles (Retsina and Coucoulas, 1988). Peleg (1977) believed that mechanical interlocking of particles could not be a dominant factor that hinders flowability. Although particle analysis can provide highly detailed and accurate descriptions of particle shapes, efforts to relate the various parameters showing shape-describing parameters to powder bulk behaviour are relatively rare (Svarovsky, 1990).

2.2.1.2 Particle size and particle size distribution

If the food particles are spheres or cylinders, then the particle size can be described satisfactorily by one or a few measurements. However, most food particles have an irregular shape, so any measurable physical property that correlates with characteristic dimensions is used to obtain an equivalent dimension. Schubert (1987) defined equivalent dimension as “the dimension of an imaginary spherical particle having the same value in the physical properties measured as the irregularly shaped particle”. Unfortunately, very few particulate systems have uniform sizes. For example, dry milk particles produced in commercial spray dryers range from 10 to 100 μm (Hayashi et al., 1969). Similarly the size of grain-based, bran products range from more than 2,380 to less than 149 μm (Neale, 1997). Large numbers of such particles must be measured to produce an accurate size distribution. Svarovsky (1990) claimed that the cumulative distribution as well as the frequency distribution are usually employed in preparing such particle size distributions.

When a population of particles is described by a single number, many different means occur, such as arithmetic, geometric, quadratic, cubic, bi-quadratic and harmonic (Svarovsky, 1990) as illustrated in Figure 2.2. Figure 2.2 also shows that two different size distributions may have an identical arithmetic mean, although all the other means are different. A comparison made on the basis of the arithmetic mean shows identical particle distributions, while a comparison based upon any of the other means shows different to significantly different size distribution. Therefore, choosing a proper mean

or a group of means is very important in obtaining the correct conclusion (Svarovsky, 1990).

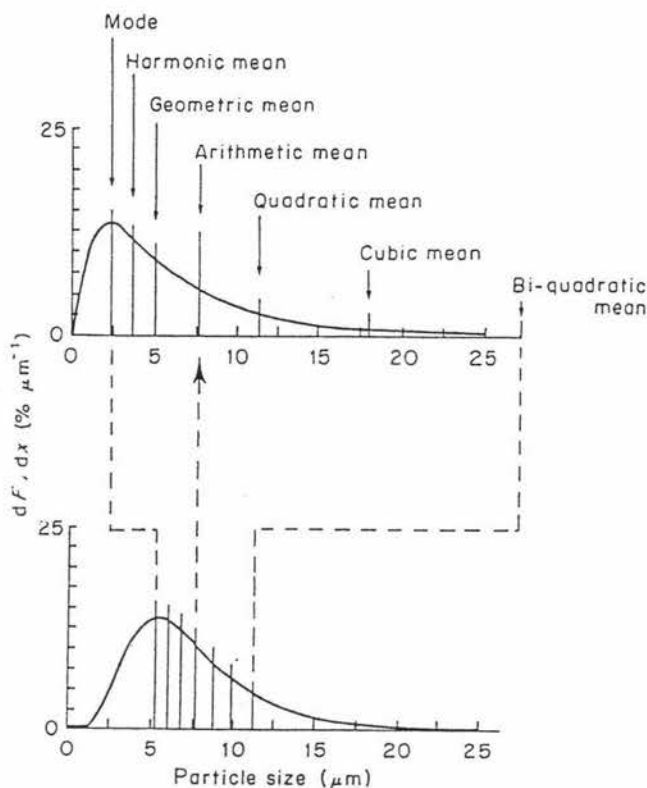


Figure 2.2 Two different size distributions with the same arithmetic mean

Where F: fraction or percentage oversize or undersize

x: particle size

dF / dx is defined as distribution frequency

(Svarovsky, 1987:49)

The importance of the size distribution is shown by the ability of these parameters to correlate with many individual particle properties and bulk characteristics. The particle size distribution and shape greatly influence flowability. The more uniform the size and shape of a mass of particles, the more flowable the particles are likely to be (Lascelles, 1975; Retsina and Coucoulas, 1988; Tuohy, 1989; and Yan and Barbosa-Canovas, 1997). Teunou et al. (1999) observed that product flowability typically decreases as the particles become finer. Woodhams and Murray (1974) pointed out that larger particles are required to improve flow properties of spray-dried milk powders. Retsina and

Coucoulas (1988) also mentioned that a larger range of particles proportionally reduce the flowability of powders. According to Lascelles (1975), a random mix of particles with different shapes and sizes can form strong agglomerates by providing many fine particles to fill the voids between large particles. Small and/or rough particles lock together and wedge between each other to stabilise the mass, even at low packing pressures. Interlocking binds particles across cleavage planes, as particles flow around each other to give a plastic and thus cohesive structure. Masses of particles of uniform size truss one another, so particles cannot slide with respect to each other, thereby reducing plasticity and minimizing surface contact. This suggests that removal of fine particles will aid flowability (Lascelles, 1975).

2.2.2 Particle density, surface area and porosity

Particle density is defined as the total mass divided by total particle volume. Depending on the method of defining total volume, other densities can be defined as follow:

- True density when the volume measured excludes both open and closed pores;
- Apparent density when the volume measured includes closed pores or bubbles of gas within the particle;
- Effective particle density when the measured volumes includes both the closed and the open pores (Svarovsky, 1987).

Dried milk particle density is the density of the dried milk particles, which includes the entrapped air and the volume of the voids between the particles (King, 1965). Buma (1965) observed that sprayed dried milk powders usually consist of particles with variable particle density due to vacuoles of different size present in the majority of the particles. The true densities of dry milk solids are determined by gas or liquid displacement techniques (Buma, 1965). The true density of milk powders and the methods used to measure the density of milk solids have been reviewed by Buma (1965). True densities of 1.28-1.32 and 1.46-1.68g/cm³ are reported for whole milk powder and skim milk powder solids respectively (Tuohy, 1989). Powder particle densities are somewhat lower due to the occlusion of air during spray drying (Tuohy, 1989). Factors affecting the occluded air content have been discussed by Pisecky (1978).

Particle structure can be estimated by calculating the surface area from the particle size distribution and by assuming a general shape for the particles (Retsina and Coucoulas, 1988). Additionally, the ratio S_v , defined as surface area per unit volume of particles is inversely proportional to particle size. Therefore, small particles have a large S_v (Schubert, 1987). For example, if a material with 1cm^3 in volume is subdivided into equal spheres of $1\text{ }\mu\text{m}$ diameter, the surface volume is more than 3m^2 (Schubert, 1987). This allows many free-flow agents to adsorb considerable amounts of water as a result of their very large surface area, thence improving the flowability of powders (Schubert, 1987).

Porosity has been defined as the ratio of void volume to total volume of the powder (Schubert, 1987):

$$\varepsilon = V_v/V_t = 1 - V_s/V_t \quad (1)$$

Where ε : porosity

V_s : solid volume

V_v : void volume

V_t : total volume

Particle porosity may affect moisture movement in solids. The movement of water out of the pores in particles creates moisture films surrounding these particles to produce clumping (Lascelles, 1975). Buma (1971a) discovered a correlation between particle porosity and the free fat content in spray-dried whole milk powder. Less porous powders apparently reduce the movement of moisture and fat within whole milk powder particles, thereby producing a powder with great internal strength, and decreasing the potential for cohesion (Lascelles, 1975). Buma (1971a) concluded that the particle porosity is of major importance for the extraction of milk fat from whole milk powders. Photographs taken with a scanning electron microscope shows cracks and pores in particles with high porosity, whereas similar photographs of less porous powder particles showed only surfaces folds and occasional cracks (Buma, 1971a).

Currently, particle density or porosity has not been directly related to the flow properties, with the exception that porosity influences the release of moisture and fat within the powder particle. Moisture and fat reduce flowability and cause clumping.

2.2.3 Bulk density and tapping

Bulk density is defined as the particle mass that occupies a unit volume (Svarovsky, 1987). Peleg (1983) pointed out that the bulk density of food powders depends on the combined effects of interrelated factors including the intensity of attractive interparticle forces, the particle size and the number of contact points. Therefore, changes in any of the powder characteristics may significantly alter the powder bulk density. Lascelles (1975) found that bulk density decreased with increasing temperature, due to expansion of the particles. Pisecky (1978) presented a detailed discussion on the influence of various factors on bulk densities of milk powders. Determinations of bulk density are highly influenced by the prescribed tapping procedure (e.g., tapping times), by the method for filling the measuring cylinder and by the number of times the cylinder is tapped manually or mechanically. According to Pisecky (1978), the bulk density is subdivided into:

- Poured bulk density – gently filling without tapping;
- Loose bulk density – tapped 10 times;
- Tapped bulk density – tapped 100 times;
- Tapped to extreme bulk density – tapped 1250 times.

The differences between the various values are small if the powder has a good flowability. On the contrary, the flowability of agglomerated powders is substantially affected by differences in bulk density due to mechanically poorly stable agglomerates that are crushed by intensive tapping (Pisecky, 1978).

Sone (1972) reported that in a variety of food powders there is a relationship between the volume reduction fraction γ_n and the number taps (N) expressed as:

$$\gamma_n = (V_o - V_n)/V_o = abN(1 + bN) \quad (2)$$

Where V_0 : the initial volume;

V_n : the volume after N taps;

a & b : constants.

The formula can be expressed as follows:

$$N/\gamma_n = 1/ab + N/a \quad (3)$$

so that a plot of N/γ_n against N is linear. Sone (1972) found only minor volume changes either in whey powder or in powdered skim milk for instant use evaluated by tapping because the apparent density in the loose package at an initial stage is relatively high in these samples. Sone (1972) related the volume changes to the porosity of the powder and to interactions between powder particles. Peleg (1977) suggested the bulk density changes due to tapping may be an index to the presence of attractive forces and friction. Packed bulk density is the best-defined and most widely referenced bulk density measurement for powders (Niro Atomiser, 1978). However, poured, loose and other bulk densities as defined by Pisecky (1978) are used in the milk powder industry. Bulk density plays an important role during packaging and shipping (Woodhams and Murray, 1974). For example, a powder with a low bulk density requires a larger bag and occupies more shipping space per tonne than a powder with a higher bulk density. Bulk density is not significant in this study because all the samples are packed into rigid canisters rather than large flexible packages before shipment.

2.2.4 Angle of repose and other handling angles

According to Svarovsky (1987), the angle of repose is defined as the angle of the free surface of a pile of powder to the horizontal plane. The magnitude of this angle is influenced by frictional forces (especially in free-flowing powders) and affected by interparticle attractive forces, a factor that dominates wet and cohesive powders (Peleg, 1983). The variations of the angle of repose with moisture content are due to the surface layer of moisture that surrounds each particle. Such layers of surface moisture create surface tension effects that become the major factor holding aggregates of solids together (Mohsenin, 1986). Depending on the conditions under which the pile has been created and the method with which the angle is measured, somewhat different angles can be obtained for the same powder (Svarovsky, 1987). However, the angle of repose is widely quoted as an index of the flowability of powder. Powder with a lower angle of

repose proportionally shows high flowability (Tuohy, 1989). Other handling repose, such as drained angle of repose, angle of slide, covering angle and angles of spatula are obtained from the angle of repose. Procedures for obtaining these angles are discussed by Sone (1972) and Mohsenin (1986).

2.2.5 Moisture content, A_w and moisture sorption isotherm

2.2.5.1 Basic definitions and fundamental properties

The term, “moisture content”, implies the total amount of water present in a sample as shown by quantitative analysis. Moisture content determination is essential in meeting product compositional targets and nutritional labelling regulations, specifying recipes and monitoring process performance. However, there are limitations of using the measurement of moisture content to show the intensity with which water associates and interacts with other food components (Aqualab Water Activity Meter Operator’s Manual). Measurements of water activity A_w frequently are much more accurate in showing the effect of moisture upon product characteristics than measurements of moisture contents. Water activity is defined as the ratio of the vapour pressure of solution and solvent (Troller and Christain, 1978). Karel (1973) pointed out that not all the water in foods is free to act as solvent, because some of this water is bound to specific groups of insoluble components. The A_w of a food containing substantial amounts of insoluble constituents cannot be calculated accurately from knowledge of the concentrations of the component solutes. Therefore, a moisture sorption isotherm is determined to show the relationship between total moisture content and corresponding A_w over a range of values at a constant temperature (Labuza, 1968).

Figure 2.3 shows typical isotherm for many food powders. Such an isotherm can be divided into several regions depending on the state of the water present. Region A corresponds to the adsorption of a monolayer film of water which ranges up to 5-10% water (Labuza et al., 1970). This region seems to contain the most stable water content for most foods (Salwin, 1959), which is thought to be related to lipid oxidation (Maloney et al., 1966). Region B is related to the adsorption of additional layers of water over this monolayer. Non-enzymatic browning dominates in this region (Labuza, 1968). Region C corresponds to condensation of water in the pores of the material

followed by dissolution of the soluble material present (Labuza, 1968). Although water in this region is mechanically trapped in the system, it is subjected to only weak restrictive forces (Troller and Christain, 1978).

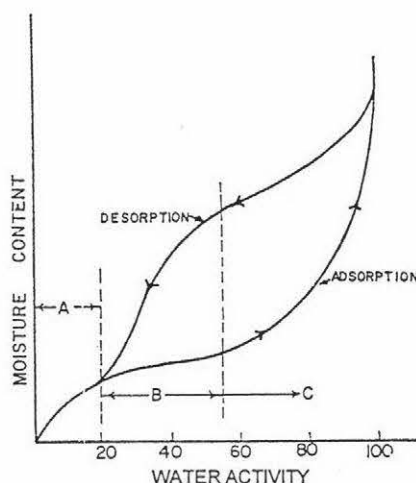


Figure 2.3 General Sorption Isotherm

(Labuza, 1968:264)

Figure 2.3 also shows a hysteresis, the higher water content when the A_w is achieved by desorption of water from a moist material, and a lower water content when the moisture content of a dry food is increased by the adsorption of water. Obviously, moisture sorption hysteresis reveals the irreversibility of the sorption process (Kapsalis, 1987). Several theories proposed by Kapsalis, (1987) to explain hysteresis include:

- Incomplete wetting theory;
- Ink bottle neck theory;
- Open-pore theory;
- The domain theory.

Detailed descriptions can be found in Kapsalis (1987).

Temperature affects the mobility of water molecules and the dynamic equilibrium between the vapour and adsorbed phases (Kapsalis, 1987). Figure 2.4 shows schematically the effects of temperature shifts on both the moisture content and A_w . If

A_w remains constant, then an increase in temperature reduces the amount of adsorbed water. This indicates that the food becomes less hygroscopic (Kapsalis, 1987).

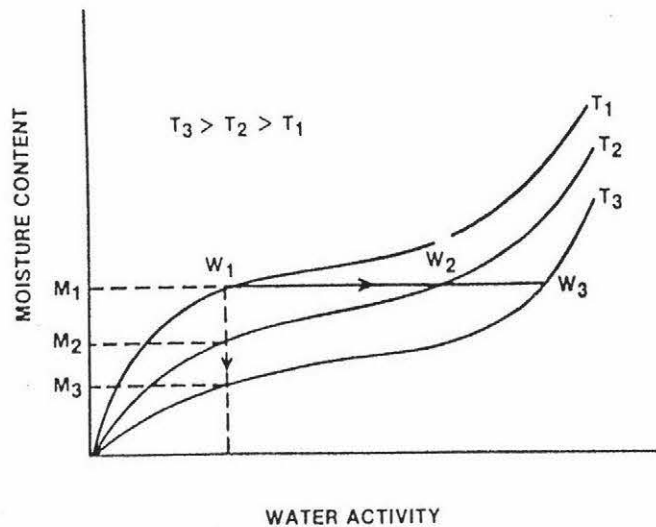


Figure 2.4 Schematic representation of the effect of temperature on A_w and moisture content

(Kapsalis, 1987:192)

Gradients in A_w are common, with surface layers of foods hydrating or dehydrating rapidly in response to changes in the ambient atmosphere, where no immediate changes in A_w occur in the depths of the food. Similarly, a wetter food of higher water content may transfer moisture to a drier food of low water content until the final equilibrium A_w is reached (Salwin and Slawson, 1959).

2.2.5.2 A_w and flowability

Water activity significantly affects the stability of food powders. Moreyra and Peleg, (1981) reviewed the physical aspects of water activity related to agglomeration. Moisture sorption is generally associated with increased cohesiveness due to the formation of interparticle liquid bridges (Peleg, 1983). The presence of liquid bridges in the food powders is sufficient to alter the bulk characteristics of powders such as flowability (Moreyra and Peleg, 1981). Teunou et al. (1999) reported that an increase in A_w decreased the flowability of whey-permeate powder due to an increased cohesion between the powder particles in the presence of water. Buma and Henstra (1971) observed that there were small surface folds and cracks on the surface of some particles by using scanning electron microscopy. This showed that the cohesion of milk powders

can be influenced by water content. Buma (1971c) found that cohesion initially decreased when the moisture content was increased from 2 to 4%, but at higher moisture contents the cohesion increased sharply. He attributed the decrease to the swelling of the casein that causes the disappearance of surface folds or cracks and to a lower 'apparent' cohesion. The latter increase of cohesion was due to increasing stickiness of hydrated lactose glass and subsequent lactose crystallisation (Buma, 1971c).

Moreyra and Peleg (1981) observed that cohesion rapidly increased as the A_w increased for infant formula type food powders. The bulk density was less effected by the water activity level because the structure has reached maximum 'openness' at low moisture contents for such a fine powder system, thereby preventing further reduction of the density (Peleg, 1983). Fowler and Wyatt (1960) showed that an increase in the angle of repose occurred with the increase of moisture content of granular solids. They suggested this variation was due to the surface layer of moisture that surrounded each granular and that surface tension effects became predominant in holding aggregates of granules together.

2.2.5.3 Free fat

The term free fat was defined by Holm et al. (1925), as 'fat not protected by protein film'. Buma (1971b) defined free fat as the fraction of the fat which can be extracted with organic solvents under standardised conditions. King (1965) observed that fat in dried milk can occur either in a finely emulsified state or in a coalesced, de-emulsified state. In the latter case, the membrane around the fat globules has been damaged or entirely removed, allowing the globules to flow together and to form 'pools' of fat. Such fat is extracted with fat solvents and was designated by Holm et al., (1925) as free fat. King (1966) suggested that the unprotected fat permeates the dried milk particles and that a portion of this fat reaches the surface. The surface fat presumably influences the stickiness of milk powders, because stickiness is a specific surface property (Buma, 1971a). Buma (1971b) observed bridges between particles of spray-dried whole milk that are presumably formed by the crystallization of the free fat. McKenna (1997) observed that surface fat appeared to pool at the jointing points of agglomerated powder

particles by using Confocal Laser Scanning Microscopy (CLSM). Peleg (1977) related flow difficulties in fat containing powders to the effect of fat content and environmental temperature. Elevated temperatures during processing or storage may melt a portion of the fat, thereby forming liquid fat bridges between particles. If the temperature subsequently drops, the fat comprising the liquid bridges will crystallize to bind the powder particles together as lumps. However, Peleg (1977) did not identify the source of the fat forming the liquid bridges.

Buma (1968) found that the free fat content significantly decreases in whole milk powder when the moisture content of the powder increased from 2 to 8%. Presumably the pores in the powder would seal as moisture is absorbed, reducing the free fat content. This could explain Buma's observation (Buma, 1971c) why cohesion decreases with the increase of moisture content (Buma, 1968).

2.2.5.4 Protein

Evaluating the aggregation of solid pharmaceutical proteins at elevated temperatures and water activity, Costantino et al. (1994) found that proteins containing disulfide bonds and free thiol residues may aggregate via thiol-disulfide interchange. Proteins containing disulfides but not free thiol residues, may still form intermolecular disulfide bondings via β -elimination of intact disulfide that yield free thiols. The free thiols can catalyse disulfide scrambling. Finally, proteins without cysteine/cystine residues may aggregate by other covalent pathways or by non-covalent routes. Buma (1971c) inferred that an increase in the A_w allows both protein and lactose to become stickier, thereby altering the plasticity of particles and changing the contact area between the particles. The literature currently contains limited information about the ability of proteins to clump in food powders.

2.2.5.5 Miscellaneous factors

Flowability of food powders is a complex phenomenon involving physical properties, components of the powder itself and storage conditions such as the storage temperature, relative humidity or the storage time (Provent et al., 1993). The ability of lactose crystallisation to cause caking in dairy powders depends upon both temperature and length of storage (Slade and Levine, 1991). Changes in the storage temperature may

create free fat which subsequently crystallizes to create clumps in high fat food powders (Peleg, 1977). The stress applied to powders during processing, transporting or storage may deform the particles and thereby increase the number of points of contact between particles. Longer periods of storage promote clumping and flowability problem by all the processes described (Provent, et al., 1993).

2.3 Evaluation of Flowability

2.3.1 Sampling

Unlike fluids, powders are susceptible to change under an applied load and may consolidate with time. Therefore, powdered foods experience attrition and segregation during transfer because the particle size distribution of a powder affects so many properties, and because segregation and stratification by size is common. Representative sampling therefore is critical to ensure the relevance of any subsequent testing (Svarovsky, 1987).

However, sampling methods were not as critical in this study because the samples had been packed into canisters (227g/canister) from a production stream with stable quality. Therefore, any canister of sample from the same specification properly represents the total sample from which it was taken. Furthermore, all samples were exposed to the atmosphere while sampling, which reduces the A_w of the product because the A_w of the product is higher than the atmospherical relative humidity. Because A_w is a very important factor that influences flowability and because even minor changes of A_w will alter the results, it is critical that the A_w must be adjusted under controlled conditions. Therefore, specified sampling methods were not used in this study.

2.3.2 Evaluation of flowability

Results showing the flowability of different materials are difficult to relate because these results depend upon the design and characteristics of the analytical device as well as on the properties of the powder. Savrovsky (1987) defined direct measurements of flowability as the time required for a standard amount of powder pass through a specified funnel or hopper. White et al. (1967) developed a flow meter with a vibrational attachment to measure the flow properties of food powders. Results obtained with this device showed that the mass flow rate is proportional to bulk density, and that

the particle size, particle size distribution, particle shape, relative humidity, moisture content, oil content and temperature influence the mass flow properties (White et al., 1967). Woodhams and Murray (1974) used a rotating drum to assess the flow properties of milk powders. The drum contains two longitudinal slits in the cylindrical wall through which the milk powder is discharged by rotating. This device imitates the flow of powder from the container when used at home by a consumer (Woodhams and Murray, 1974).

2.4 Properties Related to Flowability

2.4.1 Particle size distribution

The properties of a food powder system such as flow properties are highly dependent on its particle size distribution (Peleg, 1977). An accurate description of this distribution is essential to the analysis of the handling, processing and prediction of the functionality of the food powder (Yan and Barbosa-Canovas, 1997). Sieving a sample of the powder through a stack of standardized sieves and recording the mass of powder retained on each screen is a common method of measuring the particle size distribution (Woodhams and Murray, 1974). This procedure is restricted to powders that are mostly larger than 75 μm . Finer powders generally are not analyzed by this method because of the great expense of producing sieves with uniform small apertures (Allen, 1974). The difficulties of sieving dry milk powder are described by Buma (1971d), and Lammers et al. (1987). The tendency of small dry milk particles to aggregate as much larger lumps disrupts the ability of sieving to accurately show the particle size distribution (Buma, 1971d). The use of flow agents prevents clumping during sieving (Niro Atomiser, 1978).

Microscopic analysis is the most widely used method in dairy industry (Retnam et al., 1989). Microscopic analysis is primarily used to measure small particles within the range of 2 to 100 μm . If the distribution of particle sizes is wide, then this procedure can be highly inaccurate (Hayashi et al., 1969). For example, if the microscope is focused on the smaller particles, larger particles will not be visible in the field. If the microscope is focused on the large particles, resolution of small particles is lost (Hayashi et al., 1969). Furthermore, this procedure is tedious as minimum of 500 particles must be scanned to obtain reliable results (Retnam et al., 1989). Janzen et al (1953) concluded

that counting the size of all particles in ten fields of view produces results which are representative for a powder sample. Despite these disadvantages, the microscopic method provides a direct observation of particle size and shapes (Hayashi et al., 1969). Other available methods include the photosedimentation method, light diffraction methods, sedimentation balance method and centrifugal method. These methods are rarely used due to the high cost and relative complexity (Retnam et al., 1989).

Sieving was chosen as the primary method for defining the particle size distribution for this study because the particles of Grated and Dried Parmesan are much larger than the particles in most milk powders. Previous work (Elston, private communication) shows the particle size distribution of hard and grated Parmesan ranges from $>1700\text{ }\mu\text{m}$ to $<355\text{ }\mu\text{m}$. The method generated timely results for the size distribution for grated Parmesan when compared to the other methods.

Few authors describe methods of mathematical analysis for determining the size distribution of food powders (Buma, 1971d). Hayashi et al (1969) mentioned a log-normal distribution. Yan and Barbosa-Canovas (1997) identified the availability of five particle size functions for characterising the particle size distribution for different types of food powders.

2.4.2 Angle of repose

The methods for determining the angle of repose are fully described by Savrovsky (1987) and Teunou et. al.(1995). The angle of repose is widely quoted as an index of the flowability of a powder (Tuohy, 1989). However, Jenike (1967) argued that the angle of repose only is useful in the determination of the contour of a pile and is not a measure of the flowability of solids. According to Peleg (1977), powders with angle of repose of less than about 40° are free flowing. Powders exhibiting an angle of repose of $>50^\circ$ are likely to block flowability. A decrease in the angle of repose proportionally increases the flowability of the powder (Peleg, 1977). Because the forces that form a pile of powder include both frictional forces and interparticle cohesive forces, the actual measurements depend on the experimental method and procedure (Peleg, 1977). Results showing the angle of repose determined by different methods and procedures cannot be compared.

For example, the angles of repose reported by Tuohy (1989) for whole milk powder (56°) are within the range for whole milk powder ($53\text{--}65^\circ$) reported by Sjollema (1963) while those for skim milk powder ($33\text{--}38^\circ$) are lower than the 45° reported by Sjollema (1963).

The estimations of flowability indicated by the angle of repose frequently are not accepted as valid (Jenike, 1967) because the different methods produce different results. Therefore, measurements of the angle of repose were not used in this study.

2.4.3 Evaluation related to failure properties

The methods used for evaluating the flowability of powders generally detect failure properties, producing data which can be used for quantitative designs and qualitative comparisons (Savrovsky, 1987). These methods typically use shear techniques and are based upon theories developed by Jenike (1967) for determining yield loci by use of Jenike shear cell apparatus as applied to food powders (Peleg, 1977; 1983). Despite recent improvements, such as the annular shear cell (Walker, 1967), rotational cell (Peschl, 1989) and the Johanson indicizer (Johanson, 1992), this method is laborious and time consuming, requires specialized equipment and skilled technologists (Teunou et al., 1999). The procedure shows the response of the material to a stress that is equivalent to the stress encountered during storage, and therefore is not suitable when samples are packed in a canister containing 227g of product.

2.5 Conclusions

Flowability of food powders is a complex phenomenon of simultaneous and complementary interactions that involves internal and external factors. Flowability is influenced by the physical properties of a powder such as particle shape, particle size and size distribution, and A_w and by elements of powder composition such as the free fat content. Flowability is also affected by environmental conditions such as storage temperature, relative humidity, storage time and the stress applied to powders.

Flowability is measured as the rate of the mass flow by using funnel or rotating drum or is evaluated by defining failure properties. The former method qualitatively compares

food behavior for quantitative and design purposes. The measurement of flowability by using a rotating drum is preferred to the other methods in this study, being the most accurate approximation of the shaking of product from canisters by consumers.

CHAPTER 3

MATERIALS AND METHODS

3.1 Materials

3.1.1 Samples used to examine the suitability of the method to measure flowability and particle size distribution

3.1.1.1 Samples used to examine the suitability of the method to measure flowability

Six samples of Grated and Dried Parmesan cheese were used to investigate the flowability test method. Five of them had been made at the Greenwood Valley Cheese Company (GVC), Wisconsin (WI), USA. The remaining sample had been made by Kraft, Pennsylvania (PA), USA. The five GVC Grated and Dried Parmesan products were labelled as GVC-1, GVC-2, GVC-3, GVC-4 and GVC-5. The Kraft sample was labelled as Kraft-1.

3.1.1.2 Samples used to examine the suitability of the method to measure particle size distribution

One Kraft's and one GVC's Grated and Dried Parmesan cheese sample were used to examine the method of determining the particle size distribution. The Kraft sample was labelled as Kraft-2 and the GVC sample was labelled as GVC-6.

3.1.2 Preliminary study on the effect of moisture content on flowability

Nine Grated and Dried Parmesan cheese samples were obtained from the GVC. These samples had been made for a storage trial to be conducted at the GVC. The moisture contents of these samples were 18, 21 and 22% at the time of manufacture. In addition to the three different moisture levels, the samples at each moisture content had been made with or without additives. The Grated and Dried Parmesan cheese made by Kraft was purchased from supermarkets in Harrisburg and Reading, PA, USA. All the

samples had been packed in ½ lb canisters. A key to identify these samples is given in Table 3.1.

Table 3.1 A key to identify the samples used in the preliminary study

Moisture content (%)	Additive		
	No additive	Potassium sorbate	Sodium benzoate
18	18 NA	18 Sorb	18 Benz
21	21 NA	21 Sorb	21 Benz
22	22 NA	22 Sorb	22 Benz

3.1.3 Samples used for confirming the effects of A_w , particle size distribution and free fat on flowability

The Grated and Dried Parmesan cheese samples used at this stage include three GVC samples and one Kraft sample. The three GVC products were:

- Specification 50017HM (high moisture)
- Specification 50017
- Specification 69430

Kraft Grated and Dried Parmesan cheese purchased from supermarkets in USA was used at this stage as well. All the samples were in ½ lb canisters when they arrived at the NZDRI.

These samples were used for adjusting or manipulating the A_w and particle size distribution to evaluate their effects on flowability. Specification 50017HM was used to evaluate the effect of free fat on flowability.

3.1.4 Commercial samples with different A_w s that were used for further confirmation of the effect of A_w on flowability

Grated and Dried Parmesan cheese made on the 10th January 2000 under different GVC specifications (specification 50017 and 50064) were used in this study. Several samples from each commercial manufacturing run with different A_w s were chosen to repeat the analysis of the effect of A_w on flowability.

The variation in moisture content that naturally occurs in most drying operations contributed to the variation in water activity of the product. The variation in moisture content was due to the small changes in various processing parameters such as inlet and outlet airflow rates, temperature, the humidity in the drying air and flow rate of the product through the fluid bed. Therefore, the samples with different water activities that were belonging to the same production specification and that had been made on the same day using the same raw materials are ideally suited to study the effect of A_w on flowability.

3.2 Methods

Appropriate methods were selected for characterising the physical properties and for modifying the physical properties of the Grated and Dried Parmesan cheese samples.

3.2.1 Characterisation of selected physical properties

3.2.1.1 Examining the suitability of the method to measure flowability by a rotating drum

The NZDRI method (MP – MT – FL01) of measuring flowability involves a motorised rotating drum as shown in Figure 3.1.

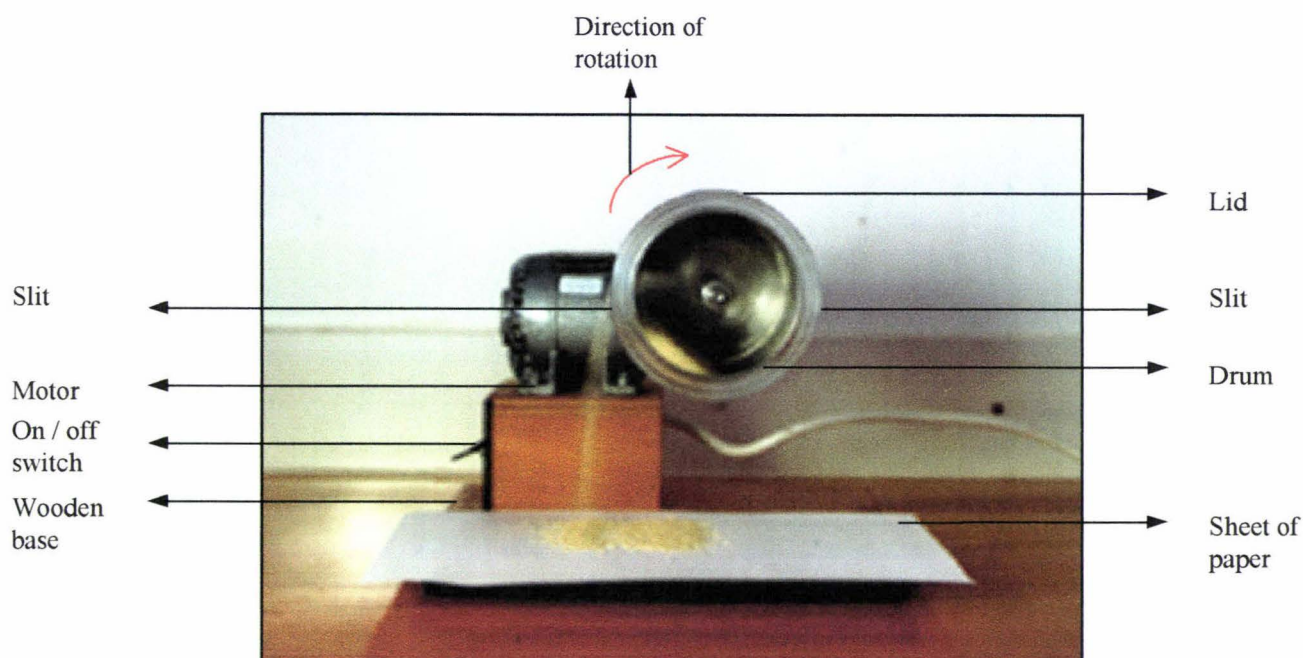


Figure 3.1 A photo of the motorised rotating drum used for measuring flowability

This instrument is designed to measure the flowability of a powder. It mainly includes three parts: a rotating drum ($\varnothing$ 110 mm) with two slits (diagonally opposing), a motor to rotate the drum at a constant speed and a wooden base to support the motor. When the motor is switched on, the drum rotates at a preset speed of 30 rpm and the powder flows out of the drum through the two longitudinal slits in the cylindrical wall of the drum.

The test procedure is described as below:

- Weigh 30.00g powder
- Adjust the drum so that the slits are aligned horizontally
- Transfer the powder into the drum and put on the lid. Screw on the nut to tighten the lid
- Place a sheet of paper (A4 size) under the drum to collect the powder that comes out

through the slits

- Start the drum (motor preset at 30 rpm) and switch a stopwatch on simultaneously
- Collect the powder while the drum is rotating
- After 10.00 ± 1.00 seconds, insert a second sheet of paper between the drum and the first A4 paper collecting powder. Switch off the power at the same time
- Weigh the powder collected on the first sheet of paper during the 10.00 ± 1.00 second period of drum rotation
- Record the weight of the powder flowing out from the drum within the 10.00 ± 1.00 seconds
- Calculate the flowability in g / sec

As outlined in section 3.1.1, one Kraft and five GVC Grated and Dried Parmesan cheese samples were used to test the suitability and especially, the repeatability of the flowability test method. Three sub-samples taken from each of the above samples were used to test for flowability. Each of these sub-samples was tested three times repeatedly. The mean flowability was obtained by averaging nine readings (three sub-samples $\times$ three measurements on each sub-sample). These flowability data are given in Table 3.2.

Table 3.2 Investigating the repeatability of the flowability test method for five GVC and one Kraft Grated and Dried Parmesan cheese samples

GVC-1

Flowability (g/s)			
Sub-sample	1#	2#	3#
Measurement 1	0.97	0.94	0.93
Measurement 2	1.01	1.02	1.05
Measurement 3	1.06	1.03	1.12
Range	0.93 – 1.12		
Mean flowability	1.01		
Standard deviation	0.06		

GVC-2

Flowability (g/s)			
Sub-sample	1#	2#	3#
Measurement 1	0.98	1.27	1.29
Measurement 2	1.28	1.24	1.24
Measurement 3	1.19	1.19	1.29
Range	0.98 – 1.29		
Mean flowability	1.22		
Standard deviation	0.10		
(to be continued)			

GVC-3

Flowability (g/s)			
Sub-sample	1#	2#	3#
Measurement 1	0.93	0.95	0.95
Measurement 2	1.10	1.09	1.14
Measurement 3	1.19	1.13	1.21
Range	0.93 – 1.21		
Mean flowability	1.08		
Standard deviation	0.11		

GVC-4

Flowability (g/s)			
Sub-sample	1#	2#	3#
Measurement 1	0.85	0.88	0.88
Measurement 2	1.02	1.02	0.93
Measurement 3	1.01	1.01	1.21
Range	0.85 – 1.02		
Mean flowability	0.93		
Standard deviation	0.06		

(to be continued)

GVC-5

Flowability (g/s)			
Sub-sample	1#	2#	3#
Measurement 1	0.97	0.94	0.93
Measurement 2	1.01	1.02	1.05
Measurement 3	1.06	1.03	1.12
Range	0.93 – 1.12		
Mean flowability	1.01		
Standard deviation	0.08		

Kraft

Flowability (g/s)			
Sub-sample	1#	2#	3#
Measurement 1	1.29	1.43	1.26
Measurement 2	1.40	1.32	1.24
Measurement 3	1.39	1.26	1.21
Range	1.21 – 1.40		
Mean flowability	1.31		
Standard deviation	0.08		

The data given in Table 3.2 show that the flowability values were distributed within a wide range for each of the above samples. To minimise the errors caused by the variability, it was decided to make 12 repeated flowability measurements on each of the sub-samples.

3.2.1.2 Examining the suitability of the test method to measure particle size distribution

The method used to determine the particle size distribution has originated from the Niro method No. A 8 a (Niro Atomiser A/S, 1978). By sieving a powder sample through a number of sieves with different mesh sizes, the sample was divided into fractions with different particle size. A shaker (Model No G-25, Pascall Engineering Co. Ltd, England) was used to test the particle size distribution. A stack of 5 sieves with apertures of 1700 μm , 1000 μm , 850 μm , 600 μm , to 355 μm were used in this study. The mesh sizes selection was based on the previous work undertaken at the NZDRI (P. Elston, personal communication).

Before sieving, the tare weights of each sieve and the pan were recorded; after sieving all sieves were weighed again to measure the material retained on each one. The precision in reading the weight fell within $\pm 0.01\text{g}$. The results were obtained as percent cumulative undersize.

The method used at the NZDRI (P. Elston, personal communication) to measure the particle size distribution of Grated and Dried Parmesan cheese is briefly described below: 200g sample was loaded on the top sieve and then shaken for 25 minutes. Serious blockages on each sieve, especially on the sieve with smaller particle size, were observed.

Afterwards, the sample size was reduced from 200g to 100g. Unfortunately, blockages were still observed on the surface of those sieves. Then, the sample size was further reduced from 100g to 25g. Syloid, which is a free-flowing agent, was also used to prevent the particles from lumping. One percent of syloid (0.25g) was added into the

sample before sieving. The function of syloid is presumably to interrupt the liquid bridges (Peleg and Hollenbach, 1984) and then enhance the particles passing through the sieves.

Usually, it is the smallest particles that block the sieve pore. The sieve with smaller aperture is more likely to be blocked. Therefore, a 5-minute pre-separation by using the smallest sieve (355 μm) was adopted (see Figure 3.2) in order to reduce the blockage. After the pre-separation, the remaining sample on the 355 μm sieve was transferred to the top sieve (1700 μm). The lid was put on and the sample was sieved again for another 25 minutes. A flow chart of the procedure is given in Figure 3.2.

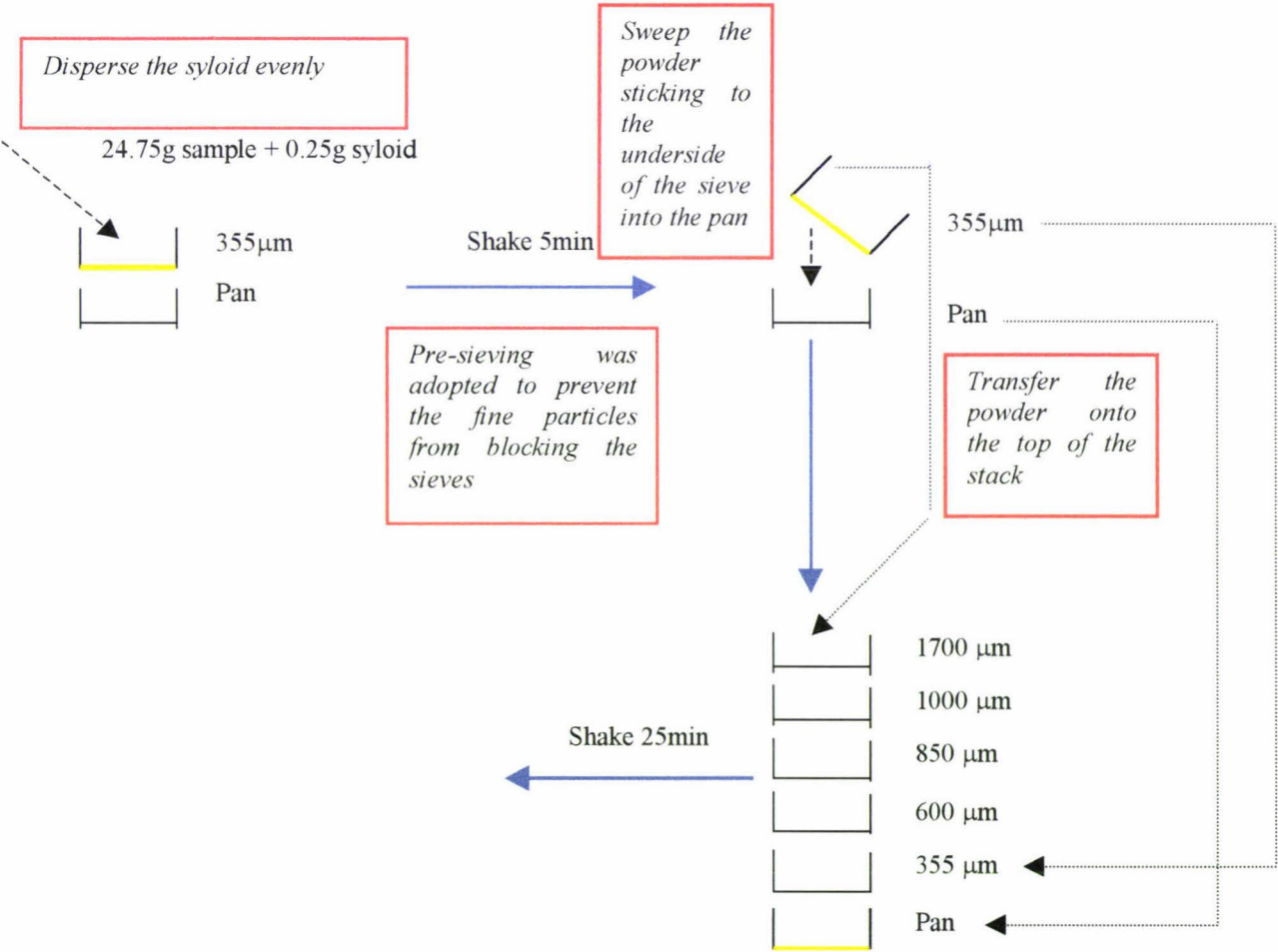


Figure 3.2 Flow chart of the procedure of determining particle size distribution by sieving

The method described above was used on the samples in section 3.1.1.2 to examine the suitability, especially the repeatability, of the test method. Three sub-samples were taken from each of the samples and the measurements of particle size distribution were repeated twice for each sub-sample. Table 3.3 shows the particle size distribution data for Kraft-2 and GVC-6.

Table 3.3 Investigating the repeatability of the test method to measure the particle size distribution of Kraft and GVC Grated and Dried Parmesan cheese samples

Kraft-2				
	Cumulative undersize (%)	Measurement 1	Measurement 2	Measurement 3
Sub-sample 1	< 1700 μm	98.01	97.76	97.78
	< 1000 μm	71.00	68.56	66.74
	< 850 μm	58.00	53.88	49.38
	< 600 μm	45.36	40.08	32.99
	< 355 μm	29.24	18.32	4.17
Sub-sample 2	< 1700 μm	98.58	97.86	97.90
	< 1000 μm	71.17	68.27	66.33
	< 850 μm	59.10	54.01	49.12
	< 600 μm	44.57	37.81	29.97
	< 355 μm	31.33	18.80	6.34
Sub-sample 3	< 1700 μm	98.72	98.32	98.21
	< 1000 μm	73.01	72.05	70.90
	< 850 μm	60.86	59.90	57.75
	< 600 μm	48.10	46.06	42.84
	< 355 μm	34.18	31.08	26.85
(to be continued)				

GVC-6

	Cumulative undersize (%)	Measurement 1	Measurement 2	Measurement 3
Sub-sample 1	< 1700 μm	99.19	99.10	97.78
	< 1000 μm	80.29	70.35	66.74
	< 850 μm	65.90	47.78	49.38
	< 600 μm	53.16	38.37	32.99
	< 355 μm	29.54	23.60	4.17
Sub-sample 2	< 1700 μm	98.45	98.24	98.17
	< 1000 μm	75.28	56.88	48.69
	< 850 μm	50.96	24.44	16.34
	< 600 μm	39.29	11.30	4.72
	< 355 μm	26.40	3.93	1.21
Sub-sample 3	< 1700 μm	99.31	99.04	98.28
	< 1000 μm	70.04	56.76	44.64
	< 850 μm	45.95	22.34	15.46
	< 600 μm	32.21	7.52	3.90
	< 355 μm	27.07	3.95	1.95

The results for sub-sample 1 of Kraft-2 show that the cumulative undersize (%) of each fraction < 1000 μm decreased when the test was repeated using the same sample. This might have been due to the loss of smaller particles. This was unavoidable even under very careful removal of particles. The other possible reason could be the stickiness of free fat on the sieves especially the sieves with smaller apertures (e.g. 355 μm). This blocked the particles passing through the sieves (Niro Atomiser A/S, 1978). If repeated using the sieves without the proper cleaning of free fat sticking on the sieves, the efficiency to separate the particles decreases. This reduction was shown for each of the sub-samples in Table 3.3.

Therefore, it was more meaningful to measure the particle size distribution on different sub-samples rather than to repeat the tests several times on each of the sub-samples. For this reason, it was decided to take two sub-samples from each of the samples to determine the particle size distribution. Only one measurement was taken on each of the sub-samples. Meanwhile, the sieves were cleaned after each measurement. The cleaning procedure is described below:

- Soak the sieves in 2-4 g/l hot detergent (Cleanaid, Intermed Scientific Ltd, Auckland, New Zealand) for 5 Min
- Wash the sieves using the same detergent after soaking
- Rinse the sieves with water
- Dry the sieves in an oven at 100°C
- Cool the sieves at ambient temperature

3.2.1.3 Measurement of free fat content

Free fat content was determined by evaluating the fat using petroleum ether (40 - 60°C, AnalaR, BDH Laboratory Supplies, England) as specified in the NZDRI method of determining the free fat content in milk powder (MP-MT-FF01, NZDRI). All the free fat tests were performed in duplicate. The free fat content was recorded as the average value of two determinations. The detailed procedure is given in Appendix A.

3.2.1.4 Measurement of A_w

An Aqua Lab model CX3 water activity meter (Decagon Devices, Inc, Washington, USA) was used to measure A_w . The detailed procedure is given below:

- Turn on the instrument and let it warm up for 15 Min to an hour
 - Calibrate the instrument by using water activity verification standards: 6M Sodium Chloride and distilled water with A_w s of 0.760 and 1.000 respectively
 - Fill the sample cup no more than half full without contaminating the rim and outside of the sample cup
 - Place the sample cup in sample drawer, close drawer carefully to avoid spillage
-

- Turn the drawer knob from OPEN/LOAD to READ to start measurement
- Take the reading when the measurement is complete

Samples were measured at ambient temperature. All the water activity tests were performed in triplicate and A_w was recorded as the average value of the three measurements. The precision of A_w measurement was ± 0.005 .

There were other standard solutions available. The reason for choosing 6M Sodium Chloride with A_w of 0.760 was because its value is closest to the A_w of Grated and Dried Parmesan cheese. The reason for using distilled water to calibrate was to make sure that the offset had not occurred so the instrument was still in good performance.

3.2.2 Manipulation of physical and compositional parameters

The purpose of this study is to investigate how the flowability changes with varying the parameters such as water activity, particle size distribution and free fat separately. Therefore, each parameter was adjusted to span a wide range around the original level.

3.2.2.1 Water activity manipulation

First, A_w of each of the original samples was measured. The A_w of each sample was increased or decreased through moisture adsorption or moisture desorption to obtain a range of water activities.

A simple device was used to manipulate the water activity of the product. It consisted of a plastic box of approximate dimensions of 36 cm $\times$ 24 cm $\times$ 12.5 cm (purchased from Payless Plastic Ltd, New Zealand), some petri dishes ($\varnothing$ 8.5cm) for containing the samples, a plastic platform to support these petri dishes and some plastic containers to contain either distilled water or silical gel. The details of the apparatus are shown in Figure 3.3.

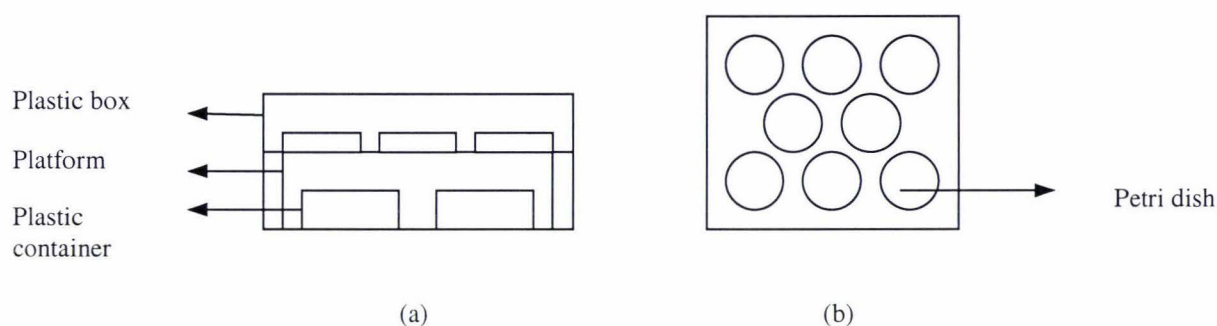


Figure 3.3 Schematic diagram of the apparatus used to manipulate A_w

The reasons for using the plastic boxes rather than desiccators were because of their cheaper price, the larger size to modify a large amount of samples (e.g. 100g) at one time and the same performance as a desiccator. The advantage of using either distilled water or silica gel was that the adjustment of A_w either by distilled water or silica gel was faster than by using saturated salt solutions. However, the disadvantage was that the A_w was not equilibrated evenly from part to part, from particle to particle within one sample instantly. Therefore, the samples were heat-sealed in aluminium foil sachets and then were allowed to equilibrate for seven days before the measurements were taken.

3.2.2.1.1 Increasing the water activity through moisture sorption

Moisture adsorption by Grated and Dried Parmesan cheese due to the exposure to distilled water was the basis used to increase the A_w . The detailed procedure is described below:

- Disperse approximately 100g of sample evenly onto 8 petri dishes. Spread the sample to form a thin and loose layer to facilitate the moisture sorption
- Put the plastic containers filled with distilled water into a plastic box
- Place the platform into the plastic box
- Seal the box tightly and let the sample absorb moisture
- Check A_w from time to time until the sample is adjusted in the required range

- Remove the sample into an aluminium foil sachet, heat-seal the sachet and allow time for moisture equilibration
- If the sample is in the required range then it is ready for measuring flowability according to the method in section 3.2.1.1; if is not in the required range, put it back to the box and remodify the A_w until it is in the required range

3.2.2.1.2 Decreasing the water activity through moisture desorption

Water activities were decreased through moisture desorption by exposing the sample to silica gel (BDH Laboratory Supplies, England). The detailed procedure was the same as above.

3.2.2.2 Particle size distribution manipulation

The particle size distribution of each original sample was measured prior to the manipulation. Each of the original samples was sieved by using the 600 μm sieve individually to obtain two fractions: fraction A and fraction B. Fraction A was composed of large particles ($> 600 \mu\text{m}$) and some fine particles ($< 600 \mu\text{m}$). Fraction B contained only fine particles.

Fractions A and B were then mixed together in different proportions to either increase the amount of fine particles in the original sample to three higher levels or to decrease the amount of fine particles to three lower levels. During the preparation of each mixture, a reduction in the A_w of the sample was possibly due to the evaporation of moisture from the sample into the atmosphere. The A_w of the final samples was checked to make sure that the variance of the A_w among the mixtures in a sample was within ± 0.005 and adjustment was made if necessary. The flowability was measured as described in section 3.2.1.1 and the particle size distribution was measured as described in section 3.2.1.2 for every mixture of each of the samples.

3.2.2.3 Free fat manipulation

The original free fat content of the sample 50017HM was determined prior to the manipulation. Free fat content was increased to several higher levels by spraying different amounts of anhydrous milk fat (AMF) produced by Anchor Products Ltd., Edgecumbe, New Zealand.

3.2.2.3.1 Obtaining the higher free fat levels

The detailed procedure is shown as below:

- Load several hundred grams of frozen fat cut from a 25 kg FFMR block into a sprayer and melt in a water bath
- Weigh a certain amount* of 50017HM and place them in a plastic bag
- Spray the required amount* of melted free fat onto the sample
- Seal the sample tightly and put it into an oven at 60°C for 15 Min
- Remove the sample from the oven and shake the plastic bag manually for 5 Min in order to evenly disperse the free fat throughout the sample
- Place the sample bag back in the oven at 60°C for another 15 Min
- Shake the bag for another 5 Min
- Pack the sample in aluminium foil sachets and heat-seal the sachets

3.2.2.3.2 Obtaining the lower free fat levels

- Extract the free fat in the sample as specified in section 3.2.1.3 by using petroleum ether (AnalaR, BDH Laboratory Supplies, England)
 - Increase the A_w of the sample to the original value. This is necessary to compensate for the moisture loss during the extraction of fat
 - Follow the method given in section 3.2.2.3.1 to modify the free fat to the required level
 - Pack the sample in aluminium foil sachets and heat-seal the sachets
-

* Either before increasing or decreasing the free fat level, the required amount of free fat sprayed into sample was calculated. For example, suppose the free fat in the original sample was 20%, one greater level was at 22% and 160g of the sample with 22% free fat was required, so X g free fat was sprayed into the sample to increase the free fat level from 20 to 22%. According to this, the formulas were set up as below:

$$(20\% \times Y + X) / (Y + X) = 22 / 100 \quad (1)$$

$$X + Y = 160 \quad (2)$$

Where X : the amount of free fat sprayed into the sample

Y: the amount of the original sample needed.

Therefore, X and Y were obtained by combining (1) and (2). However, the free fat level in this case (e.g. 22%) may not be accurate enough, so the exact free fat content was determined by using the method in section 3.2.1.3.

For the samples with different free fat contents, the A_w was measured to make sure the variance was within ± 0.005 , flowability and particle size distribution were measured.

3.3 Data Analysis

Initially One-way analysis of variance (ANOVA) was used to test the statistical significance of the effect of A_w on flowability. Sigmaplot linear regression was used to obtain median particle size and also used to extrapolate flowability versus A_w , median particle size and free fat.

CHAPTER 4

THE EFFECT OF A_w ON FLOWABILITY

——— RESULTS AND DISCUSSION

This chapter describes the effect of moisture content / A_w on flowability. The other factors such as particle size distribution and free fat were kept constant in this study. The effects of particle size distribution and free fat are discussed in the next two chapters.

4.1 Preliminary Study on the Relationship between Flowability and Moisture Content

Nine GVC Grated and Dried Parmesan cheese samples (in section 3.1.2) were used in the preliminary study. The samples used in the preliminary study had moisture levels of 18%, 21%, and 22% at the time of manufacture. At each moisture level, samples containing no additive (NA) and samples incorporated with 0.25% potassium sorbate (Sorb) and 0.25% sodium benzoate (Benz) were included in this study.

From each of these samples, three sub-samples were drawn to test for flowability. Each of these sub-samples was repeatedly tested for flowability 12 times (see Appendix B-1). The flowability data are presented in Figure 4.1(a) to (c). Each of the flowability data points is represented by a circle. These graphs were obtained by using Microsoft Minitab 12 Dotplot.

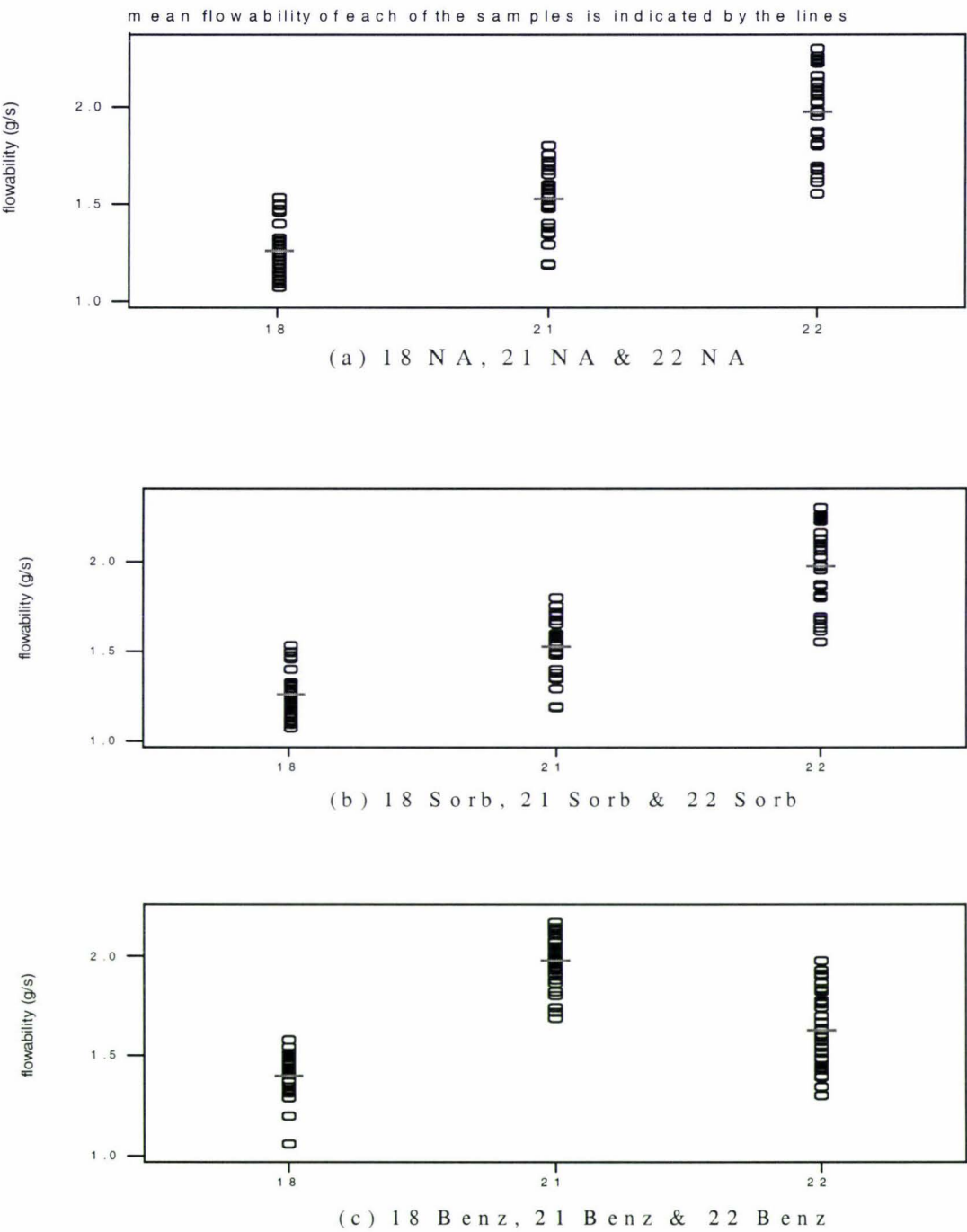


Figure 4.1 (a) to (c) Flowability of nine GVC Grated and Dried Parmesan samples that were used in the preliminary study on the effect of moisture content on flowability

Figure 4.1 shows that flowability of each of these samples spanned a wide range. For example, flowability for 18 NA ranged from 1.1 to 1.6 g/s, where for 22, NA, flowability ranged from 1.6 to 2.3 g/s. This implied the variability of flowability within each of the samples. This was the reason why 12 repeated measurements were made. Despite the variability, differences in flowability between the samples with different moisture contents were observed. Figures 4.1(a) and 4.1(b) show that the samples at higher moisture contents had greater flowability than that of the samples with lower moisture contents.

In order to measure flowability accurately, the average flowability value of 36 measurements (three sub-sample $\times$ 12 repeat measurements) was used. Table 4.1 lists the flowability for each of these samples.

Table 4.1 Flowability of nine GVC Grated and Dried Parmesan samples that were used in the preliminary study on the effect of moisture content on flowability

NA		Sorb		Benz	
Moisture content (%)	Flowability (g/s)	Moisture content (%)	Flowability (g/s)	Moisture content (%)	Flowability (g/s)
18	1.18	18	1.25	18	1.40
21	1.63	21	1.53	21	1.98
22	2.34	22	1.98	22	1.62

Table 4.1 shows that flowability increased with increasing moisture content for the two lots of samples containing no additive and 0.25% added potassium sorbate. The lot containing 0.25% sodium benzoate did not show the trend as flowability increased from 1.40 g/s to 1.98 g/s when the moisture content was up from 18 to 21%. Then flowability decreased down to 1.63 g/s as the moisture content increased from 21 to 22%.

The reason for adding either potassium sorbate or sodium benzoate was to prevent mould growth in the final product. Therefore, these two additives are antimicrobial agents rather than free-flowing agents. The amount of the two additives added was at a minor concentration of 0.25%. It was not feasible to obtain more samples to use as replicates. Therefore, based on the assumption that these two antimicrobial agents would not influence flowability, the samples made with and without additives were treated as replicates.

The flowability data of each of the samples (with or without additives) were subjected to one-way analysis of variance (Microsoft Minitab 12) by testing them as replicates (Appendix C-1). The results show that the moisture content had a significant effect ($p < 0.05$) on flowability. The average flowability for all the samples containing 18% moisture was 1.28 g/s; at 21% moisture was 1.71 g/s and at 22% moisture was 1.98 g/s. Flowability increased with increasing moisture content and this increase in flowability was statistically significant.

4.2 Hypothesis Development of the Effect of A_w on Flowability

4.2.1 Use of A_w instead of moisture content

As pointed out early in the literature review, the term “moisture content” implies the total amount of water present in the product. There are limitations of using the moisture content to show the intensity with which water associates and interacts with other food components. On the other hand, A_w is a measure of the degree of mobility of water, which affects the physical characteristics of a product and the microbial growth and chemical reactions occurring in a product. As A_w is the driving force rather than moisture content responsible for physical characteristics, chemical reactions and microbial growth, the study would be focused on the effect of A_w on flowability.

4.2.2 The relationship between A_w and moisture content

The relationship between A_w and moisture content of a product is represented by its moisture isotherm (Troller and Christian, 1978) and water activity of a product increases as moisture content increases (Labuza, 1968).

4.2.3 Hypothesis development of the effect of A_w on flowability

Preliminary study given in section 4.1 shows that flowability increases with increasing moisture content. The relationship between A_w and moisture content reveals that as the driving force, water activity increases with increasing moisture content. Based on these observations, a new hypothesis was developed.

HYPOTHESIS: Flowability of Grated and Dried Parmesan increases with increasing A_w

The following experiments were carried out to test the new hypothesis

4.3 Testing the Hypothesis of Increasing Flowability by Increasing A_w

Three GVC Grated and Dried Parmesan cheese samples: 18 NA; 21 NA and 22 NA plus Kraft’s were used to investigate the effect of A_w on flowability. Manipulation of A_w (as described in section 3.2.2.1) through adsorption or desorption of water was used to obtain a series of water activity levels in each of these samples. For every sample at each of the A_w s, the flowability was measured 12 times (see Appendix B-2). The average flowabilities calculated from 12 measurements are given in Table 4.

Table 4.2 The relationship between A_w and flowability of 22, 21, 18 NA & Kraft at different A_w s

22 NA		21 NA		18 NA		Kraft	
A_w	Flowability (g/s)	A_w	Flowability (g/s)	A_w	Flowability (g/s)	A_w	Flowability (g/s)
0.66	1.12	0.67	1.11	0.67	0.93	0.68	0.80
0.68	1.30	0.69	1.12	0.69	0.93	0.69	0.82
0.71	1.78	0.72	1.32	0.71*	1.13	0.72	0.79
0.74	1.68	0.74*	1.34	0.74	1.32	0.74	0.78
0.77*	2.11	0.76	1.53	0.77	1.41	0.79*	0.82
0.80	2.20	0.79	1.81	0.81	1.22	0.81	0.90

*: The original A_w of each sample

To illustrate the relationship between A_w and flowability, the flowability data were plotted versus A_w for each of the samples (in Figure 4.2) by using Sigmaplot 4.01.

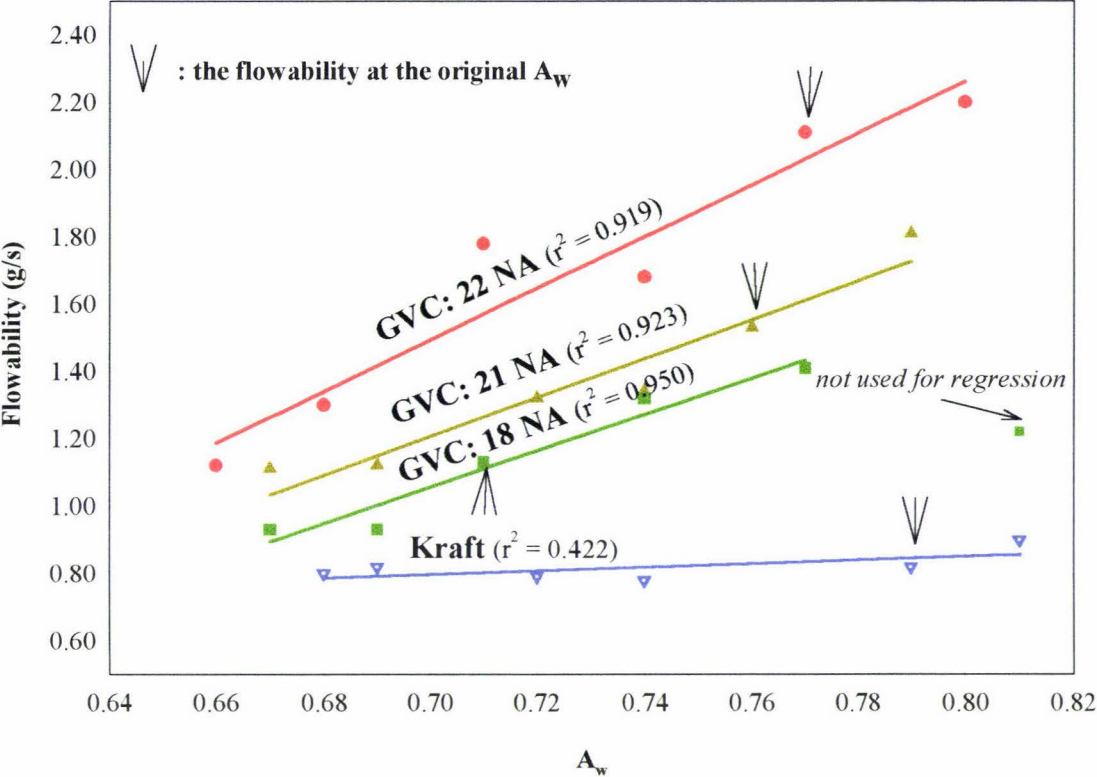


Figure 4.2 The relationship between A_w and flowability for GVC: 22 NA, 21 NA, 18, NA & Kraft samples

Figure 4.2 shows that flowability increased with increasing A_w for each of the GVC samples and the rate of increase in flowability with increasing A_w . Among the three GVC samples, 22 NA had the greatest rate of about 0.08 g/s per 0.01 A_w , followed by 21 NA and 18 NA. The rates of these two samples were approximately 0.06 g/s per 0.01 A_w .

The plots for GVC products show that the relationship between flowability and A_w fitted the first-order mathematical model. The correlation coefficients (r^2) for 22 NA, 21 NA and 18 NA were 0.919, 0.923 and 0.950 respectively.

However, Kraft's product did not show the increasing trend as much as the GVC samples and the rate for Kraft was less than 0.01 g/s per 0.01 A_w . The r^2 for Kraft was 0.422. The reason for not observing a marked improvement in flowability in Kraft was not investigated.

It is worth noting that for the GVC sample 18 NA, flowability increased from 0.93 g/s to 1.41 g/s with increasing A_w from 0.67 to 0.77, but at A_w of 0.81, flowability dropped down to 1.22 g/s. Although flowability between 0.77 to 0.81 A_w had not been obtained, the results gave an indication that there could be a critical A_w at which the product had the highest flowability. If it is the case, this value should be between 0.77 to 0.81. During the next trial, the A_w values between 0.77 and 0.81 should be chosen to investigate how flowability changes with increasing A_w within this range.

The black arrows in Figure 4.2 show flowability of each of the samples at its original A_w value. It is shown that the sample with 22% moisture content ($A_w = 0.77$) has the highest flowability of 2.11 g/s, followed by the sample with 21% moisture level ($A_w = 0.74$) of 1.53 g/s and 18% moisture level ($A_w = 0.71$), 1.13 g/s. This was in agreement with the observation that flowability increased with increasing A_w .

4.4 Confirmation of the Critical A_w

In this trial three Grated and Dried Parmesan samples under the GVC specification (specification 50017 HM, 50017 and 69430) were used to test the validity of the hypothesis. One Kraft Grated and Dried Parmesan cheese purchased from a supermarket in the USA was used also.

In order to find the critical A_w , the A_w of each sample was manipulated to get a series of A_w levels between 0.65 to 0.83, in which the possible critical value was included. Manipulation of A_w was carried out as described in section 3.2.2.1 for each of the samples. For every sample, A_w was measured as given in section 3.2.1.4 and A_w was obtained by averaging the three readings. Flowability was measured for each of the samples according to the method in section 3.2.1.1 (see Appendix B-3). The flowability value for each of the sample was obtained by averaging 36 measurements (three sub-samples $\times$ 12 repeated measurements). Table 4.3 shows the flowability data against A_w for each of the samples.

Table 4.3 The flowability data against A_w of 50017HM, 50017, 69430 and Kraft

50017HM		50017		69430		Kraft	
A_w	Flowability (g/s)	A_w	Flowability (g/s)	A_w	Flowability (g/s)	A_w	Flowability (g/s)
0.67	0.90	0.67	1.18	0.67	1.02	0.67	0.78
0.69	0.93	0.68	1.14	0.69	0.98	0.69	0.80
0.72	1.01	0.71	1.24	0.73	1.19	0.72	1.02
0.75*	1.05	0.74*	1.50	0.74*	1.27	0.75	0.91
0.77	1.41	0.76	1.77	0.77	1.31	0.77	0.87
0.79	1.46	0.79	1.93	0.79	1.33	0.79*	0.95
0.80	1.27	0.80	1.77	0.80	1.35	0.80	1.01
0.81	1.25	0.81	1.61	0.81	1.42	0.81	0.97
0.82	1.29	0.82	1.61	0.82	1.36	0.82	0.95
0.83	1.36	0.83	1.53	0.83	1.32	0.83	1.04

*: the original A_w of each of the samples

To observe the critical A_w value flowability versus A_w was plotted for each of the samples by using Sigmaplot (see Figure 4.3).

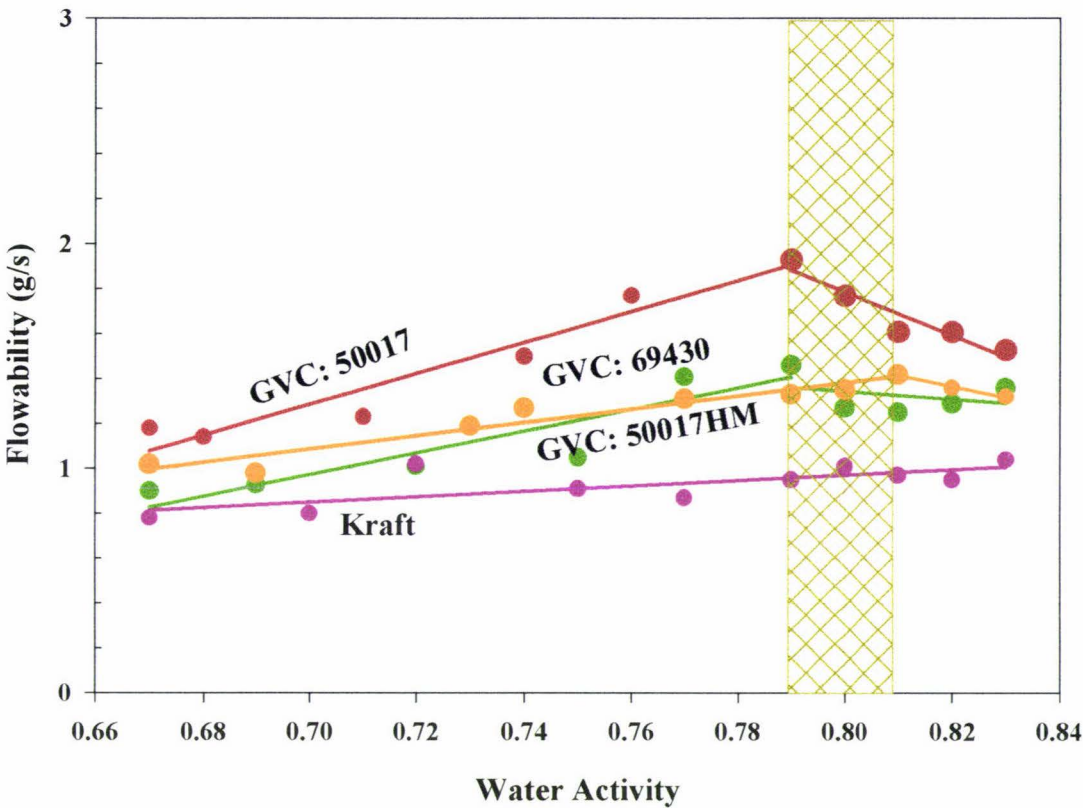


Figure 4.3 Flowability versus A_w of 50017HM, 50017, 69430 and Kraft

Figure 4.3 shows how flowability changed with increasing A_w . For all the three GVC products, flowability was increased up to a critical A_w level (the dark-yellow hatched region in Figure 4.3) and then flowability declined when A_w value was increased beyond the critical value. This critical value for the specification 50017 or 50017HM was at about 0.79 and for 69430 was around 0.81.

The Kraft sample did not show a critical value as prominently as in the case of the GVC samples. This could be because that the flowability variation within each of the samples was much larger than the response on flowability when increasing A_w for Kraft. The greater flowability variation might have hidden the increasing trend for Kraft. On the contrary, the GVC products show the increasing trends significantly. This is because the flowability variation was not great enough to conceal the increasing trend for GVC products.

On the whole, the results show that flowability at first increased when A_w was increased from 0.65 up to the critical A_w value. However, at A_w values higher than the critical values flowability decreased with increasing A_w . This trend was evident in the GVC products rather than in Kraft product. It shows that the critical A_w value is 0.80 ± 0.01 for GVC products.

4.5 Further Testing the Hypothesis of Increasing Flowability by Increasing A_w

In this trial, Grated and Dried Parmesan made on the 10th January 2000 under different GVC specifications (specification 50017 and 50064) were used. Several samples from each specification with different A_w were chosen to do the re-confirmation of the effect of A_w on flowability.

The variation in moisture content that naturally occurred in most drying operations contributed to the variation in water activity of the product. This makes the product naturally suited to study the effect of A_w on flowability.

The samples were packed from the fluid bed into aluminium foil sachets, then heat-sealed to avoid moisture loss and sent to the NZDRI. First the A_w s of each of these samples were measured as described in section 3.2.1.4 and flowability of each of the

samples was measured using the method given in section 3.2.1.1 (see Appendix B-4). The A_w was recorded as the mean value of three measurements, where the flowability was the mean value of 18 measurements (three sub-samples $\times$ six repeated measurements). If there was not enough sample to take three sub-samples, two sub-samples were taken out from each of the samples. Table 4.4 shows the flowability data against A_w of each of the samples.

Table 4.4 The flowability data against A_w for the commercial Grated and Dried Parmesan cheese samples: GVC 50017 and 50064

50017		50064	
A_w	Flowability (g/s)	A_w	Flowability (g/s)
0.70	1.10	0.74	1.06
0.71	1.00	0.74	0.97
0.72	1.13	0.75	1.04
0.72	1.19	0.76	1.21
0.73	1.19	0.75	1.10
0.74	1.21	0.77	1.24
0.74	1.09	0.76	1.17
0.76	1.29	0.77	1.32
0.77	1.33		

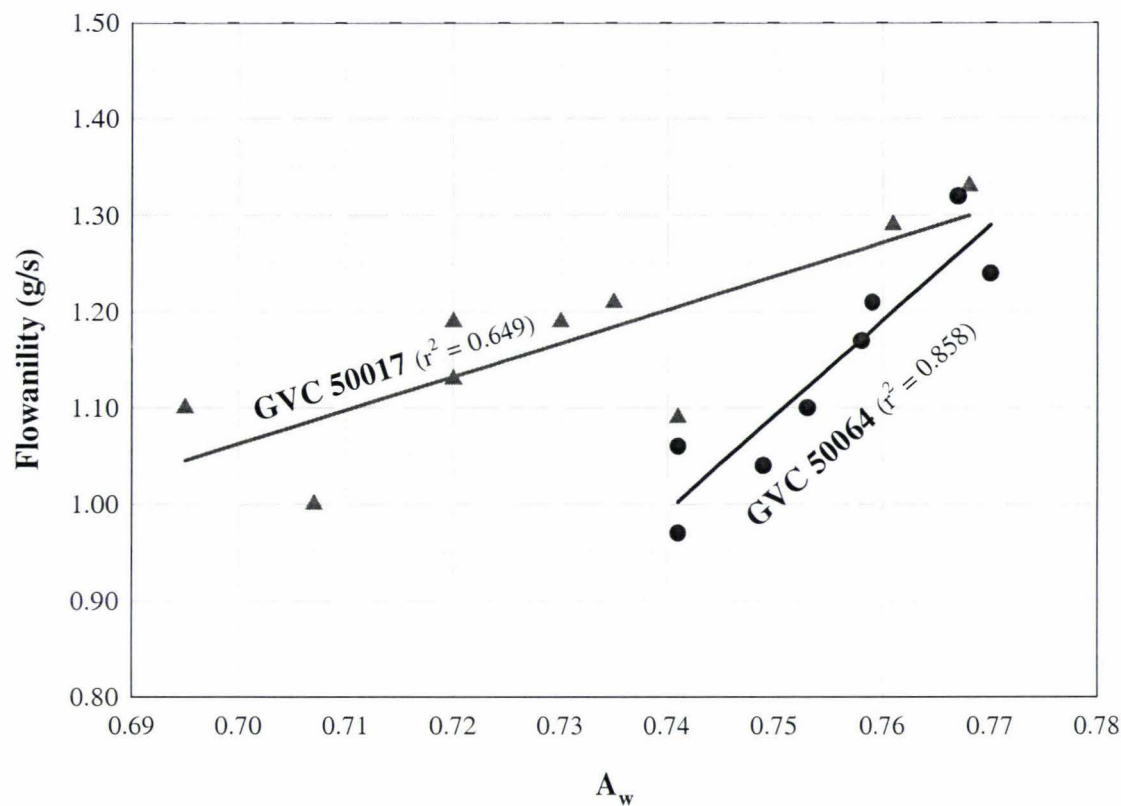


Figure 4.4 The effect of A_w on flowability for the commercial Grated and Dried Parmesan cheese samples: GVC 50017 and 50064

The flowability data against A_w was plotted by using Sigmaplot 4.01 in Figure 4.4. It shows in Figure 4.4 that flowability increased linearly with increasing A_w for both of the GVC samples. The correlation coefficients (r^2) between flowability and A_w for 50017 and 50064 were 0.649 and 0.858 respectively.

4.6 Discussion

To investigate the effect of A_w on flowability of Grated and Dried Parmesan, first, nine GVC Grated and Dried Parmesan cheese samples were used. These nine GVC samples had moisture contents at 18, 21 and 22% at the time of manufacture. For each of these moisture levels, samples were incorporated with or without antimicrobial agents (NA, Sorb and Benz). The result shows that flowability increased with increasing moisture content for both lots of samples with Sorb (0.25% potassium sorbate) and the sample with NA (without additive). The samples with Benz (0.25% sodium benzoate) did not show the trend. If the incorporation with the antimicrobial agents were treated as the replicates, the result of One-way ANOVA analysis of variance shows that the higher the moisture content, the more flowable the sample was.

The hypothesis: flowability increases with increasing A_w was then set up based on the preliminary results and the relationship between moisture content and A_w . Several trials were set up to examine the relationship between flowability and A_w . Three GVC Grated and Dried Parmesan cheese samples (18 NA, 21 NA, 22 NA) plus a Kraft sample were used for A_w manipulation. The relationship between flowability and A_w was obtained for each of the samples. The results show that flowability increased with increasing A_w for each of the GVC Grated and Dried Parmesan cheese samples. There is a correlation between flowability and A_w which fitted the first order mathematical model for the GVC products. It has been realised that there could be a critical A_w between 0.80 to 0.83 above which the GVC product showed a decreasing trend on flowability.

Three GVC products (specification 50017HM, 50017 and 69430) plus one Kraft product were used to reconfirm the hypothesis. The A_w was manipulated into a series of levels. In order to find the critical value, A_w was controlled at an interval of 0.01 between 0.79 to 0.83. Results show that flowability increased with increasing A_w from about 0.65 up to the critical value and then decreased with continuously increasing A_w for each of the GVC samples. The critical A_w for the GVC products was within 0.80 ± 0.01 .

Two commercial GVC Grated and Dried Parmesan cheese samples under different specifications (specification 50017 and 50064) were used to re-confirm the hypothesis. The samples with different A_w values within each of the specification were obtained. The samples with different A_w that naturally occurred during the drying processing were ideally suited to study the effect of A_w on flowability. The results for both the specifications show that the flowability increased lineally with increasing A_w from 0.66 to 0.77.

On the whole, all the results provided the evidence to support the hypothesis: flowability increases with increasing A_w up to the critical value for GVC Grated and Dried Parmesan cheese samples.

The results obtained for the GVC products on the effect of A_w on flowability are in contradiction to what most researchers have observed: either cohesiveness increases or flowability decreases with increasing either moisture content or A_w (Peleg, 1977, Teunou et. al., 1999).

When studying the effect of water activity on the bulk properties of some food powders, Moreyra and Peleg (1981) found that the cohesion increases when increasing A_w for coffee creamer, infant formula, powdered salt and powdered sucrose (Figure 4.5). For example, the cohesion increased when increasing A_w from 0 to 0.73 for a coffee creamer; the cohesion also increases when increasing A_w from 0.22 to 0.73 for a powdered salt. For those two samples, after reaching the peak of cohesion at A_w of 0.73, the cohesion dropped down when continuing to increase A_w due to the liquefaction in the product. Other observations include increased cohesion for onion powder when the moisture content was up from 1% to approximately 6% (Peleg et al, 1973), the decreased flowability for both the flour and the whey-permeate powder when A_w was increased from 0.36 to 0.66 (Teunou et al., 1999).

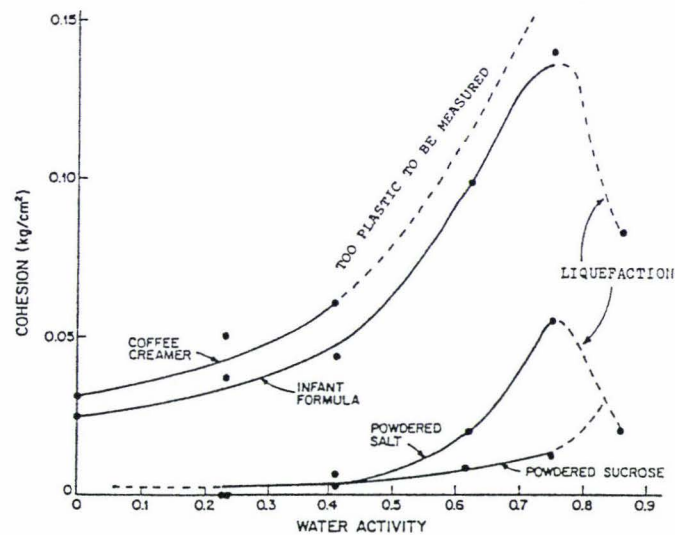


Figure 4.5 Cohesion of powders at different water activities

(Moreyra and Peleg, 1981:1918)

However, the GVC results appear to be in agreement with Buma (1971c)'s results. When he (1971c) studied the influence of water content on cohesion for milk powders, he observed that cohesion first decreased up to 4% moisture content and then increased sharply with continuing increasing water content. This is similar to the results in this study that flowability was increased from A_w 0.65 up to A_w 0.80, and then declined.

Buma (1971c) thought that this is due to either the small surface folds or cracks on the surface of casein particles of milk powders. According to the author, moisture adsorption made swelling of the casein particles and then made surface folds and cracks disappear (Buma, 1971c). In this case, surface roughness declined by reducing the number of contact points between adjacent particles and thence flowability was increased. There might be another reason when free fat is taken into account. When powder particles adsorb water, the pores of powder particles would be reclosed, this would result in a decrease of the free fat content (Buma, 1968) as free fat is a major problem causing cohesiveness by forming liquid bridges on the surface of milk powders (McKenna, 1997). This is likely to have happened in the case of Grated and Dried

Parmesan also, because free fat does exist in it. Based upon the assumption, flowability increases with either an increase in the surface smoothness of particles or a reduction in the free fat content or the combined effects of these two.

When considering the reason responsible for a decreased flowability with continuing increase in A_w , excessive water adsorption could be a possible one. This is because the excessive water adsorption leads to the accumulation of water content on the surface of powder particles. The excessive water content contributes to the formation of the liquid bridges responsible for the decreased flowability. This could explain why after reaching the critical value of A_w , flowability of Grated and Dried Parmesan declined with increasing A_w continuously.

These liquid bridges formed by the excessive water contents were observed by using electron microscopy on milk powders by Buma (1971) and McKenna (1997). Buma (1971c) also observed the folds and small cracks on the surface of milk powder particles.

However, there is no evidence to show these liquid bridges and folds on the surface of Grated and Dried Parmesan particles. The explanations were based on the others' work. If necessary the examination of surface properties of Grated and Dried Parmesan could be carried out and electron microscopy would be a very powerful tool.

With the GVC products, it is concluded that flowability was increased when increasing A_w from 0.65 to the critical value, above which flowability declined. The different specifications had different critical values. The behaviour of the increased flowability with increasing A_w up to the critical value was presumably due to the swelling of either the fold or cracks on the surface of protein particles by water giving an increase in surface smoothness. The latter decrease of flowability was probably because of liquid bridges formed by the excessive water. Confocal microscopy may be a useful tool to elucidate it.

CHAPTER 5

THE EFFECT OF PARTICLE SIZE

ON FLOWABILITY

- RESULTS AND DISCUSSION

This chapter describes the effect of particle size on flowability. The other two factors, A_w and free fat levels of Grated & Dried Parmesan were kept unaltered. Three GVC products (Specification 50017MH, 50017 and 69430) plus one Kraft sample were used in this study.

To study how particle size affects flowability, flowability versus median particle size was plotted for each of the samples. Median particle size (50% of cumulative undersize) was obtained by using the curves of cumulative undersize versus particle size. There are two methods to plot the curves. One is obtained by using Sigmaplot linear regression in which magnitudes of correlation coefficients (r^2) for each of the mixtures were used as the overall measurement of goodness of fit. The other method is by simply joining the points. The flowability against the median particles obtained by both methods were plotted to compare which method is more appropriate.

5.1 The Effect of Particle Size on Flowability of 50017HM

The particle size distribution of one product sample (50017HM) was determined (according to the method in section 3.2.1.2) to find out the proportions of the coarser ($> 600 \mu\text{m}$) and the finer ($< 600 \mu\text{m}$) particles in sample. Table 5.1 shows the sieve analysis data of sample 50017HM.

Table 5.1 Sieve analysis results of sample 50017HM

Sieve size (μm)	Cumulative retained (%)		
	1	2	Average
1700	1.13	1.18	1.16
1000	29.90	29.46	29.68
850	41.12	41.77	41.45
600	55.10*	57.54*	56.32*
355	74.75	80.05	77.40
Pan	25.25	19.95	22.60

**: percentage of the coarser particles(> 600 μm) of 50017HM*

Table 5.1 shows that there were approximately 56% of coarser particles and 44% of finer particles in the original 50017HM. Two different fractions, fraction A (mainly particles > 600 μm) and fraction B (particles < 600 μm) were obtained by sieving the sample through a 600 μm sieve. For the original 50017HM, when only the 600 μm sieve was used to separate, the ratio between fractions A and B was 70/30 approximately. Fraction A and fraction B were then mixed together with different proportions in order to obtain a series of recomposed samples with different particle size distribution (see Table 5.2):

Table 5.2 The mixtures of 50017HM with various proportions of fraction A & B

	M1	M2	M3	M4	M5*	M6	M7	M8	M9
Fraction A (%)	100	85	80	75	70	65	60	55	0
Fraction B (%)	0	15	20	25	30	35	40	45	100
Expressed as	100/0	85/15	80/20	75/25	70/30	65/35	60/40	55/45	0/100

**: 50017HM with the original particle size distribution*

For each of these mixtures, the A_w was measured in triplicate. These samples were tested for their particle size distribution in duplicate and the mean flowability values were calculated for the reported results (Table 5.3). Special attention was paid to the whole testing process when measuring the particle size distribution, because the most easily lost particles were always the finest. For every sieving test, a mass balance was done. In order to eliminate possible error in size distribution determination, the experimental data were rejected if the weight error was greater than 2%.

Table 5.3 The sieve analysis results of each of the mixtures of 50017HM

50017HM			
Cumulative undersize (%)			
Sieve size (µm)	M1 (100/0)	M2 (85/15)	M3 (80/20)
1700	99.03	98.81	99.10
1000	55.80	59.46	61.92
850	37.30	42.57	45.51
600	10.43	20.39	23.50
355	2.08	7.79	10.09
Sieve size (µm)	M4 (75/25)	M5 (70/30)	M6 (65/35)
1700	99.16	99.00	99.40
1000	65.78	73.96	75.14
850	49.97	62.93	63.48
600	28.66	45.78	43.94
355	12.89	19.75	20.44
Sieve size (µm)	M7 (60/40)	M8 (55/45)	M9 (0/100)
1700	99.54	99.44	100.00
1000	78.62	78.46	94.55
850	68.74	69.54	89.68
600	52.70	55.28	79.81
355	25.62	28.25	40.44

To obtain the median particle, the curve of cumulative undersize versus sieve size was plotted either by using Sigmaplot linear regression (Figure 5.1(a)) or by simply joining the points (Figure 5.1(b)). In Figure 5.1(a) all the curves were fitted by using the third-order linear regression based on that the magnitudes of correlation coefficients (r^2) for each of the mixtures were used as the overall measurement of goodness of fit. In Figure 5.1(b) all the point were simply joined together for each of the mixtures as some researchers have done (Neale, 1997, Teunou et al., 1995 and Tuohy, 1989).

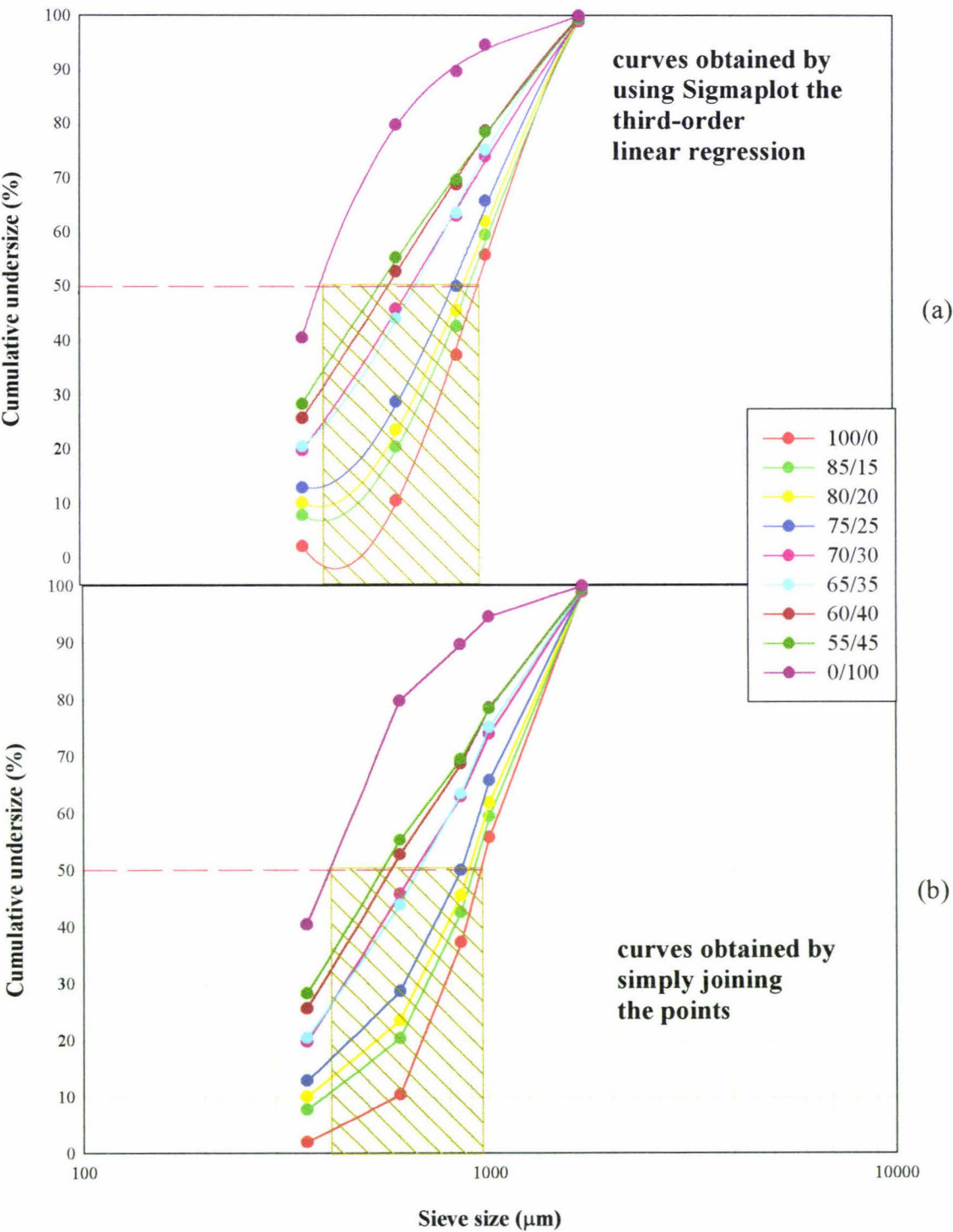


Figure 5.1 Cumulative undersize (%) versus sieve size of 50017HM

The median particle size corresponding to 50% of cumulative undersize for each of the mixtures fell into the range marked by the dark yellow frame in both Figure 5.1(a) and Figure 5.1(b). Median particle size was calculated by using the curves. A_w for each of the mixtures was controlled at 0.740 ± 0.005 around the original A_w of 50017HM with avoidance of the influence of A_w on flowability simultaneously.

For each of the mixtures flowability was measured by using the method in section 3.2.1.1 (see Appendix B-6) and flowability for each of the mixtures was obtained by averaging 36 readings (three sub-samples $\times$ 12 repeated measurements).

Table 5.4 shows median particle size obtained either by using Sigmaplot linear regression (coded as Median size C) or by simply joining the points (coded as Median size D), correlation coefficients (r^2) for each of the curves in Figure 5.1(a), A_w , and flowability of each of the mixtures.

Table 5.4 Correlation coefficient (r^2 , the third-order linear regression) and median particle size, A_w and flowability for each of the mixtures of 50017HM

	M1 (100/0)	M2 (85/15)	M3 (80/20)
r^2	0.999	0.999	0.999
Median particle size $C_{50017HM}$ (μm)	955	912	871
Median particle size $D_{50017HM}$ (μm)	953	916	891
A_w	0.74	0.74	0.74
Flowability (g/s)	1.89	1.78	1.71
	M4 (75/25)	M5 (70/30)	M6 (65/35)
r^2	0.999	0.999	0.999
Median particle size $C_{50017HM}$ (μm)	832	671	655
Median particle size $D_{50017HM}$ (μm)	850	678	662
A_w	0.74	0.74	0.74
Flowability (g/s)	1.52	1.26	1.16

	M7 (60/40)	M8 (55/45)	M9 (0/100)
r^2	0.999	0.999	0.999
Median particle size $C_{50017HM}(\mu m)$	575	550	389
Median particle size $D_{50017HM}(\mu m)$	576	552	414
A_w	0.74	0.74	0.74
Flowability (g/s)	1.14	1.07	0.88

Where Median particle size $C_{50017HM}$ is obtained by using Sigmaplot linear regression;
Median particle size $D_{50017HM}$ is obtained by simply joining the points.

To study the relationship between particle size distribution and flowability, flowability data against two series of median particle size was plotted in Figure 5.2.

Figure 5.2 shows that flowability was greater for samples with a larger median particle size than samples with a smaller median particle size. It was expected because it is well known in powder technology that fine powders are less flowable than coarser powders of the same kind (Buma, 1971).

Figure 5.2 also shows that the relationship between flowability and median particle size distribution fitted the first-order mathematical model. The correlation coefficients between flowability and median particle size obtained by using Sigmaplot regression was 0.963 and by simply joining the points was 0.963 also. There is no difference to set up the relationship between flowability and median particle size obtained by using these two methods.

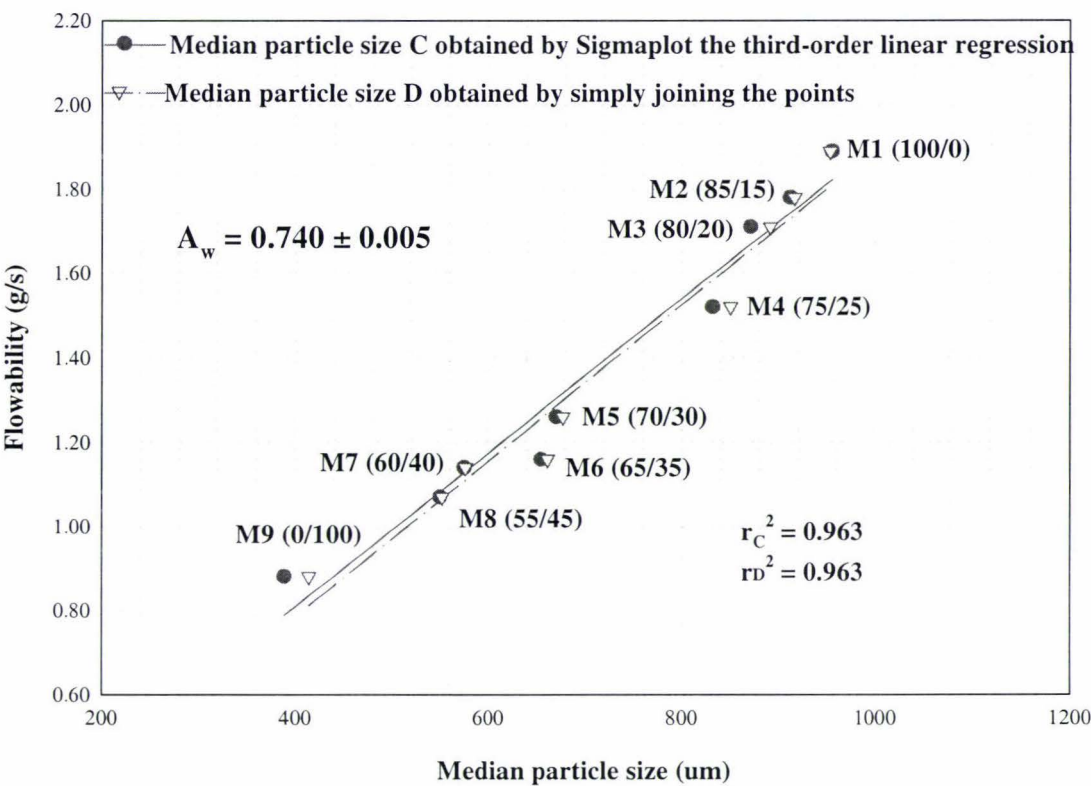


Figure 5.2 Flowability versus median particle size of 50017HM

5.2 The Effect of Particle Size Distribution on Flowability of 50017, 69430 and Kraft

To further study the relationship between flowability and particle size, the same procedures were carried out on GVC samples (Specification 50017 and 69430) plus Kraft sample.

First in order to manipulate the particle size for each of these samples, two different fractions: fraction A (mainly particles > 600 μm) and fraction B (particles <600 μm) were obtained by using the sieve of 600 μm individually. For GVC 50017 and 69430, if only the sieve of 600 μm was used the separation ratio between fraction A and fraction B was about 70/30 on mass whereas it was 75/25 for Kraft sample. Fraction A and fraction B were then mixed together with different proportions to obtain a series of mixtures with different particle size for each of the samples (see Table 5.5).

Table 5.5 The Mixtures with various proportions of fraction A & B of 50017, 69430 & Kraft

		M1	M2	M3	M4	M5*	M6	M7	M8	M9
GVC 50017 & 69430	Fraction A (%)	100	85	80	75	70	65	60	55	0
	Fraction B (%)	0	15	20	25	30	35	40	45	100
	Expressed as	100/0	85/15	80/20	75/25	70/30	65/35	60/40	55/45	0/100
Kraft	Fraction A (%)	100	90	85	80	75	70	65	60	0
	Fraction B (%)	0	10	15	20	25	30	35	40	100
	Expressed as	100/0	90/10	85/15	80/20	75/25	70/30	65/35	60/40	0/100

*: the samples with original particle size distribution

For each of the mixtures, particle size distribution was measured in duplicate and their mean values were calculated for the reported results (See Appendix D-1). In order to eliminate possible error in size distribution determination, the experimental data were rejected if the weight error was greater than 2%.

In order to express the relationship between flowability and particle size, the curve of cumulative undersize (%) versus sieve size was plotted either by using Sigmaplot linear regression where the magnitudes of correlation coefficients (r^2) for each of the mixtures were used as the overall measurement of goodness of fit or by simply combining the points for each of the mixtures of every sample (See Appendix D-3). Then the median particle size for each of the mixtures was calculated from the curves.

As A_w is another factor affecting flowability, in order to minimise the influence of A_w on flowability simultaneously, A_w was controlled at the original A_w value ± 0.005 for each of the mixtures within each of the samples. For each of the mixtures flowability was measured (see Appendix B-6) and flowability values were obtained by averaging 36 measurements (three sub-samples $\times$ 12 repeated measurements). Table 5.6 shows the median particle size obtained by the two methods (coded as median size C obtained by regression and median size D obtained by simply joining the points, A_w and flowability for each of the mixtures.

Table 5.6 The median particle size, A_w and flowability for each of the mixtures of 50017, 69430 and Kraft

	50017		
	M1 (100/0)	M2 (85/15)	M3 (80/20)
Median particle size C ₅₀₀₁₇ (µm)	973	916	889
Median particle size D ₅₀₀₁₇ (µm)	968	923	901
A_w	0.73	0.73	0.73
Flowability (g/s)	1.83	1.82	1.71

	M4 (75/25)	M5 (70/30)	M6 (65/35)
Median particle size C ₅₀₀₁₇ (μm)	843	836	817
Median particle size D ₅₀₀₁₇ (μm)	867	861	838
A _w	0.72	0.73	0.72
Flowability (g/s)	1.61	1.64	1.42
	M7 (60/40)	M8 (55/45)	M9 (0/100)
Median particle size C ₅₀₀₁₇ (μm)	781	760	454
Median particle size D ₅₀₀₁₇ (μm)	816	778	496
A _w	0.72	0.72	0.72
Flowability (g/s)	1.37	1.34	0.85

Where median particle size C₅₀₀₁₇ is obtained by Sigmaplot linear regression;
Median particle size D₅₀₀₁₇ is obtained by simply joining the points.

	69430		
	M1 (100/0)	M2 (85/15)	M3 (80/20)
Median particle size C ₆₉₄₃₀ (μm)	1009	982	962
Median particle size D ₆₉₄₃₀ (μm)	995	969	952
A _w	0.74	0.74	0.74
Flowability (g/s)	1.35	1.20	1.25
	M4 (75/25)	M5 (70/30)	M6 (65/35)
Median particle size C ₆₉₄₃₀ (μm)	935	899	867
Median particle size D ₆₉₄₃₀ (μm)	938	915	900
A _w	0.74	0.74	0.74
Flowability (g/s)	1.28	1.10	1.09
	M7 (60/40)	M8 (55/45)	M9 (0/100)
Median particle size C ₆₉₄₃₀ (μm)	839	828	619
Median particle size D ₆₉₄₃₀ (μm)	889	867	623
A _w	0.74	0.74	0.73
Flowability (g/s)	1.07	0.97	0.85

Where median particle size C_{69430} is obtained by using Sigmaplot linear regression;
Median particle size D_{69430} is obtained by simply joining the points.

	Kraft		
	M1 (100/0)	M2 (85/15)	M3 (80/20)
Median particle size C_{Kraft} (μm)	724	695	664
Median particle size D_{Kraft} (μm)	714	703	670
A_w	0.78	0.78	0.78
Flowability (g/s)	1.38	1.19	1.05
	M4 (75/25)	M5 (70/30)	M6 (65/35)
Median particle size C_{Kraft} (μm)	614	594	574
Median particle size D_{Kraft} (μm)	612	587	561
A_w	0.79	0.78	0.78
Flowability (g/s)	1.23	1.11	1.04
	M7 (60/40)	M8 (55/45)	M9 (0/100)
Median particle size C_{Kraft} (μm)	500	452	161
Median particle size B_{Kraft} (μm)	487	432	214
A_w	0.78	0.78	0.78
Flowability (g/s)	1.08	0.92	0.68

Where median particle size C_{Kraft} is obtained by using Sigmaplot linear regression;
Median particle size D_{Kraft} is obtained by simply joining the points.

To illustrate the relationship between particle size distribution and flowability, flowability versus median particle size obtained by both the methods was plotted. Figures 5.3 (a) to (c) show the relationship between flowability and median particle size for each of the samples.

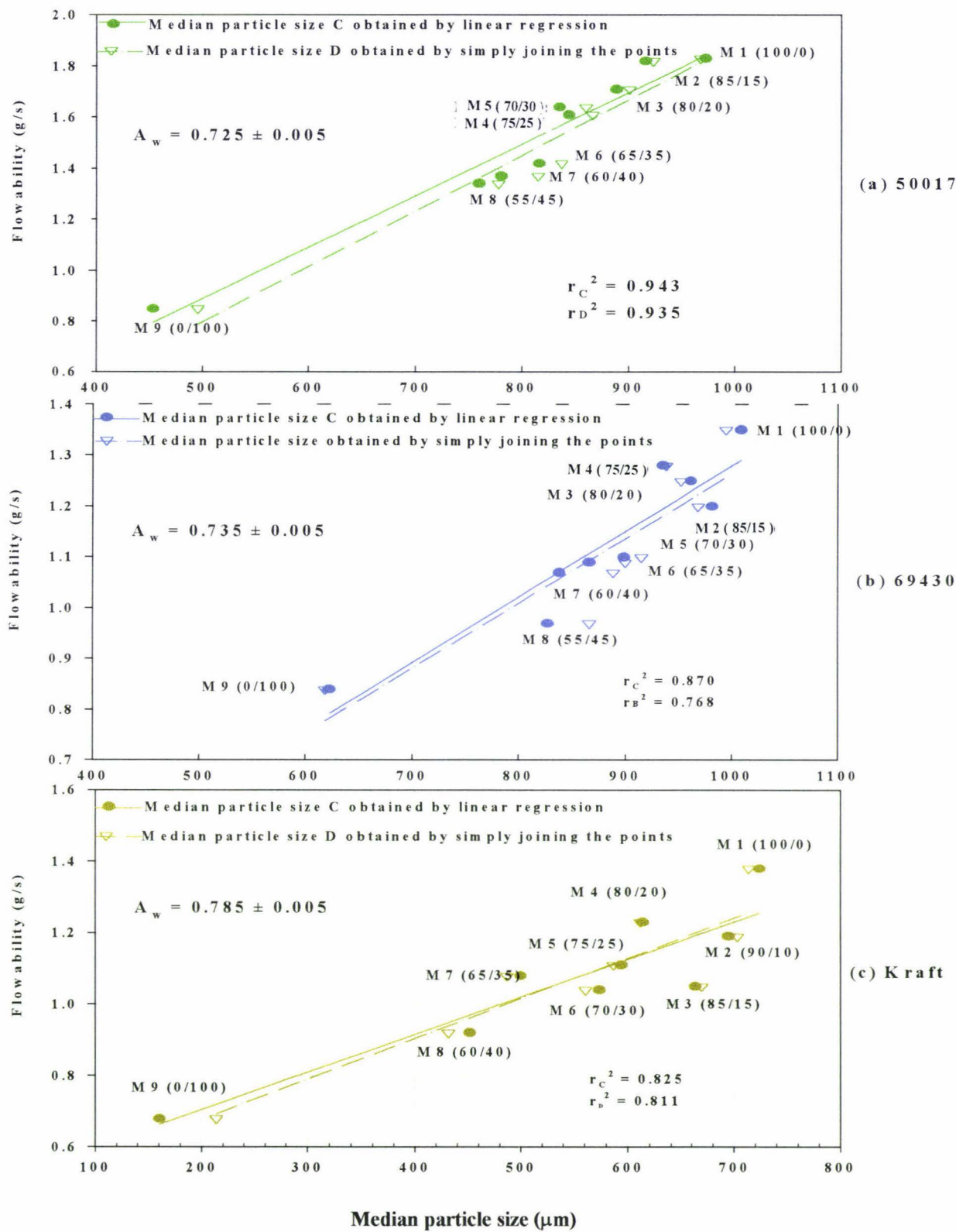


Figure 5.3 (a) to (c) Flowability versus median particle size of 50017, 69430 and Kraft

Figure 5.3 shows that flowability increases with increasing median particle size for each of the samples. The relationship between flowability and median particle size obtained by both methods fitted a first order mathematical model.

5.3 Discussion

5.3.1 The effect of particle size on flowability

The effect of particle size on flowability had been studied by using four different samples. Particle size was expressed in term of median particle size, then the relationship between flowability and median particle size was obtained. Figure 5.4 summarises the relationship between flowability and median particle size obtained by using Sigmaplot linear regression for each of the samples. Generally speaking, flowability increases with increasing median particle size for every sample. The relationship between flowability and median particle size for each of these samples fitted a first-order mathematical model.

It is also shown in Figure 5.4 the rates of increase on flowability with increasing median particle size. Among the four samples, GVC 50017 had the greatest rate of about 0.19 g/s per 100 μ m, followed by 50017HM, 0.18 g/s per 100 μ m and 69430, 0.13 g/s per 100 μ m. Kraft had the lowest rate of increasing flowability with increasing median particle size, which was 0.10 g/s per 100 μ m. This could suggest that increasing flowability by increasing particle size on GVC Grated and Dried Parmesan would be more effective than by increasing particle size on Kraft.

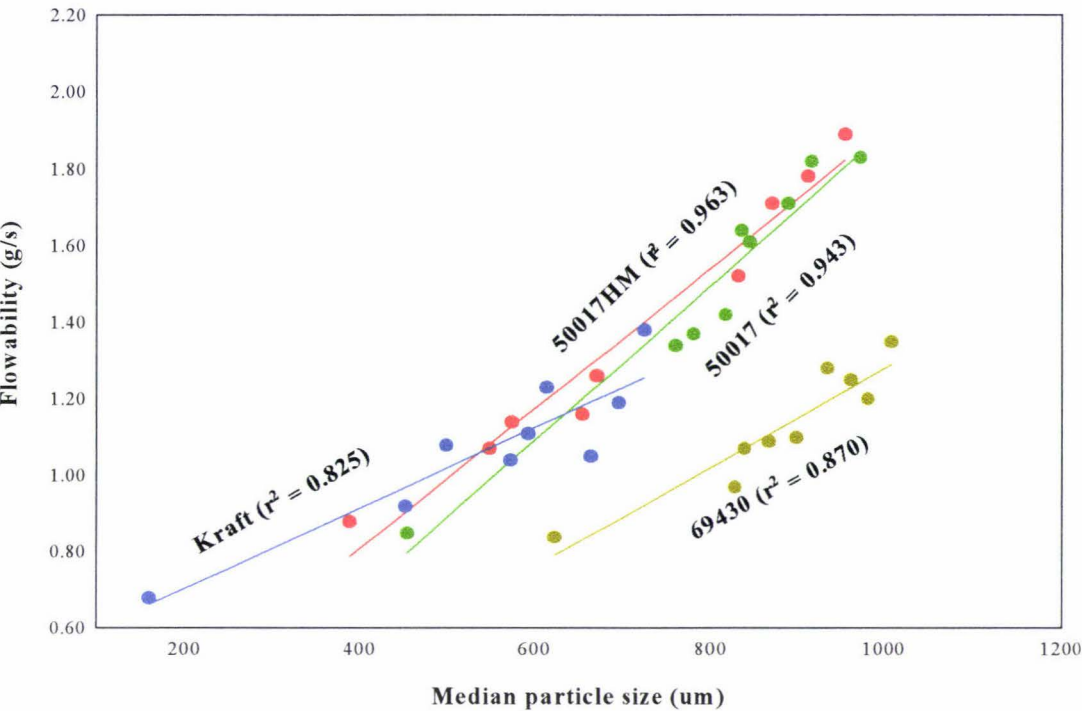


Figure 5.4 Flowability versus median particle size obtained by using Sigmaplot linear regression of various products

It is well accepted in powder technology that coarser powders are more flowable than finer powders of the same kind (Buma, 1971a; Peleg, 1977 and Retsina and Coucoulas, 1988). According to Retsina and Coucoulas (1988), this is because large particles minimise surface contact between particles and therefore reduce the interparticle forces (cohesiveness) and this leads to an increase on flowability (Peleg, 1977).

Furthermore, there could be another reason if Buma (1971a)’s observation on whole milk powder is taken into account. He found that small particles in a milk powder sample contained much more free fat than the larger particles of the same sample. It has been found that the free fat on the surface of particles can form liquid bridges that block the particles flow (Peleg, 1977). Logically, this could contribute to the flow problem on

Grated and Dried Parmesan also. Further investigation to observe the distribution of free fat could be worthwhile. Techniques such as confocal laser microscopy may be appropriate.

5.3.2 Methods chosen to plot the cumulative undersize versus sieve size

As to the method used to obtain the median particle size, simply joining the points is widely used (Teunou et al., 1997 and Tuohy, 1989). In this study, the median particle size was obtained either by simply joining the points or by using Sigmaplot linear regression to give a best fit.

The correlation coefficients (in Table 5.7) for the relationship between flowability and median particle size obtained by using linear regression were slightly higher than those on the flowability against median particle size obtained by simply joining the points. This could implicate that median particle size obtained by linear regression is more suitable to plot the flowability against median particle size rather than those obtained by simply joining the points.

Table 5.7 Coefficient (r^2) of the relation between flowability and median particle size obtained either by using regression or by simply joining the points

		50017HM	50017	69430	Kraft
r^2	Median particle size obtained by regression	0.963	0.943	0.870	0.825
	Median particle size obtained by simply joining the points	0.963	0.935	0.768	0.811

The advantage by using Sigmaplot linear regression is that all the points are taken into account. By using simply joining the points, only the two values adjacent to the median particle size are used to get the median particle size. The difference between using the best fit regression to obtain the median particle size and using simply joining the points to obtain the median particle is illustrated by giving a hypothetical example below:

Powder M and powder N are two different powders with different particle size distribution which is shown in Table 5.8.

Table 5.8 Sieve analysis results of two hypothetical powder samples

Cumulative undersize (%)		
Sieve size (μm)	Powder M	Powder N
100	10	0
200	20	30
300	40	40
400	80	80
500	82	85
600	100	100

To obtain the median particle size of each powder, the cumulative undersize (%) against sieve size was plotted either by using best-fit regression line (Figure 5.5 (a)) or by using simply joining the points (Figure 5.5 (b)). Then median particle sizes, coded as D_M for powder M and D_N for powder N, which correspond to 50% of cumulative undersize, are obtained.

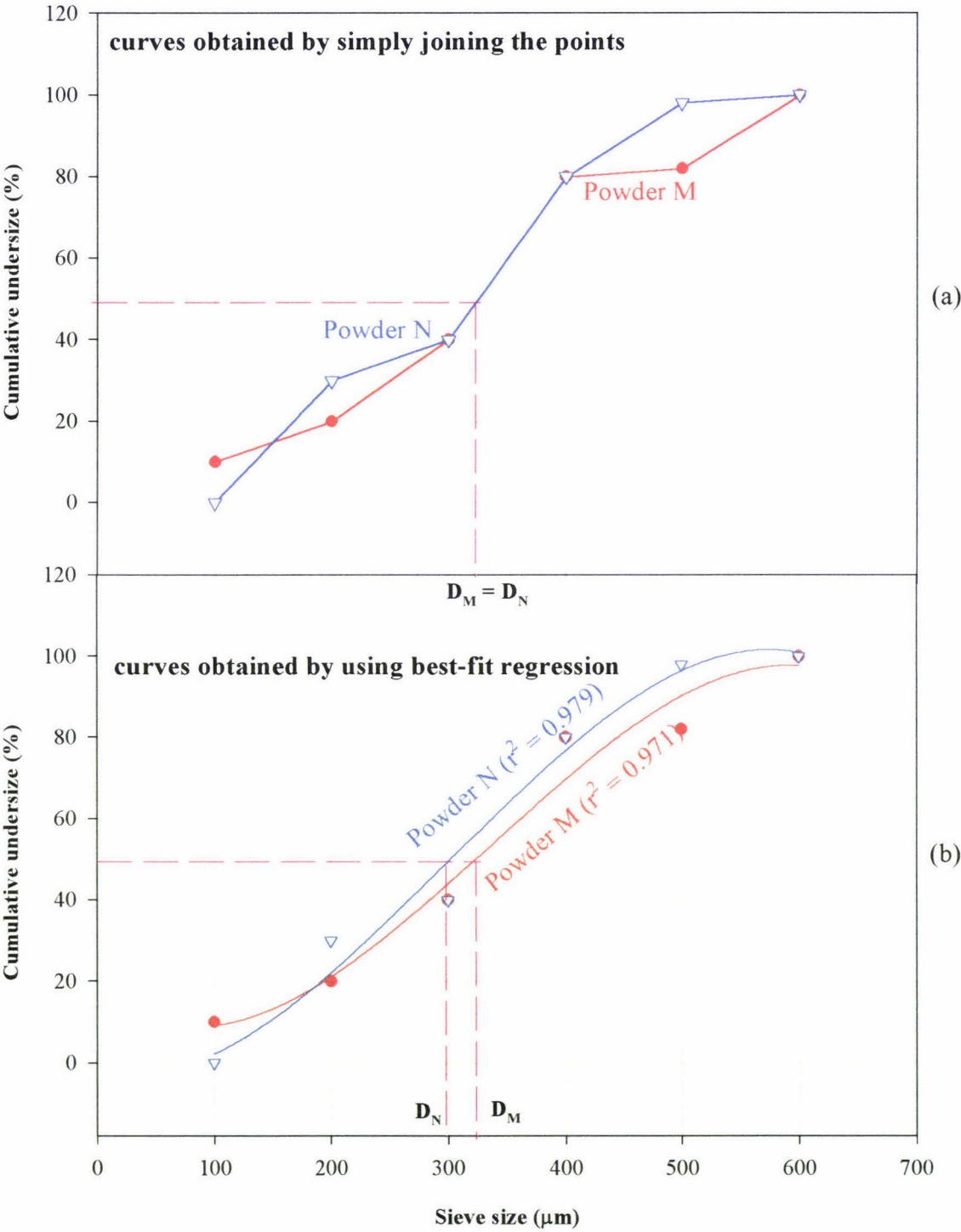


Figure 5.5 Cumulative undersize (%) versus sieve size of hypothetical powders M & N (D_M & D_N : median particle size of powder M & N)

Figure 5.5 (a) shows if the curves of cumulative undersize (%) versus sieve size is obtained by simply the points, powder M and powder N have the same median particle size ($D_M = D_N$) as the two powders have the same cumulative undersize value at the two sieve size (300 and 400 μm) around 50% cumulative undersize. In this case, only two points (40% and 60% cumulative undersize) are taken into account. In fact, powder M and N are totally different powders, they have different median particle size as shown in Figure 5.5 (b).

The advantage of using best-fit regression line to plot the curve of cumulative undersize (%) against sieve size is that all the points are taken into account, which is thought to represent the more accurate particle size distribution than simply joining the points does. Therefore obtaining the cumulative curves by using linear regression is recommended rather than that by using simply joining the points.

CHAPTER 6

THE EFFECT OF FREE FAT ON FLOWABILITY

- RESULTS AND DISCUSSION

The purpose of this chapter was to study the effect of free fat content on flowability. The water activity of each of the samples at different free fat levels was kept constant. The particle size distribution of each of the samples was measured.

6.1 The Effect of Free Fat

One GVC sample (specification 50017HM) was used to evaluate the effect of free fat on flowability. The original free fat content of the sample was determined to be 22%. The free fat contents of eight sub-samples were changed to span a range (see Table 6.1).

The following procedure was used to obtain the range of free fat levels to study the effect of free fat on flowability. A large amount (500 g) of sub-sample from the original samples were subjected to free fat extraction (refer to section 3.2.1.3). This sample was then divided into three portions. Different levels of anhydrous milk fat (AMF) were then sprayed onto these three portions. The quantity of AMF was adjusted to span a range up to 22% free fat. Different quantities of free fat were sprayed onto five sub-samples drawn from the original sample to cover a range above 22%. The method for calculating the quantity of AMF sprayed was given in section 3.2.2.3.2. Thereby, a series of free fat levels that were higher and lower than 22% were obtained (see Table 6.1).

Table 6.1 The target free fat levels of the sample 50017HM

	F1	F2	F3	F4*	F5	F6	F7	F8	F9
Free fat (%)	0	17	18	22	23	24	25	26	35

**: 50017HM with the original free fat content*

The A_w was measured in triplicate for each of the sub-samples with different free fat levels. During the preparation of samples with different free fat levels, A_w values for some of the sub-samples dropped down due to moisture loss. In order to minimise the influence of A_w on flowability, A_w s were re-adjusted to 0.745 ± 0.005 , which was the A_w of the original sample. The A_w adjustments were made by using the method given in section 3.2.2.1.

The samples with different free fat contents but with the same water activity were tested for their flowability. The flowability was reported by averaging the 36 readings (three sub-samples $\times$ 12 repeated measurements) taken for each of the sub-samples (see Appendix B-7). The exact free fat contents of each of the sub-samples were also measured in duplicate by using the method given in section 3.2.1.3. Table 6.2 shows the A_w , free fat content and flowability of each of the samples with different free fat contents.

**Table 6.2 A_w , free fat and flowability
for each of the samples with different free fat contents of 50017HM**

	F1	F2	F3	F4	F5	F6	F7	F8	F9
A_w	0.75	0.75	0.75	0.75	0.74	0.75	0.75	0.75	0.74
Free fat (%)	0.7	16.8	18.4	22.5	22.9	24.0	24.9	26.3	35.3
Flowability (g/s)	1.56	2.29	2.02	2.00	1.42	1.34	0.92	0.56	*Flow failure

**: Samples did not flow from the rotating drum due to aggregation*

To illustrate how flowability varies with changing free fat content, flowability versus free fat was plotted in Figure 6.1.

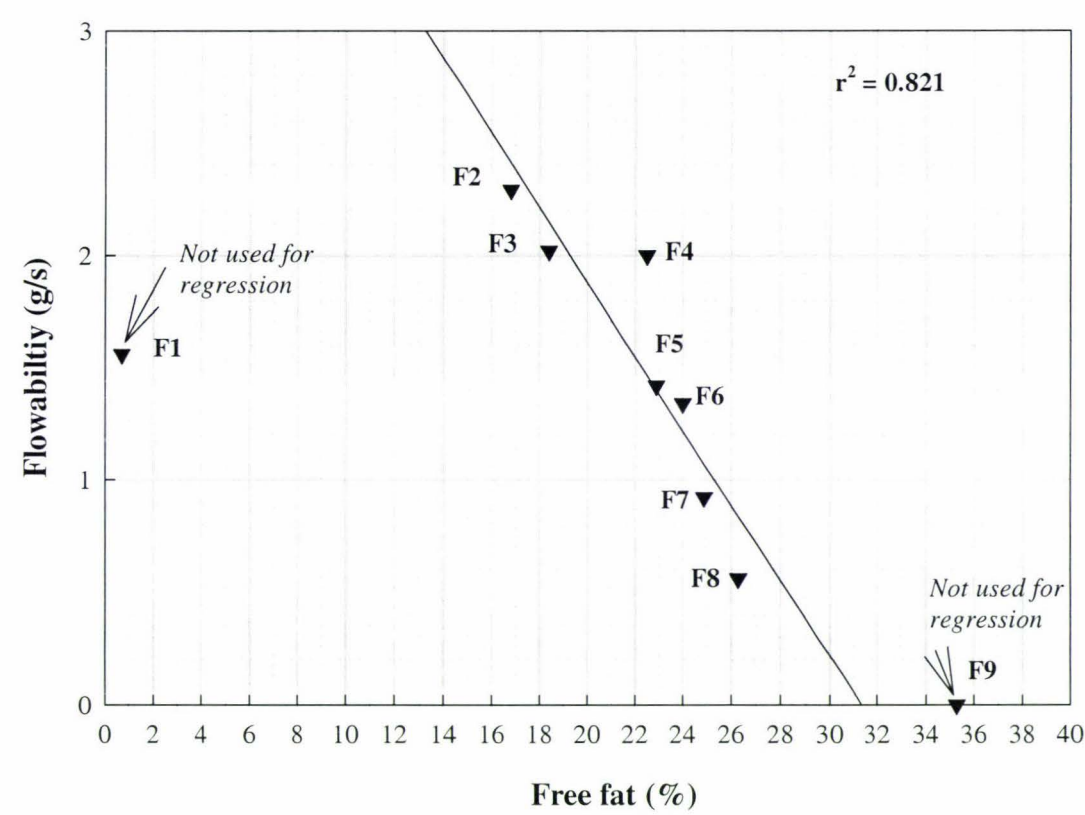


Figure 6.1 Flowability versus free fat content of 50017HM

Figure 6.1 shows the effect of free fat on flowability. Neither F1 nor F9 are used for regression as these two extreme situations seldom happen. As F9 had a high free fat level of 35.3%, serious clumping presumably formed by the viscous fat bridges had been observed. This led to flow failure in F9. The figure shows that the flowability decreased when increasing free fat content from 16.8% to 26.3%. The relationship between flowability and free fat content fitted a first-order mathematical model, where the correlation coefficient (r^2) was 0.821.

6.2 Free Fat and Particle Aggregation

The effect of free fat on particle aggregation was also investigated. The particle size was measured of each of the mixtures in duplicate and sieve analysis results were averaged for the two measurements (see Appendix D-2). The particle size was expressed in terms of median particle size. To obtain the median particle size for each of the samples, the cumulative undersize versus sieve size is plotted (see Appendix D-4).

The median particle size, which corresponds to 50% of cumulative undersize for each of the mixtures, was calculated by using the curves. Table 6.3 shows the median particle size, free fat content, and flowability for each of the samples with different free fat contents.

**Table 6.3 Median particle size, free fat and flowability
for the samples with different free fat contents of 50017HM**

	F1	F2	F3	F4	F5
Median particle size (µm)	619	721	736	757	778
Free fat (%)	0.7	16.8	18.4	22.5	22.9
Flowability (g/s)	1.56	2.29	2.02	2.00	1.42
	F6	F7	F8	F9	
Median particle size (µm)	924	1133	1476	too sticky to be sieved	
Free fat (%)	24.0	24.9	26.3	35.3	
Flowability (g/s)	1.34	0.92	0.56	flow failure	

Table 6.3 shows that the median particle size increased with increasing free fat content. The median particle size versus free fat level excluding F1 and F9 was plotted as shown in Figure 6.2.

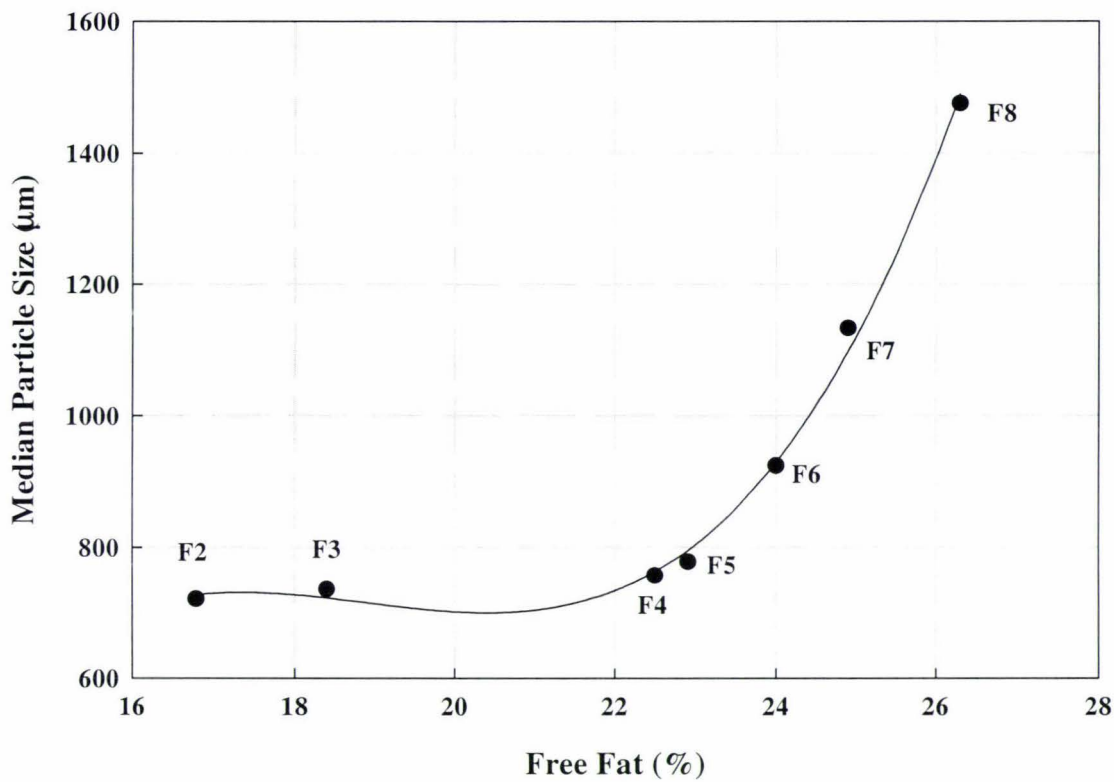


Figure 6.2 The effect of free fat on median particle size

It shows in Figure 6.2 that median particle size increased from 721 to 1476 µm when free fat level increased from 16.8 to 26.3%. It appears that median particle size increased more rapidly above 22.9% of free fat level (as shown as F5) rather than that below this free fat level.

6.3 Discussion

6.3.1 The effect of free fat on flowability

Without considering the effect of free fat on particle aggregation, the results showed that flowability decreased with increasing free fat level from 16.8 to 26.3%. This is in agreement with Eisses and Duiven's observation (1968). These workers found a negative correlation between free fat content and free flowingness of spray-dried whole milk without considering particle size. This is in contradiction to Buma (1971c)'s observation. He found that there is no correlation between free fat content and cohesion of spray-dried milk powders with approximately the same particle size.

However, the results obtained were in agreement with the current recognition of the formation of viscous liquid bridges, which affects the flowability of fat containing powders (Peleg, 1977). In this study, the particles were mixed with the free fat at 60°C at which temperature the fat was in a liquid state. Later on, when the samples were sealed in aluminium foil sachets, cooling of powders down to the ambient temperature solidified the free fat. This solidification would have been responsible for the formation of crystalline fat bridges between the particles. This would have greatly increased the cohesion and decreased flowability of the powders.

These fat bridges between particles of spray-dried whole milk have been observed by Buma (1971c) through a scanning electron microscope. No information is available in the scientific literature on Grated and Dried Parmesan so far. If necessary a further study by using scanning electron microscope could be carried out to observe the fat bridges on the surface of Grated and Dried Parmesan cheese particles.

6.3.2 The effect of free fat on particle aggregation

The results show that the median particle size increased with increasing free fat content. This is probably because the more free fat in the sample, the stickier it is likely to be due to the formation of free fat liquid bridges between particles (Peleg, 1977). It appears that the mechanical shaking has accelerated the formation of aggregates. This particle aggregation was observed on the top sieve after sieving especially for the sample F6, F7 and F8. Particle aggregation formed by free fat bridges is one of the potential reasons that led to an increase in median particle size

6.3.3 Is there a net effect of free fat and particle size on flowability?

As both free fat and particle size are factors influencing flowability, their effects need to be considered together. The median particle size data of the free fat study (from Table 6.3) were plotted against the free fat data given in Table 6.3 (see Figure 6.2). The samples with different free fat contents had a median particle size range of 721 – 1476 μm . Then the flowability values of the free fat study were plotted against the median particle size of the respective samples. The flowability data of the study conducted on effect of particle size distribution on flowability were also plotted on the same graph and they are shown in Figure 6.3.

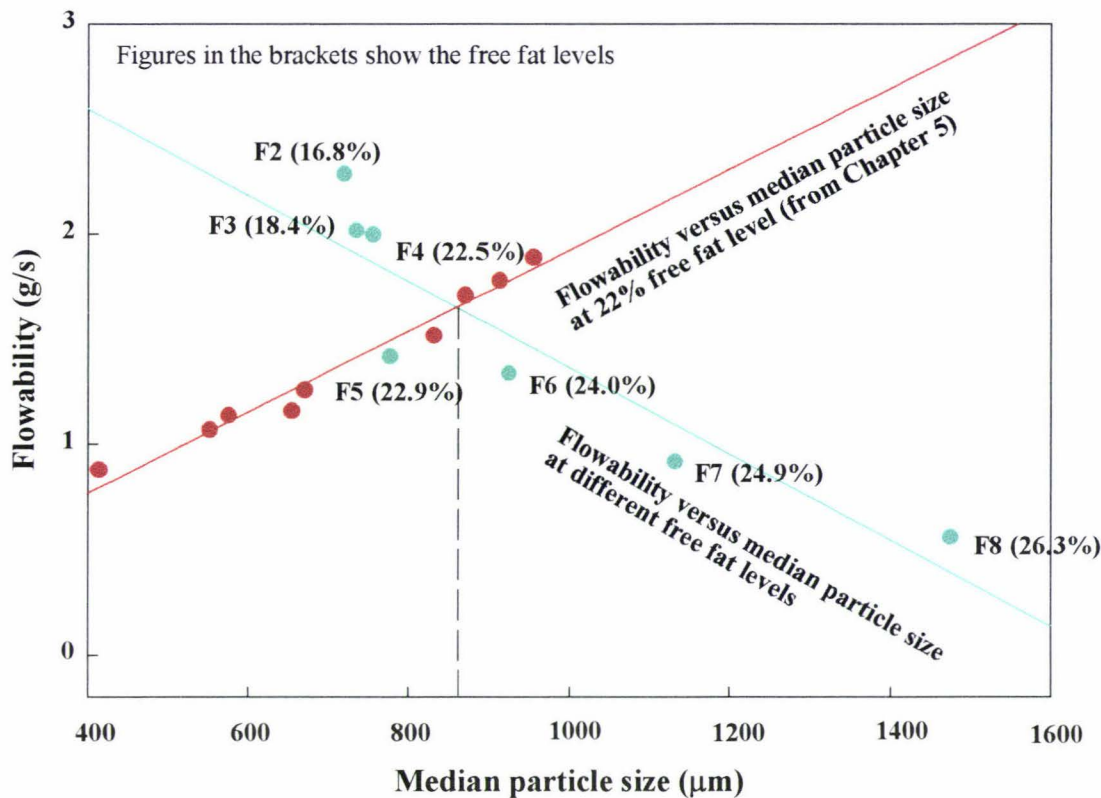


Figure 6.3 Flowability versus median particle size at both the same and different free fat levels

It appears in Figure 6.3 that at median particle size about 850 μm , the two series have the same flowability. Below 850 μm , lower free fat levels indicated by F2 to F5 demonstrated higher flowability than the samples with higher free fat levels. Above 850 μm , lower free fat levels (22%) also demonstrated higher flowability than the points indicated by F6, F7 and F8. However, these results show that the positive effect of particle size could not counteract the effect of free fat. The net effect of free fat and particle size led to a decrease on flowability.

Figure 6.3 only shows flowability versus median particle size at 22% free fat level together with flowability versus median particle size at different free fat levels. So the observation is hypothetical and incomplete. Further investigation on the net effects of free fat and particle size could be developed by manipulating samples to different free fat levels such as 24% or 20% but with the sample particle size distribution.

CHAPTER 7

CONCLUSIONS

This is a fundamental study on flowability of Grated and Dried Parmesan. The study was focused on evaluating the three basic and important factors influencing flowability. The three factors are A_w , particle size distribution and free fat content.

The data obtained on the effect of A_w on flowability shows that flowability increased with increasing A_w for GVC products. There was a critical A_w value for GVC product. Below this value, flowability increased with increasing the A_w . Above this value, flowability decreased with increasing A_w . The critical A_w value for GVC products was determined at 0.80 ± 0.01 .

The effect of particle size distribution on flowability was evaluated. The results show that flowability increased linearly with increasing particle size for both GVC and Kraft products.

Particle size distribution was expressed in terms of median particle size (at 50% undersize on mass). The median particle size was obtained either by using a Sigmaplot regression line or by simply joining the points. It is shown that there is no significant difference in extrapolating flowability versus median particle size obtained by either method in this study. Nevertheless, using a regression line to obtain median particle size is still recommended as all the points are taken into account in this method, whereas, only two points are considered when using simply joining the points.

The study of free fat on flowability shows that flowability decreased when the free fat content was increased from 16.8% to 26.3%. Sieving analysis results show that median particle size increased with increasing free fat. This is because the more free fat, the more likely the sample is to aggregate. Shaking accelerated the particle aggregation. It seems that the positive effect of particle size could not counteract the negative effect of free fat on flowability. Whether there is a net effect of free fat and particle size or not should be further studied.

FURTHER WORK

Flowability is influenced by both internal and external factors or interaction of both. This study was just focused on the effect of A_w , particle size distribution and free fat. Further investigations from this study are recommended as follows:

- Study the combined effects of A_w , particle size distribution and free fat on flowability.
 - Investigate the effect of external conditions such as storage temperature and storage time on flowability.
 - Examine the combined effects of external and internal factors in order to find the optimum combinations of production, transportation and storage conditions to provide the product with good flowability.
 - Explore the possibilities of using confocal microscopy. This would be useful in observing the surface properties of particles to give particular evidence on moisture dispersion and liquid fat bridges.
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APPENDICES

Appendix A Free fat measurement (MP-MT-FF01)

1. Dry 125 ml Erlenmeyer flask containing a few anti-bumping granules in an oven at 102°C for an hour.
 2. Cool for one hour.
 3. Weigh accurately (± 0.0001 g) (record as W_1)
 4. Weigh 10.0 g (± 0.1 g) powder into 250 ml Erlenmeyer flask.
 5. Measure 50 ml of petroleum spirit and pour into the 250 ml flask containing the powder sample.
 6. Stopper the flask and shake using a shaking machine for 5 minutes.
 7. Allow the powder to settle for a few seconds and pour the petroleum spirit through a filter paper into the weighted 125 ml flask, retaining as much powder as possible in the 250 ml flask.
 8. Measure a further 50 ml petroleum spirit into the 250 ml flask and shake 5 minutes.
 9. Add this to the 125 ml flask, filtering through the same filter paper.
 10. Rinse the 250 ml flask thoroughly with 25 ml petroleum and filter into the same 125 ml flask.
 11. Evaporate the petroleum spirit to dryness on a steam bath.
 12. Dry the 125 ml flask in an oven at 102°C for 1 hour.
 13. Cool for an hour and re-weigh (record as W_2).
 14. Calculate free fat as: $(W_2 - W_1 - W_b) \times 100 / \text{weight of sample}$
 W_2 : weight of flask and free fat
 W_1 : weight of flask
 W_b : weight change of blank
 15. Report to nearest 0.1%.
-

Appendix B-1

The flowability data against the moisture content in the preliminary study

Repeated measurement	18, NA 1#	18, NA 2#	18, NA 3#	21, NA 1#	21, NA 2#	21, NA 3#
1	1.26	1.03	0.99	1.25	1.22	1.60
2	1.34	1.16	1.06	1.41	1.37	1.75
3	1.35	1.20	1.13	1.40	1.46	1.70
4	1.35	1.22	1.08	1.45	1.54	1.79
5	1.31	1.26	1.10	1.50	1.59	1.81
6	1.21	1.21	1.18	1.57	1.68	1.79
7	1.16	1.14	1.11	1.61	1.61	1.89
8	1.19	1.23	1.17	1.56	1.70	1.88
9	1.18	1.16	1.14	1.61	1.66	1.87
10	1.11	1.18	1.15	1.65	1.67	1.75
11	1.13	1.17	1.18	1.61	1.63	1.84
12	1.15	1.18	1.17	1.66	1.68	1.82
Repeated measurement	22, NA 1#	22, NA 2#	22, NA 3#			
1	2.31	2.32	2.10			
2	2.32	2.30	2.08			
3	2.28	2.30	2.24			
4	2.31	2.29	2.24			
5	2.36	2.36	2.27			
6	2.38	2.37	2.32			
7	2.38	2.40	2.33			
8	2.43	2.45	2.31			
9	2.42	2.40	2.34			
10	2.42	2.41	2.36			
11	2.36	2.42	2.34			
12	2.43	2.41	2.35			
Repeated measurement	18, Sorb 1#	18, Sorb 2#	18, Sorb 3#	21, Sorb 1#	21, Sorb 2#	21, Sorb 3#
1	1.47	1.24	1.15	1.48	1.35	1.18
2	1.53	1.26	1.15	1.53	1.19	1.29
3	1.49	1.29	1.17	1.57	1.48	1.34
4	1.47	1.26	1.18	1.55	1.53	1.35
5	1.50	1.32	1.16	1.65	1.54	1.38
6	1.46	1.30	1.14	1.67	1.53	1.40
7	1.40	1.31	1.09	1.67	1.59	1.60
8	1.42	1.25	1.07	1.55	1.55	1.70
9	1.30	1.18	1.14	1.54	1.58	1.71
10	1.22	1.22	1.11	1.49	1.54	1.75
11	1.22	1.15	1.12	1.58	1.53	1.72
12	1.30	1.19	1.10	1.57	1.50	1.80
Repeated measurement	22, Sorb 1#	22, Sorb 2#	22, Sorb 3#			
1	1.87	1.69	1.61			
2	2.06	1.68	1.63			
3	2.10	1.80	1.67			
4	2.13	1.81	1.55			
5	2.16	1.97	1.69			
6	2.23	1.97	1.86			
7	2.25	2.02	1.95			
8	2.24	2.02	2.03			
9	2.25	1.95	2.07			
10	2.30	1.95	2.12			
11	2.25	1.98	2.08			
12	2.26	1.87	2.03			
Repeated measurement	18, Benz 1#	18, Benz 2#	18, Benz 3#	21, Benz 1#	21, Benz 2#	21, Benz 3#
1	1.29	1.29	1.06	1.93	1.72	1.69
2	1.31	1.34	1.20	2.03	1.86	1.74
3	1.32	1.45	1.33	2.08	1.94	1.80
4	1.38	1.47	1.42	2.05	1.96	1.80
5	1.45	1.49	1.37	2.11	2.01	1.82
6	1.43	1.58	1.42	2.13	1.99	1.88
7	1.43	1.51	1.44	2.12	2.07	1.91
8	1.42	1.50	1.38	2.17	2.02	1.88
9	1.43	1.54	1.47	2.13	2.05	1.95
10	1.41	1.44	1.38	2.15	2.07	1.95
11	1.43	1.43	1.38	2.14	1.99	1.94
12	1.38	1.43	1.33	2.12	2.01	1.97
Repeated measurement	22, Benz 1#	22, Benz 2#	22, Benz 3#			
1	1.49	1.30	1.39			
2	1.45	1.34	1.44			
3	1.44	1.43	1.51			
4	1.40	1.54	1.51			
5	1.48	1.59	1.64			
6	1.52	1.70	1.67			
7	1.37	1.74	1.77			
8	1.63	1.84	1.76			
9	1.60	1.93	1.82			
10	1.61	1.98	1.83			
11	1.61	1.91	1.78			
12	1.49	1.90	1.86			

Appendix B-2

The flowability data against the A_w of 18 NA, 21 NA, 22 NA and Kraft

Repeated measurement	22, NA, 0.80	22, NA, 0.77	22, NA, 0.74	22, NA, 0.71	22, NA, 0.68	22, NA, 0.66
1	1.64	1.90	1.76	1.93	1.35	1.18
2	1.77	1.95	1.76	1.90	1.38	1.14
3	1.88	2.08	1.78	1.87	1.37	1.11
4	2.10	2.09	1.68	1.93	1.30	1.09
5	2.16	2.14	1.66	1.86	1.37	1.10
6	2.24	2.18	1.73	1.81	1.30	1.10
7	2.34	2.17	1.66	1.82	1.33	1.07
8	2.37	2.16	1.70	1.77	1.23	1.12
9	2.44	2.19	1.68	1.74	1.28	1.12
10	2.46	2.16	1.55	1.69	1.28	1.12
11	2.48	2.19	1.60	1.56	1.19	1.12
12	2.49	2.12	1.60	1.52	1.17	1.12
Average	2.20	2.11	1.68	1.78	1.30	1.12
StdDev	0.28	0.09	0.07	0.13	0.07	0.03

Repeated measurement	21, NA, 0.79	21, NA, 0.76	21, NA, 0.74	21, NA, 0.72	21, NA, 0.69	21, NA, 0.67
1	1.36	1.52	1.47	1.43	1.20	1.10
2	1.42	1.54	1.39	1.41	1.14	1.11
3	1.53	1.55	1.37	1.32	1.09	1.12
4	1.66	1.56	1.36	1.37	1.10	1.12
5	1.82	1.48	1.38	1.31	1.14	1.07
6	1.84	1.60	1.27	1.30	1.11	1.10
7	1.92	1.53	1.29	1.28	1.13	1.09
8	1.95	1.53	1.35	1.29	1.11	1.20
9	2.00	1.51	1.27	1.31	1.12	1.11
10	2.06	1.49	1.32	1.31	1.10	1.07
11	2.06	1.49	1.31	1.25	1.12	1.15
12	2.13	1.49	1.27	1.23	1.11	1.06
Average	1.81	1.53	1.34	1.32	1.12	1.11
StdDev	0.25	0.03	0.06	0.06	0.03	0.04

Repeated measurement	18, NA, 0.81	18, NA, 0.77	18, NA, 0.74	18, NA, 0.71	18, NA, 0.69	18, NA, 0.67
1	0.95	0.98	1.16	1.12	0.92	0.93
2	0.97	1.13	1.26	1.12	0.96	0.91
3	0.98	1.23	1.27	1.07	0.94	0.88
4	1.00	1.32	1.31	1.11	0.94	0.93
5	1.06	1.37	1.36	1.14	0.92	0.92
6	1.13	1.41	1.33	1.11	0.93	0.92
7	1.23	1.50	1.36	1.15	0.88	0.92
8	1.36	1.52	1.40	1.13	0.95	0.91
9	1.43	1.58	1.34	1.15	0.96	0.97
10	1.42	1.56	1.35	1.15	0.93	0.94
11	1.54	1.65	1.38	1.13	0.94	0.95
12	1.57	1.64	1.32	1.16	0.92	0.95
Average	1.22	1.41	1.32	1.13	0.93	0.93
StdDev	0.22	0.20	0.06	0.02	0.02	0.02

Repeated measurement	Kraft, 0.81	Kraft, 0.79	Kraft, 0.74	Kraft, 0.71	Kraft, 0.69	Kraft, 0.68
1	0.86	0.74	0.70	0.77	0.81	0.73
2	0.83	0.79	0.74	0.75	0.82	0.76
3	0.87	0.85	0.79	0.79	0.79	0.72
4	0.89	0.81	0.76	0.80	0.84	0.78
5	0.89	0.83	0.78	0.78	0.82	0.77
6	0.90	0.82	0.81	0.82	0.84	0.82
7	0.89	0.83	0.81	0.77	0.84	0.82
8	0.90	0.86	0.81	0.79	0.79	0.83
9	0.96	0.82	0.80	0.79	0.78	0.80
10	0.92	0.80	0.78	0.82	0.83	0.83
11	0.92	0.82	0.81	0.81	0.82	0.87
12	0.91	0.84	0.81	0.83	0.81	0.85
Average	0.90	0.82	0.78	0.82	0.82	0.80
StdDev	0.03	0.03	0.03	0.02	0.02	0.04

Appendix B-3

The flowability data against the A_w of GVC 50017HM, 50017, 69430 & Kraft

50017HM

Sample	A1			A2		
A_w	0.83			0.82		
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.96	0.88	1.14	1.15	1.19	1.17
2	0.94	1.08	1.36	1.30	1.18	1.21
3	0.99	1.22	1.35	1.29	1.19	1.25
4	0.99	1.33	1.40	1.31	1.20	1.27
5	1.01	1.35	1.48	1.29	1.19	1.27
6	1.05	1.42	1.54	1.35	1.19	1.31
7	1.13	1.45	1.53	1.37	1.25	1.33
8	1.20	1.53	1.55	1.37	1.25	1.36
9	1.24	1.55	1.65	1.37	1.21	1.35
10	1.34	1.63	1.72	1.41	1.31	1.40
11	1.36	1.71	1.74	1.45	1.31	1.40
12	1.42	1.73	1.83	1.42	1.32	1.40
Average	1.14	1.41	1.52	1.34	1.23	1.31
StDev	0.16	0.24	0.19	0.08	0.05	0.07
Average		1.36			1.29	

Sample	A3			A4		
A_w	0.81			0.80		
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						

1	0.92	1.03	1.16	1.15	1.15	1.15
2	0.94	1.18	1.21	1.27	1.19	1.21
3	0.95	1.20	1.22	1.29	1.16	1.23
4	0.97	1.22	1.34	1.30	1.19	1.20
5	1.01	1.26	1.37	1.30	1.16	1.20
6	1.04	1.35	1.35	1.32	1.16	1.24
7	1.07	1.36	1.39	1.37	1.18	1.27
8	1.14	1.38	1.40	1.31	1.24	1.37
9	1.21	1.47	1.43	1.33	1.27	1.36
10	1.19	1.48	1.41	1.36	1.30	1.36
11	1.27	1.46	1.47	1.28	1.36	1.42
12	1.21	1.47	1.53	1.34	1.42	1.43
Average	1.08	1.32	1.36	1.30	1.23	1.29
StDev	0.12	0.14	0.11	0.05	0.08	0.09
Average		1.25			1.27	
Sample		A5			A6	
A _w		0.79			0.77	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.22	1.07	1.02	1.30	1.17	1.17
2	1.29	1.22	1.25	1.30	1.27	1.28
3	1.40	1.31	1.33	1.33	1.34	1.37
4	1.42	1.32	1.34	1.50	1.36	1.36
5	1.45	1.44	1.42	1.37	1.45	1.41
6	1.54	1.43	1.43	1.39	1.42	1.46
7	1.54	1.52	1.50	1.43	1.44	1.45
8	1.54	1.54	1.52	1.48	1.49	1.47
9	1.62	1.58	1.58	1.48	1.41	1.49
10	1.64	1.61	1.63	1.46	1.47	1.49
11	1.65	1.59	1.67	1.52	1.47	1.42
12	1.65	1.69	1.68	1.49	1.51	1.52
Average	1.50	1.44	1.45	1.42	1.40	1.41
StDev	0.14	0.17	0.18	0.08	0.10	0.10
Average		1.46			1.41	
Sample		A7			A8	
A _w		0.75			0.72	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.03	0.86	1.02	0.89	1.03	0.90
2	1.03	0.90	1.02	1.02	1.06	0.93
3	1.05	0.86	1.09	1.03	1.08	0.89
4	1.04	0.96	1.07	1.05	1.07	0.94
5	1.02	0.95	1.15	1.10	1.03	0.92
6	1.05	0.94	1.14	1.05	0.99	0.94
7	1.02	1.06	1.13	1.07	1.04	0.98
8	1.00	1.13	1.20	1.08	0.99	1.00
9	0.98	1.11	1.18	1.08	0.99	0.96
10	0.98	1.13	1.26	1.03	1.04	1.06

11	0.98	1.14	1.22	1.14	0.95	1.06
12	0.97	1.09	1.20	1.07	0.96	1.08
Average	1.01	1.01	1.14	1.05	1.02	0.97
StDev	0.03	0.11	0.07	0.06	0.04	0.06
Average		1.05			1.01	
Sample		A9			A10	
A _w		0.69			0.67	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.01	0.87	0.85	0.99	0.89	0.86
2	1.03	0.86	0.86	0.94	0.89	0.89
3	1.02	0.87	0.88	0.97	0.92	0.87
4	1.02	0.90	0.88	0.95	0.88	0.86
5	1.00	0.90	0.89	0.95	0.91	0.86
6	1.00	0.92	0.92	0.93	0.89	0.85
7	1.00	0.90	0.94	0.97	0.90	0.86
8	0.98	0.95	0.87	0.94	0.87	0.86
9	0.91	0.90	0.98	0.96	0.90	0.85
10	0.92	0.94	0.90	0.98	0.89	0.87
11	0.93	0.92	0.91	0.94	0.88	0.86
12	0.89	0.90	0.98	0.94	0.88	0.86
Average	0.98	0.90	0.91	0.96	0.89	0.86
StDev	0.05	0.03	0.04	0.02	0.01	0.01
Average		0.93			0.90	

50017

Sample		A1			A2	
A _w		0.83			0.82	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.04	1.17	1.21	0.99	1.13	1.31
2	1.26	1.32	1.29	0.99	1.33	1.42
3	1.31	1.26	1.37	1.04	1.41	1.48
4	1.36	1.39	1.35	1.07	1.48	1.56
5	1.48	1.41	1.46	1.17	1.52	1.61
6	1.50	1.50	1.55	1.28	1.68	1.76
7	1.55	1.59	1.68	1.39	1.85	1.88
8	1.65	1.59	1.67	1.46	1.79	1.95
9	1.68	1.59	1.72	1.71	1.91	2.05
10	1.68	1.77	1.75	1.64	1.99	2.06
11	1.83	1.70	1.81	1.89	1.97	2.09
12	1.74	1.84	1.83	1.98	2.09	2.11
Average	1.51	1.51	1.56	1.38	1.68	1.77
StDev	0.22	0.20	0.21	0.34	0.29	0.28
Average		1.53			1.61	

Sample		A3			A4		
A _w		0.81			0.80		
Sub-sample	1#	2#	3#	1#	2#	3#	
Repeated measurement							
1	1.19	1.25	1.28	1.21	1.23	1.40	
2	1.24	1.40	1.39	1.36	1.33	1.54	
3	1.45	1.36	1.42	1.37	1.43	1.57	
4	1.42	1.46	1.45	1.42	1.51	1.68	
5	1.50	1.54	1.54	1.56	1.69	1.73	
6	1.57	1.56	1.64	1.73	1.74	1.85	
7	1.68	1.68	1.64	1.83	1.83	1.97	
8	1.60	1.80	1.70	1.94	1.88	1.98	
9	1.79	1.77	1.85	2.02	1.97	2.08	
10	1.81	1.78	1.84	2.08	2.06	2.08	
11	1.84	1.85	1.84	2.12	2.14	2.10	
12	1.91	1.85	1.96	2.09	2.16	2.14	
Average	1.58	1.61	1.63	1.73	1.75	1.84	
StDev	0.22	0.20	0.21	0.32	0.30	0.24	
Average		1.61			1.77		
Sample		A5			A6		
A _w		0.79			0.77		
Sub-sample	1#	2#	3#	1#	2#	3#	
Repeated measurement							
1	1.44	1.40	1.33	1.40	1.27	1.30	
2	1.60	1.58	1.54	1.59	1.47	1.48	
3	1.66	1.74	1.71	1.71	1.59	1.43	
4	1.79	1.81	1.73	1.74	1.61	1.62	
5	1.87	1.86	1.84	1.79	1.68	1.67	
6	1.97	1.94	2.05	1.92	1.83	1.76	
7	2.03	2.00	2.11	1.88	1.83	1.79	
8	2.03	2.05	2.15	1.91	1.87	1.78	
9	2.16	2.10	2.15	1.99	1.91	1.84	
10	2.23	2.17	2.20	1.99	1.98	1.85	
11	2.17	2.27	2.24	2.12	2.04	1.97	
12	2.24	2.26	2.23	2.15	2.09	1.98	
Average	1.93	1.93	1.94	1.85	1.76	1.71	
StDev	0.25	0.26	0.29	0.21	0.23	0.20	
Average		1.93			1.77		
Sample		A7			A8		
A _w		0.74			0.71		
Sub-sample	1#	2#	3#	1#	2#	3#	
Repeated measurement							
1	1.20	1.20	1.10	1.13	1.04	1.12	
2	1.27	1.28	1.21	1.26	1.07	1.10	
3	1.52	1.38	1.24	1.25	1.08	1.16	
4	1.49	1.49	1.33	1.24	1.19	1.17	
5	1.50	1.54	1.42	1.29	1.18	1.22	
6	1.50	1.61	1.47	1.28	1.19	1.19	

7	1.55	1.61	1.52	1.25	1.24	1.22
8	1.70	1.60	1.52	1.30	1.27	1.27
9	1.65	1.70	1.55	1.28	1.26	1.25
10	1.65	1.70	1.55	1.31	1.28	1.28
11	1.56	1.72	1.59	1.29	1.31	1.25
12	1.61	1.69	1.63	1.35	1.29	1.25
Average	1.52	1.54	1.43	1.27	1.20	1.21
StDev	0.14	0.17	0.16	0.05	0.09	0.06
Average		1.50			1.23	

			A9			A10		
Sample			0.68			0.67		
A _w								
Sub-sample	1#	2#	3#	1#	2#	3#		
Repeated measurement								
1	1.08	1.02	1.06	1.23	1.10	1.08		
2	1.09	1.01	1.09	1.24	1.12	1.13		
3	1.09	1.00	1.12	1.23	1.13	1.15		
4	1.09	1.04	1.12	1.21	1.11	1.15		
5	1.17	1.02	1.16	1.24	1.12	1.16		
6	1.13	1.06	1.18	1.23	1.15	1.18		
7	1.18	1.13	1.19	1.24	1.12	1.20		
8	1.17	1.17	1.24	1.25	1.13	1.22		
9	1.21	1.15	1.23	1.23	1.09	1.22		
10	1.16	1.18	1.24	1.26	1.12	1.22		
11	1.14	1.19	1.25	1.24	1.17	1.26		
12	1.14	1.26	1.26	1.22	1.18	1.26		
Average	1.14	1.10	1.18	1.24	1.13	1.19		
StDev	0.04	0.08	0.07	0.01	0.03	0.05		
Average		1.14			1.18			

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Sample			A1			A2		
A _w			0.83			0.82		
Sub-sample	1#	2#	3#	1#	2#	3#		
Repeated measurement								
1	0.88	1.14	1.21	1.20	1.15	1.14		
2	0.96	1.19	1.24	1.20	1.24	1.18		
3	1.01	1.22	1.28	1.24	1.26	1.36		
4	1.09	1.27	1.33	1.33	1.33	1.40		
5	1.21	1.31	1.33	1.29	1.35	1.37		
6	1.24	1.32	1.37	1.34	1.36	1.38		
7	1.28	1.41	1.36	1.33	1.36	1.42		
8	1.41	1.42	1.42	1.38	1.39	1.40		
9	1.38	1.45	1.46	1.38	1.46	1.40		
10	1.41	1.48	1.42	1.36	1.50	1.42		
11	1.46	1.47	1.50	1.43	1.52	1.46		

12	1.46	1.52	1.52	1.47	1.52	1.47
Average	1.23	1.35	1.37	1.33	1.37	1.37
StDev	0.20	0.12	0.09	0.08	0.11	0.10
Average		1.32			1.36	
Sample		A3			A4	
A _w		0.81			0.80	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.24	1.21	1.25	1.28	1.21	1.18
2	1.28	1.36	1.35	1.26	1.27	1.23
3	1.32	1.38	1.33	1.35	1.30	1.24
4	1.33	1.39	1.35	1.34	1.25	1.29
5	1.39	1.42	1.34	1.36	1.32	1.27
6	1.35	1.43	1.38	1.36	1.33	1.33
7	1.42	1.45	1.48	1.34	1.34	1.37
8	1.44	1.50	1.44	1.45	1.36	1.38
9	1.47	1.54	1.49	1.47	1.37	1.42
10	1.48	1.57	1.52	1.41	1.36	1.38
11	1.47	1.53	1.53	1.43	1.41	1.45
12	1.51	1.57	1.56	1.46	1.44	1.41
Average	1.39	1.45	1.42	1.38	1.33	1.33
StDev	0.08	0.10	0.09	0.07	0.06	0.08
Average		1.42			1.35	
Sample		A5			A6	
A _w		0.79			0.77	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.24	1.21	1.15	1.21	1.17	1.18
2	1.31	1.17	1.20	1.30	1.20	1.17
3	1.28	1.26	1.20	1.28	1.22	1.20
4	1.36	1.28	1.18	1.30	1.28	1.23
5	1.32	1.30	1.28	1.33	1.26	1.30
6	1.39	1.33	1.25	1.34	1.33	1.30
7	1.38	1.35	1.28	1.41	1.32	1.33
8	1.41	1.37	1.32	1.40	1.36	1.31
9	1.44	1.39	1.30	1.38	1.32	1.37
10	1.46	1.46	1.37	1.37	1.39	1.33
11	1.46	1.38	1.43	1.38	1.40	1.38
12	1.44	1.43	1.37	1.40	1.41	1.37
Average	1.37	1.33	1.28	1.34	1.31	1.29
StDev	0.07	0.08	0.08	0.06	0.08	0.07
Average		1.33			1.31	
Sample		A7			A8	
A _w		0.74			0.73	

Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.16	1.08	1.13	1.09	1.11	1.06
2	1.17	1.13	1.18	1.15	1.14	1.04
3	1.22	1.22	1.24	1.17	1.17	1.10
4	1.21	1.19	1.25	1.20	1.15	1.17
5	1.30	1.27	1.27	1.18	1.22	1.21
6	1.24	1.28	1.29	1.16	1.22	1.24
7	1.30	1.30	1.28	1.24	1.24	1.19
8	1.32	1.31	1.37	1.18	1.20	1.17
9	1.29	1.35	1.36	1.18	1.25	1.21
10	1.30	1.35	1.38	1.20	1.21	1.21
11	1.33	1.30	1.39	1.21	1.25	1.26
12	1.31	1.37	1.42	1.24	1.23	1.24
Average	1.26	1.26	1.30	1.18	1.20	1.18
StDev	0.06	0.09	0.09	0.04	0.04	0.07
Average		1.27			1.19	
Sample		A9			A10	
A _w		0.69			0.67	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.94	0.90	0.82	1.01	0.98	0.97
2	0.98	0.95	0.92	1.00	0.97	0.98
3	0.99	0.97	0.90	1.05	1.00	0.99
4	0.98	1.01	0.94	1.07	0.97	0.98
5	0.97	0.99	0.95	1.03	1.00	0.99
6	1.01	1.01	0.94	1.08	1.00	0.99
7	1.00	1.07	0.97	1.10	1.00	1.01
8	1.01	1.04	0.95	1.10	1.01	1.00
9	1.05	1.02	0.95	1.04	1.01	0.99
10	1.04	1.03	1.00	1.08	1.04	0.99
11	1.01	1.01	0.97	1.10	1.01	1.01
12	0.99	1.02	0.97	1.07	1.02	1.02
Average	1.00	1.00	0.94	1.06	1.00	0.99
StDev	0.03	0.04	0.04	0.03	0.02	0.01
Average		0.98			1.02	

Kraft

Sample		A1			A2	
A _w		0.83			0.82	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.84	0.91	0.90	0.92	0.98	0.98
2	0.86	1.08	1.08	0.95	1.03	1.07
3	0.84	1.04	1.03	0.99	1.00	1.02
4	0.85	1.06	1.02	0.98	1.00	0.96

5	0.96	1.15	0.95	0.94	0.95	0.87
6	1.09	1.15	0.98	0.92	0.83	0.89
7	1.09	1.12	1.01	0.94	0.98	0.94
8	1.13	1.16	0.96	0.90	0.92	0.84
9	1.17	1.16	0.90	0.85	0.99	0.91
10	1.19	1.19	0.89	0.91	1.00	0.89
11	1.17	1.14	0.96	0.99	1.00	0.94
12	1.20	1.13	0.93	0.91	0.98	0.94
Average	1.03	1.11	0.97	0.93	0.97	0.94
StDev	0.14	0.07	0.06	0.04	0.05	0.06
Average		1.04			0.95	
Sample		A3			A4	
A _w		0.81			0.80	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.95	0.99	1.06	0.93	0.92	0.91
2	1.02	1.01	1.06	0.99	0.98	0.96
3	0.98	1.00	0.95	0.99	1.12	1.00
4	1.02	0.95	0.99	0.96	1.10	1.05
5	0.94	1.03	0.97	0.98	1.08	1.01
6	0.96	0.92	0.91	0.96	1.09	0.96
7	0.89	0.86	0.98	0.93	1.14	0.98
8	0.96	0.92	0.95	1.04	1.11	0.98
9	0.88	0.96	0.96	0.95	1.11	1.00
10	0.96	0.92	1.03	1.03	1.12	0.95
11	0.95	0.93	1.06	1.01	1.17	0.95
12	1.03	0.95	1.08	1.06	1.13	0.88
Average	0.96	0.95	1.00	0.99	1.09	0.97
StDev	0.05	0.05	0.05	0.04	0.07	0.04
Average		0.97			1.01	
Sample		A5			A6	
A _w		0.79			0.77	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.81	0.73	0.91	0.80	0.79	0.80
2	0.84	0.92	0.98	0.82	0.83	0.85
3	0.99	0.87	0.98	0.86	0.84	0.82
4	1.01	0.93	0.94	0.87	0.84	0.86
5	0.92	0.98	0.92	0.83	0.83	0.85
6	0.97	1.03	0.95	0.80	0.88	0.85
7	0.92	0.94	0.92	0.83	0.83	0.89
8	0.91	0.99	0.93	0.84	0.85	0.93
9	0.92	1.01	0.99	0.85	0.89	0.95
10	0.95	1.00	0.99	0.87	0.87	0.96
11	0.94	1.04	0.96	0.92	0.90	0.94
12	0.98	1.05	1.00	0.94	0.93	0.97
Average	0.93	0.96	0.96	0.85	0.86	0.89
StDev	0.06	0.09	0.03	0.04	0.04	0.06

Average		0.95			0.87	
Sample		A7			A8	
A _w		0.75			0.72	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.87	0.90	0.84	1.09	1.02	0.92
2	0.86	0.87	0.83	1.09	1.05	0.91
3	0.86	0.88	0.81	1.11	1.01	0.94
4	0.85	0.86	0.88	1.13	1.05	1.02
5	0.87	0.86	0.92	1.14	0.99	0.95
6	0.92	0.91	0.88	1.21	1.00	0.91
7	0.89	0.90	0.94	1.11	1.00	0.95
8	0.94	0.91	0.97	1.16	1.04	0.93
9	0.94	0.94	0.94	1.10	0.96	0.93
10	0.92	0.93	0.93	1.11	0.95	0.95
11	0.93	0.99	0.96	1.18	0.97	0.93
12	0.95	0.95	0.99	1.11	0.93	0.96
Average	0.90	0.91	0.91	1.13	1.00	0.94
StDev	0.04	0.04	0.06	0.04	0.04	0.03
Average		0.91			1.02	
Sample		A9			A10	
A _w		0.70			0.67	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.86	0.80	0.79	0.79	0.77	0.75
2	0.84	0.81	0.75	0.80	0.78	0.78
3	0.82	0.77	0.75	0.79	0.77	0.78
4	0.84	0.81	0.78	0.75	0.80	0.75
5	0.81	0.80	0.75	0.76	0.79	0.74
6	0.77	0.81	0.80	0.76	0.78	0.79
7	0.80	0.81	0.79	0.76	0.78	0.75
8	0.81	0.82	0.79	0.75	0.80	0.77
9	0.81	0.80	0.80	0.77	0.80	0.77
10	0.79	0.81	0.79	0.80	0.80	0.80
11	0.81	0.79	0.77	0.78	0.80	0.78
12	0.81	0.79	0.80	0.80	0.81	0.75
Average	0.81	0.80	0.78	0.78	0.79	0.77
StDev	0.02	0.01	0.02	0.02	0.01	0.02
Average		0.80			0.78	

Appendix B-4

The flowability data against the A_w of the commercial Grated and Dried Parmesan cheese samples: GVC 50017 and 50064

50017

Sample		A1			A2		
A_w		0.77			0.76		
Sub-sample	1#	2#	3#	1#	2#	3#	
repeated measurement							
1	1.25	1.24		1.17	1.28		
2	1.32	1.32		1.14	1.25		
3	1.25	1.33		1.22	1.30		
4	1.38	1.39		1.30	1.34		
5	1.41	1.36		1.37	1.39		
6	1.37	1.36		1.34	1.39		
Average	1.33	1.33		1.26	1.32		
Average		1.33			1.29		

Sample		A3			A4		
A_w		0.74			0.74		
Sub-sample	1#	2#	3#	1#	2#	3#	
Repeated measurement							
1	0.91	0.90	1.14	0.99	1.22	1.17	
2	0.94	1.02	1.19	1.00	1.32	1.17	
3	0.98	1.08	1.20	1.04	1.24	1.26	
4	1.03	1.08	1.21	0.99	1.35	1.28	
5	1.10	1.10	1.26	1.05	1.31	1.36	
6	1.09	1.13	1.28	1.09	1.28	1.35	
Average	1.01	1.05	1.21	1.03	1.29	1.39	
Average		1.09			1.21		

Sample		A5			A6		
A_w		0.73			0.72		
Sub-sample	1#	2#	3#	1#	2#	3#	
Repeated measurement							
1	0.91	1.33	1.03	1.15	1.14	1.07	
2	1.02	1.34	1.13	1.18	1.22	1.12	
3	1.06	1.38	1.26	1.25	1.18	1.14	
4	1.01	1.36	1.20	1.16	1.22	1.18	
5	1.05	1.34	1.22	1.29	1.20	1.24	
6	1.08	1.36	1.32	1.27	1.26	1.17	
Average	1.02	1.35	1.19	1.22	1.20	1.15	
Average		1.19			1.19		

Sample		A7			A8		
A _w		0.72			0.71		
Sub-sample	1#	2#	3#	1#	2#	3#	
Repeated measurement							
1	0.99	1.02	1.10	0.95	0.97	0.93	
2	0.97	1.10	1.12	0.99	0.94	0.94	
3	1.04	1.17	1.16	1.03	1.01	0.98	
4	1.14	1.15	1.23	1.01	1.01	1.04	
5	1.11	1.19	1.24	1.01	1.07	1.06	
6	1.16	1.15	1.24	1.01	1.05	1.06	
Average	1.07	1.13	1.18	1.00	1.01	1.00	
Average		1.13			1.00		
Sample		A9					
A _w		0.70					
Sub-sample	1#	2#	3#				
Repeated measurement							
1	1.06	1.06	1.02				
2	1.11	1.12	1.06				
3	1.11	1.12	1.04				
4	1.12	1.13	1.09				
5	1.09	1.16	1.10				
6	1.11	1.11	1.10				
Average	1.10	1.12	1.07				
Average		1.10					
50064							
Sample		A1			A2		
A _w		0.77			0.76		
Sub-sample	1#	2#	3#	1#	2#	3#	
Repeated measurement							
1	1.12	1.16	1.15	0.95	1.04		
2	1.26	1.24	1.28	1.08	1.16		
3	1.27	1.34	1.35	1.18	1.18		
4	1.30	1.36	1.31	1.24	1.23		
5	1.38	1.38	1.36	1.20	1.18		
6	1.38	1.33	1.41	1.23	1.28		
Average	1.29	1.34	1.32	1.15	1.20		
Average		1.32			1.17		
Sample		A3			A4		
A _w		0.77			0.75		
Sub-sample	1#	2#	3#	1#	2#	3#	
Repeated measurement							
1	1.07	1.07	1.03	1.06	0.96	0.96	
2	1.12	1.18	1.20	1.18	0.97	1.00	
3	1.23	1.28	1.24	1.18	1.02	1.08	

4	1.23	1.27	1.32	1.23	1.09	1.11
5	1.32	1.29	1.34	1.15	1.15	1.08
6	1.37	1.37	1.30	1.23	1.14	1.19
Average	1.23	1.24	1.24	1.17	1.06	1.07
Average		1.24			1.10	
Sample		A5			A6	
A _w		0.76			0.75	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.03	0.93	1.10	0.89	0.94	0.91
2	1.10	1.09	1.23	0.90	0.99	1.00
3	1.20	1.17	1.31	1.00	1.09	1.13
4	1.25	1.22	1.30	0.96	1.13	1.11
5	1.26	1.27	1.34	1.01	1.11	1.16
6	1.21	1.30	1.39	1.04	1.19	1.17
Average	1.18	1.16	1.28	0.97	1.07	1.08
Average		1.21			1.04	
Sample		A7			A8	
A _w		0.74			0.74	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.89	0.91		1.04	0.98	0.95
2	0.90	0.94		1.06	1.00	1.01
3	0.96	0.95		1.09	1.09	1.03
4	0.96	0.98		1.08	1.03	1.05
5	0.96	1.04		1.18	1.06	1.09
6	1.02	1.08		1.14	1.11	1.08
Average	0.95	0.98		1.10	1.05	1.04
Average		0.97			1.06	

Appendix B-5

The relationship between flowability and particle size distribution for 50017HM, 50017, 69430 and Kraft

50017HM						
Sample	M1 (100% A)			M2 (85% A+15% B)		
A _w	0.74			0.74		
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.81	1.72	1.63	1.66	1.60	1.59
2	1.90	1.83	1.65	1.64	1.64	1.59
3	1.90	1.86	1.72	1.69	1.69	1.69
4	1.87	1.86	1.75	1.74	1.69	1.70

5	1.92	1.87	1.77	1.77	1.68	1.73
6	1.95	1.81	1.80	1.83	1.80	1.77
7	1.99	1.86	1.90	1.84	1.83	1.87
8	2.04	1.92	1.97	1.83	1.82	1.86
9	2.02	1.91	1.92	1.76	1.86	1.92
10	2.03	1.95	1.98	1.78	1.88	1.98
11	2.04	1.90	2.03	1.84	1.88	1.98
12	2.09	1.96	1.98	1.92	1.89	1.99
Average	1.96	1.87	1.84	1.78	1.77	1.81
StDev	0.08	0.06	0.13	0.08	0.10	0.14
Average		1.89			1.78	

Sample		M3 (80%A+20%B)			M4 (75%A+25%B)		
A _w		0.74			0.74		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement							
1	1.56	1.50	1.51	1.40	1.26	1.35	
2	1.58	1.55	1.58	1.46	1.27	1.44	
3	1.58	1.63	1.61	1.48	1.32	1.48	
4	1.61	1.63	1.67	1.48	1.32	1.52	
5	1.67	1.68	1.67	1.50	1.37	1.71	
6	1.70	1.71	1.74	1.49	1.38	1.71	
7	1.77	1.70	1.76	1.52	1.45	1.72	
8	1.79	1.76	1.78	1.47	1.52	1.77	
9	1.82	1.80	1.79	1.50	1.45	1.72	
10	1.88	1.82	1.83	1.49	1.57	1.73	
11	1.80	1.81	1.86	1.51	1.57	1.78	
12	1.84	1.82	1.84	1.50	1.56	1.78	
Average	1.72	1.70	1.72	1.48	1.42	1.64	
StDev	0.11	0.10	0.11	0.03	0.11	0.14	
Average		1.71			1.52		

Sample		M5 (70%A+30%B)			M6 (65%A+35%B)		
A _w		0.74			0.74		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement							
1	1.26	1.18	1.08	1.06	1.14	1.01	
2	1.34	1.20	1.11	1.09	1.13	1.03	
3	1.32	1.21	1.16	1.04	1.18	1.04	
4	1.33	1.21	1.15	1.16	1.15	1.06	
5	1.32	1.21	1.24	1.17	1.22	1.11	
6	1.33	1.23	1.30	1.12	1.22	1.14	
7	1.35	1.21	1.28	1.23	1.21	1.11	
8	1.32	1.21	1.27	1.23	1.21	1.11	
9	1.32	1.25	1.26	1.21	1.18	1.13	
10	1.38	1.26	1.31	1.24	1.25	1.19	
11	1.36	1.28	1.30	1.25	1.20	1.13	
12	1.35	1.30	1.34	1.28	1.25	1.13	

Average	1.33	1.23	1.23	1.17	1.20	1.10
StDev	0.03	0.03	0.08	0.08	0.04	0.05
Average		1.26			1.16	

Sample		M7 (60%A+40%B)			M8 (55%A+45%B)		
A _w		0.74			0.74		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement							
1	1.16	1.00	0.96	1.02	0.99	0.93	
2	1.20	1.03	0.98	1.05	0.98	0.94	
3	1.27	1.05	1.00	1.06	0.97	0.96	
4	1.26	1.07	1.02	1.08	1.07	1.04	
5	1.27	1.08	1.08	1.04	1.05	1.04	
6	1.28	1.09	1.07	1.16	1.03	1.07	
7	1.30	1.09	1.11	1.12	1.11	1.06	
8	1.28	1.12	1.10	1.17	1.07	1.07	
9	1.30	1.17	1.07	1.15	1.09	1.10	
10	1.28	1.20	1.08	1.14	1.10	1.08	
11	1.31	1.17	1.09	1.20	1.08	1.07	
12	1.36	1.20	1.08	1.19	1.14	1.11	
Average	1.27	1.11	1.05	1.12	1.06	1.04	
StDev	0.05	0.06	0.05	0.06	0.05	0.06	
Average		1.14			1.07		

Sample		M9 (100%B)		
A _w		0.74		
Sub-sample		1#	2#	3#
Repeated measurement				
1	0.79	0.85	0.87	
2	0.80	0.78	0.84	
3	0.81	0.79	0.89	
4	0.84	0.82	0.84	
5	0.83	0.91	0.90	
6	0.85	0.92	0.85	
7	0.84	0.86	1.03	
8	0.93	0.85	1.02	
9	0.95	0.93	1.11	
10	0.92	0.84	0.88	
11	0.83	0.87	0.87	
12	1.06	0.86	1.00	
Average	0.87	0.86	0.93	
StDev	0.08	0.05	0.09	
Average		0.88		

50017

Sample		M1 (100%A)	M2 (85%A+15%B)
A _w		0.73	0.73

Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.83	1.64	1.62	1.68	1.61	1.68
2	1.71	1.67	1.72	1.66	1.69	1.70
3	1.69	1.69	1.67	1.70	1.71	1.74
4	1.77	1.79	1.71	1.69	1.76	1.72
5	1.77	1.83	1.82	1.85	1.76	1.82
6	1.80	1.81	1.83	1.79	1.79	1.88
7	1.84	1.87	1.87	1.81	1.85	1.88
8	1.86	1.90	1.88	1.88	1.84	1.88
9	1.88	1.94	1.86	1.91	1.89	1.89
10	1.90	1.92	1.92	1.93	1.92	1.91
11	1.92	1.97	2.00	1.92	1.97	1.94
12	1.79	1.95	2.06	1.97	2.01	1.98
Average	1.81	1.83	1.83	1.82	1.82	1.84
StDev	0.07	0.11	0.13	0.11	0.11	0.10
Average		1.83			1.82	
Sample		M3 (80%A+20%B)			M4 (75%A+25%B)	
A _w		0.73			0.743	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.64	1.51	1.51	1.47	1.46	1.42
2	1.65	1.58	1.56	1.44	1.50	1.51
3	1.70	1.61	1.59	1.52	1.54	1.52
4	1.71	1.66	1.64	1.53	1.53	1.55
5	1.72	1.72	1.65	1.61	1.56	1.61
6	1.75	1.78	1.73	1.63	1.61	1.61
7	1.75	1.79	1.71	1.62	1.61	1.63
8	1.77	1.76	1.74	1.66	1.58	1.67
9	1.70	1.87	1.73	1.73	1.65	1.70
10	1.76	1.81	1.81	1.73	1.67	1.74
11	1.73	1.84	1.78	1.75	1.71	1.68
12	1.72	1.81	1.80	1.71	1.67	1.74
Average	1.72	1.73	1.69	1.62	1.59	1.62
StDev	0.04	0.11	0.09	0.10	0.07	0.10
Average		1.71			1.61	
Sample		M5 (70%A+30%B)			M6 (65%A+35%B)	
A _w		0.73			0.73	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.61	1.51	1.42	1.24	1.25	1.24
2	1.57	1.46	1.48	1.31	1.26	1.29
3	1.58	1.55	1.52	1.31	1.35	1.36
4	1.66	1.58	1.59	1.39	1.33	1.36
5	1.68	1.63	1.62	1.37	1.38	1.44
6	1.67	1.64	1.65	1.42	1.41	1.42

7	1.75	1.67	1.63	1.32	1.48	1.52
8	1.73	1.65	1.66	1.45	1.48	1.50
9	1.76	1.66	1.71	1.47	1.49	1.57
10	1.77	1.69	1.72	1.47	1.50	1.53
11	1.79	1.66	1.75	1.50	1.52	1.60
12	1.72	1.66	1.79	1.50	1.55	1.59
Average	1.69	1.61	1.63	1.40	1.42	1.45
StDev	0.07	0.07	0.11	0.08	0.10	0.11
Average		1.64			1.42	

Sample	M7 (60%A+40%B)			M8 (55%A+45%B)		
A _w	0.72			0.72		
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.28	1.22	1.23	1.32	1.24	1.16
2	1.33	1.30	1.20	1.36	1.27	1.17
3	1.34	1.30	1.26	1.41	1.26	1.22
4	1.38	1.34	1.29	1.42	1.30	1.19
5	1.41	1.37	1.32	1.42	1.30	1.26
6	1.42	1.36	1.32	1.42	1.27	1.25
7	1.47	1.37	1.35	1.47	1.30	1.24
8	1.50	1.41	1.35	1.47	1.34	1.28
9	1.47	1.43	1.34	1.43	1.38	1.30
10	1.48	1.41	1.35	1.48	1.37	1.33
11	1.51	1.40	1.42	1.49	1.39	1.33
12	1.47	1.43	1.41	1.52	1.37	1.34
Average	1.42	1.36	1.32	1.43	1.32	1.26
StDev	0.07	0.06	0.06	0.05	0.05	0.06
Average		1.37			1.34	

Sample	M9 (100%B)		
A _w	0.72		
Sub-sample	1#	2#	3#
Repeated measurement			
1	0.73	0.76	0.68
2	0.77	0.80	0.71
3	0.77	0.82	0.69
4	0.80	0.86	0.71
5	0.82	0.90	0.71
6	0.86	0.96	0.69
7	0.88	0.89	0.83
8	0.95	1.02	0.73
9	0.90	1.08	0.80
10	0.91	1.16	0.88
11	1.00	1.08	0.86
12	0.88	1.03	0.82
Average	0.86	0.95	0.76

StDev	0.08	0.12	0.07
Average		0.85	

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Sample		M1 (100% A)			M2 (85% A+15% B)		
A _w		0.74			0.74		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement	1	1.30	1.23	1.23	1.11	1.05	1.05
	2	1.29	1.20	1.26	1.08	1.06	1.13
	3	1.38	1.34	1.27	1.13	1.14	1.11
	4	1.32	1.31	1.28	1.08	1.19	1.22
	5	1.40	1.31	1.32	1.08	1.18	1.19
	6	1.39	1.38	1.33	1.11	1.21	1.19
	7	1.32	1.27	1.39	1.16	1.22	1.20
	8	1.42	1.37	1.34	1.15	1.28	1.30
	9	1.41	1.39	1.42	1.15	1.20	1.27
	10	1.46	1.39	1.41	1.18	1.28	1.25
	11	1.40	1.34	1.39	1.15	1.28	1.28
	12	1.43	1.35	1.46	1.19	1.29	1.98
	Average	1.38	1.32	1.34	1.13	1.20	1.26
StDev		0.05	0.06	0.07	0.04	0.08	0.23
Average			1.35			1.20	

Sample		M3 (80% A+20% B)			M4 (75% A+25% B)		
A _w		0.74			0.74		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement	1	1.24	1.07	1.13	1.12	1.22	1.15
	2	1.29	1.16	1.12	1.22	1.19	1.11
	3	1.25	1.19	1.23	1.23	1.29	1.20
	4	1.28	1.20	1.18	1.24	1.26	1.20
	5	1.30	1.20	1.19	1.28	1.30	1.21
	6	1.27	1.31	1.25	1.34	1.30	1.27
	7	1.25	1.29	1.23	1.37	1.29	1.30
	8	1.25	1.28	1.26	1.29	1.37	1.32
	9	1.30	1.25	1.29	1.35	1.36	1.34
	10	1.27	1.37	1.30	1.36	1.35	1.33
	11	1.32	1.34	1.30	1.33	1.33	1.36
	12	1.28	1.36	1.36	1.34	1.37	1.36
	Average	1.28	1.25	1.24	1.29	1.30	1.26
StDev		0.02	0.09	0.07	0.07	0.06	0.08
Average			1.25			1.28	

Sample		M5 (70% A+30% B)			M6 (65% A+35% B)		
A _w		0.74			0.74		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement							

1	1.01	0.94	0.96	1.07	0.99	0.97
2	1.01	0.95	0.99	1.12	1.00	0.97
3	1.02	1.03	1.07	1.11	1.02	1.00
4	1.05	1.06	1.09	1.11	1.08	1.03
5	1.04	1.04	1.17	1.14	1.10	1.09
6	1.03	1.07	1.20	1.11	1.08	0.97
7	1.10	1.15	1.23	1.12	1.11	1.05
8	1.11	1.09	1.28	1.14	1.12	1.12
9	1.12	1.11	1.25	1.13	1.12	1.03
10	1.10	1.11	1.18	1.17	1.17	1.07
11	1.15	1.17	1.20	1.15	1.18	1.10
12	1.12	1.13	1.21	1.15	1.13	1.15
Average	1.07	1.07	1.15	1.13	1.09	1.05
StDev	0.05	0.07	0.10	0.02	0.06	0.06
Average		1.10			1.09	

Sample	M7 (60%A+40%B)			M8 (55%A+45%B)		
A _w	0.74			0.74		
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.99	0.91	0.91	0.90	0.84	0.88
2	1.02	0.98	0.89	0.90	0.87	0.90
3	1.02	1.00	0.99	0.96	0.90	0.95
4	1.05	1.02	1.02	0.99	0.96	0.93
5	1.03	1.08	1.02	1.01	0.96	0.95
6	1.10	1.08	1.08	0.96	1.00	1.02
7	1.05	1.09	1.09	0.97	0.97	1.02
8	1.07	1.19	1.15	1.00	1.02	0.97
9	1.13	1.20	1.13	1.04	0.96	1.05
10	1.10	1.11	1.13	0.96	0.95	1.07
11	1.11	1.11	1.18	1.06	0.97	1.04
12	1.15	1.15	1.16	0.99	0.98	0.98
Average	1.07	1.08	1.06	0.98	0.95	0.98
StDev	0.05	0.08	0.09	0.05	0.05	0.06
Average		1.07			0.97	

Sample	M9 (100%B)		
A _w	0.73		
Sub-sample	1#	2#	3#
Repeated measurement			
1	0.70	0.79	0.72
2	0.78	0.80	0.79
3	0.77	0.88	0.77
4	0.88	0.86	0.86
5	0.86	0.87	0.86
6	0.81	0.89	0.85
7	0.87	0.86	0.90

8	0.85	0.88	0.85
9	0.87	0.90	0.83
10	0.90	0.85	0.86
11	0.83	0.83	0.88
12	0.90	0.85	0.88
Average	0.84	0.86	0.84
StDev	0.06	0.03	0.05
Average		0.85	

Kraft

Sample		M1 (100%A)			M2 (90%A+10%B)		
A _w		0.78			0.78		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement							
1	1.17	1.17	1.09	1.09	1.07	1.00	
2	1.29	1.16	1.26	1.19	1.12	1.09	
3	1.29	1.17	1.37	1.17	1.12	1.12	
4	1.31	1.11	1.45	1.22	1.12	1.21	
5	1.32	1.35	1.50	1.21	1.12	1.28	
6	1.26	1.42	1.52	1.22	1.21	1.24	
7	1.23	1.47	1.44	1.09	1.16	1.22	
8	1.25	1.47	1.52	1.17	1.27	1.23	
9	1.26	1.59	1.53	1.15	1.15	1.33	
10	1.30	1.58	1.50	1.17	1.26	1.33	
11	1.38	1.63	1.60	1.17	1.25	1.35	
12	1.33	1.66	1.56	1.18	1.25	1.40	
Average	1.28	1.40	1.45	1.17	1.18	1.23	
StDev	0.05	0.19	0.14	0.04	0.07	0.11	
Average		1.38			1.19		

Sample		M3 (85%A+15%B)			M4 (80%A+20%B)		
A _w		0.78			0.79		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement							
1	1.01	0.99	1.03	1.11	1.11	1.07	
2	1.03	0.96	1.02	1.21	1.15	1.10	
3	1.04	0.97	1.10	1.20	1.14	1.13	
4	1.02	1.03	1.07	1.23	1.17	1.16	
5	0.97	1.01	1.10	1.24	1.22	1.18	
6	1.01	0.98	1.04	1.25	1.16	1.20	
7	1.02	1.00	1.09	1.29	1.22	1.23	
8	1.04	1.08	1.12	1.30	1.27	1.26	
9	1.06	1.12	1.07	1.34	1.28	1.28	
10	1.07	1.08	1.08	1.33	1.28	1.25	
11	1.12	1.08	1.11	1.31	1.28	1.33	
12	1.09	1.12	1.08	1.36	1.35	1.36	
Average	1.04	1.04	1.08	1.26	1.22	1.21	
StDev	0.04	0.06	0.03	0.07	0.07	0.09	
Average		1.05			1.23		

Sample		M5 (75% A+25% B)			M6 (70% A+30% B)		
A _w		0.78			0.78		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement							
1		1.00	1.01	0.98	0.91	0.92	0.93
2		1.04	1.05	1.01	0.88	0.95	0.95
3		1.03	1.05	1.06	0.97	1.03	0.96
4		1.05	1.07	1.04	0.97	0.98	1.04
5		1.06	1.12	1.05	0.93	1.00	1.08
6		1.10	1.10	1.06	0.95	1.10	1.08
7		1.11	1.18	1.11	1.00	1.05	1.03
8		1.15	1.16	1.15	1.04	1.10	1.08
9		1.16	1.18	1.13	1.01	1.13	1.08
10		1.23	1.18	1.17	1.04	1.18	1.11
11		1.25	1.21	1.15	1.08	1.18	1.20
12		1.26	1.27	1.15	1.10	1.18	1.19
Average		1.12	1.13	1.09	0.99	1.07	1.06
StDev		0.09	0.07	0.06	0.06	0.09	0.08
Average			1.11			1.04	

Sample		M7 (65% A+35% B)			M8 (60% A+40% B)		
A _w		0.78			0.78		
Sub-sample		1#	2#	3#	1#	2#	3#
Repeated measurement							
1		1.02	0.97	0.97	0.86	0.83	0.85
2		1.04	1.02	0.98	0.86	0.90	0.82
3		1.08	1.02	0.98	0.90	0.91	0.88
4		1.12	1.10	1.02	0.92	0.91	0.83
5		1.15	1.05	1.02	0.93	0.95	0.90
6		1.14	1.10	1.03	0.94	0.91	0.86
7		1.13	1.06	1.02	0.97	0.93	0.91
8		1.16	1.13	1.07	0.94	0.93	0.92
9		1.16	1.10	1.07	0.97	0.98	0.90
10		1.20	1.12	1.10	1.00	0.96	0.90
11		1.22	1.15	1.07	1.01	0.97	0.95
12		1.19	1.15	1.08	1.02	1.04	1.01
Average		1.13	1.08	1.03	0.94	0.94	0.89
StDev		0.06	0.05	0.04	0.05	0.05	0.05
Average			1.08			0.92	

Sample		M9 (100% B)		
A _w		0.78		
Sub-sample		1#	2#	3#
Repeated measurement				
1		0.53	0.58	0.53
2		0.57	0.70	0.62
3		0.57	0.75	0.68

4	0.63	0.77	0.67
5	0.72	0.76	0.75
6	0.57	0.66	0.74
7	0.67	0.70	0.64
8	0.69	0.72	0.73
9	0.69	0.67	0.72
10	0.68	0.71	0.68
11	0.71	0.68	0.68
12	0.67	0.69	0.77
Average	0.64	0.70	0.68
StDev	0.06	0.05	0.06
Average		0.68	

Appendix B-6

The flowability data against free fat contents of 50017HM

Sample		A1			A2	
A _w		0.75			0.75	
Free fat		0.70%			16.80%	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.52	1.51	1.47	2.26	2.02	2.07
2	1.49	1.51	1.52	2.05	2.05	2.19
3	1.50	1.53	1.48	2.10	2.03	2.23
4	1.55	1.51	1.55	2.17	2.27	2.26
5	1.56	1.56	1.49	2.09	2.29	2.33
6	1.59	1.54	1.52	2.22	2.27	2.41
7	1.57	1.58	1.54	2.28	2.44	2.46
8	1.62	1.62	1.56	2.27	2.36	2.41
9	1.60	1.60	1.52	2.23	2.38	2.41
10	1.66	1.58	1.57	2.20	2.52	2.56
11	1.62	1.61	1.59	2.32	2.45	2.52
12	1.64	1.62	1.62	2.28	2.48	2.52
Average	1.58	1.56	1.54	2.21	2.30	2.36
StDev	0.05	0.04	0.04	0.08	0.17	0.14
Average		1.56			2.29	
Sample		A3			A4	
A _w		0.75			0.75	
Free fat		18.40%			22.50%	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.65	1.55	1.62	1.53	1.52	1.48
2	1.71	1.65	1.69	1.63	1.64	1.68
3	1.73	1.80	1.76	1.7	1.82	1.89
4	1.85	2.02	2.00	1.88	1.97	1.95

5	1.92	1.98	2.06	1.94	1.98	1.98
6	1.92	2.16	2.09	1.89	2.09	2.11
7	2.00	2.27	2.15	1.97	2.15	2.20
8	2.07	2.18	2.18	2.05	2.14	2.18
9	1.96	2.21	2.29	2.10	2.18	2.31
10	2.02	2.29	2.37	2.23	2.22	2.33
11	2.24	2.24	2.32	2.13	2.26	2.29
12	2.02	2.23	2.42	2.09	2.23	2.30
Average	1.92	2.05	2.08	1.93	2.02	2.06
StDev	0.16	0.24	0.26	0.21	0.23	0.26
Average		2.02			2.00	
Sample		A5			A6	
A _w		0.74			0.75	
Free fat		22.90%			24.00%	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	1.17	1.20	1.20	0.93	1.10	1.15
2	1.17	1.30	1.32	1.06	1.27	1.26
3	1.29	1.30	1.35	1.08	1.29	1.36
4	1.21	1.44	1.50	1.12	1.31	1.36
5	1.27	1.51	1.39	1.14	1.42	1.44
6	1.31	1.43	1.52	1.27	1.40	1.41
7	1.32	1.55	1.55	1.30	1.39	1.44
8	1.30	1.63	1.57	1.36	1.48	1.50
9	1.42	1.57	1.60	1.31	1.45	1.49
10	1.39	1.52	1.61	1.36	1.45	1.62
11	1.42	1.57	1.55	1.35	1.46	1.54
12	1.45	1.64	1.56	1.38	1.51	1.52
Average	1.31	1.47	1.48	1.22	1.38	1.42
StDev	0.09	0.13	0.12	0.14	0.11	0.13
Average		1.42			1.34	
Sample		A7			A8	
A _w		0.75			0.75	
Free fat		24.9			26.3	
Sub-sample	1#	2#	3#	1#	2#	3#
Repeated measurement						
1	0.76	0.77	0.82	0.49	0.52	0.37
2	0.85	0.85	0.91	0.48	0.52	0.45
3	0.84	0.86	0.93	0.52	0.57	0.44
4	0.87	0.88	0.92	0.53	0.60	0.51
5	0.83	0.90	0.90	0.52	0.57	0.54
6	0.87	0.97	0.97	0.55	0.61	0.54
7	0.85	0.91	1.04	0.56	0.58	0.57
8	0.92	0.94	1.06	0.60	0.60	0.55
9	0.93	0.98	1.00	0.58	0.62	0.59
10	0.94	0.93	0.99	0.60	0.62	0.57

11	0.94	0.97	1.02	0.60	0.66	0.64
12	0.99	0.94	0.99	0.60	0.67	0.61
Average	0.88	0.91	0.96	0.55	0.60	0.53
StDev	0.06	0.06	0.07	0.04	0.04	0.07
Average		0.92			0.56	
Sample		A9				
A _w		0.74				
Free fat		35.3				
Sub-sample	1#	2#	3#			
Repeated measurement						
1						
2						
3						
4						
5						
6						
7						
8		Flow Failure				
9						
10						
11						
12						
Average						
StDev						
Average						

Appendix C-1

One-way ANOVA analysis of variance for the preliminary study on the relationship between moisture content and flowability

One-way Analysis of Variance

Analysis of Variance for flowability						
Source	DF	SS	MS	F	P	
moisture	2	0.7565	0.3782	5.73	0.041	
Error	6	0.3961	0.0660			
Total	8	1.1526				
Individual 95% CIs For Mean						
Based on Pooled StDev						
Level	N	Mean	StDev			
18	3	1.2767	0.1124	(-----*-----)		
21	3	1.7133	0.2363	(-----*-----)		
22	3	1.9800	0.3600	(-----*-----)		
-----+-----+-----+-----						
Pooled StDev =		0.2569		1.20	1.60	2.00

Appendix D-1

Sieve analysis results of each of the mixtures of 50017, 69430 & Kraft

50017			
Cumulative undersize (%)			
Sieve size (µm)	M1 (100/0)	M2 (85/15)	M3 (80/20)
1700	98.87	98.77	99.13
1000	54.04	58.90	62.10
850	35.37	41.64	43.79
600	10.20	19.08	20.93
355	3.17	8.80	9.78
Sieve size (µm)	M4 (75/25)	M5 (70/30)	M6 (65/35)
1700	99.07	98.88	99.08
1000	64.72	65.60	66.50
850	48.11	48.75	51.00
600	28.05	29.37	30.34
355	15.18	16.53	14.00
Sieve size (µm)	M7 (60/40)	M8 (55/45)	M9 (0/100)
1700	99.26	99.28	99.95
1000	72.49	71.43	94.17
850	52.64	55.94	85.58
600	32.67	35.39	61.97
355	20.56	18.61	33.54

69430			
Cumulative undersize (%)			
Sieve size (µm)	M1 (100/0)	M2 (85/15)	M3 (80/20)
1700	98.58	98.24	98.66
1000	50.72	54.12	57.30
850	31.06	34.07	34.61
600	6.41	13.10	18.13
355	2.27	7.53	12.40
Sieve size (µm)	M4 (75/25)	M5 (70/30)	M6 (65/35)
1700	98.69	98.88	98.75
1000	58.65	62.36	65.50
850	37.79	40.66	42.31
600	19.30	22.69	28.63
355	13.04	14.93	17.83
Sieve size (µm)	M7 (60/40)	M8 (55/45)	M9 (0/100)
1700	98.82	98.76	100.00
1000	66.53	70.03	88.89
850	44.27	47.38	72.00
600	33.57	27.35	48.11
355	19.18	16.55	27.81

Kraft			
Cumulative undersize (%)			
Sieve size (µm)	M1 (100/0)	M2 (90/10)	M3 (85/15)
1700	991.7	99.06	99.13
1000	76.90	77.74	79.40
850	62.28	64.38	66.54
600	35.82	39.88	43.54
355	15.79	20.58	23.63
Sieve size (µm)	M4 (80/20)	M5 (75/25)	M6 (70/30)
1700	99.29	99.12	99.28
1000	81.92	81.83	82.81
850	70.39	70.52	71.91
600	35.82	50.92	52.37
355	15.79	33.51	37.66
Sieve size (µm)	M7 (65/35)	M8 (60/40)	M9 (0/100)
1700	99.46	99.35	100.00
1000	85.23	86.64	99.89
850	75.95	78.12	99.77
600	58.61	61.70	99.20
355	39.99	44.63	82.89

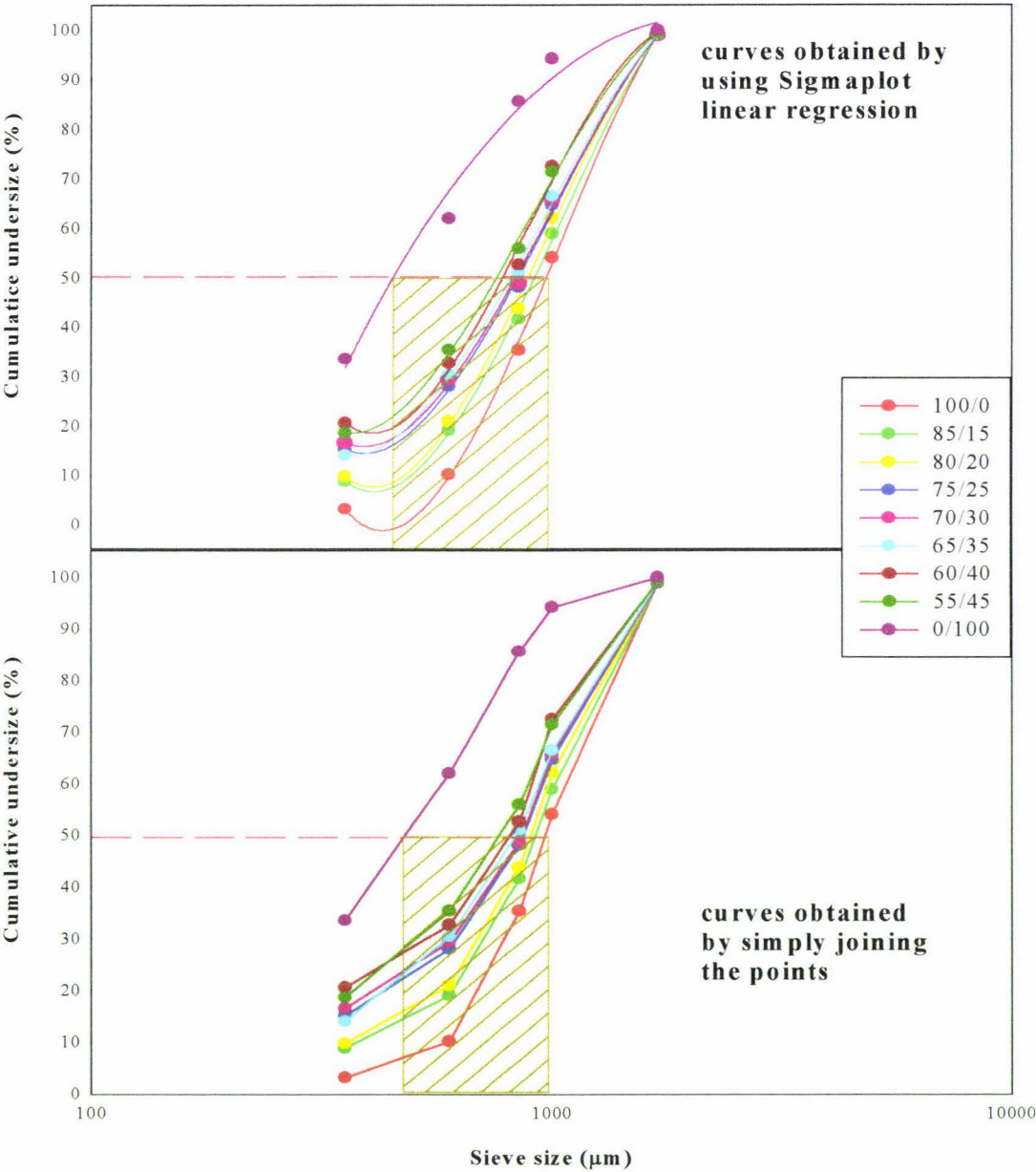
Appendix D-2

Sieve analysis results of each of the samples at different free fat levels of 50017HM

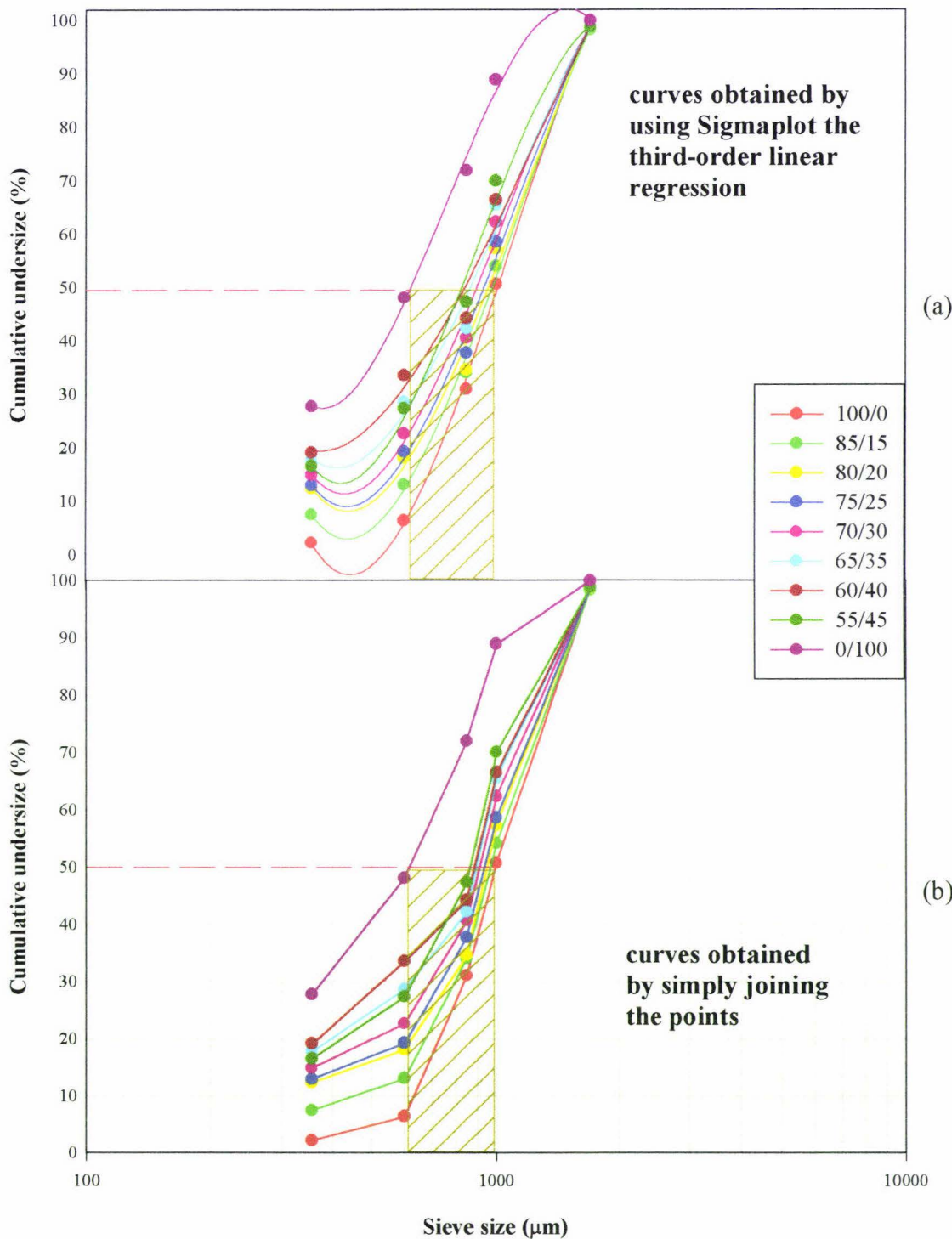
50017HM			
Cumulative undersize (%)			
Sieve size (µm)	F1	F2	F3
1700	99.57	98.42	98.80
1000	76.00	70.80	70.31
850	65.77	59.39	58.44
600	48.84	40.20	38.63
355	33.55	25.65	23.78
Sieve size (µm)	F4	F5	F6
1700	98.76	98.22	96.36
1000	68.25	66.98	57.66
850	56.24	55.27	41.80
600	37.28	34.37	16.35
355	19.36	15.43	6.99
Sieve size (µm)	F7	F8	F9
1700	94.69	76.93	Too sticky to be determined the particle size distribution by sieving
1000	42.54	8.92	
850	25.56	2.90	
600	6.37	1.36	
355	2.51	0.85	

Appendix D-3

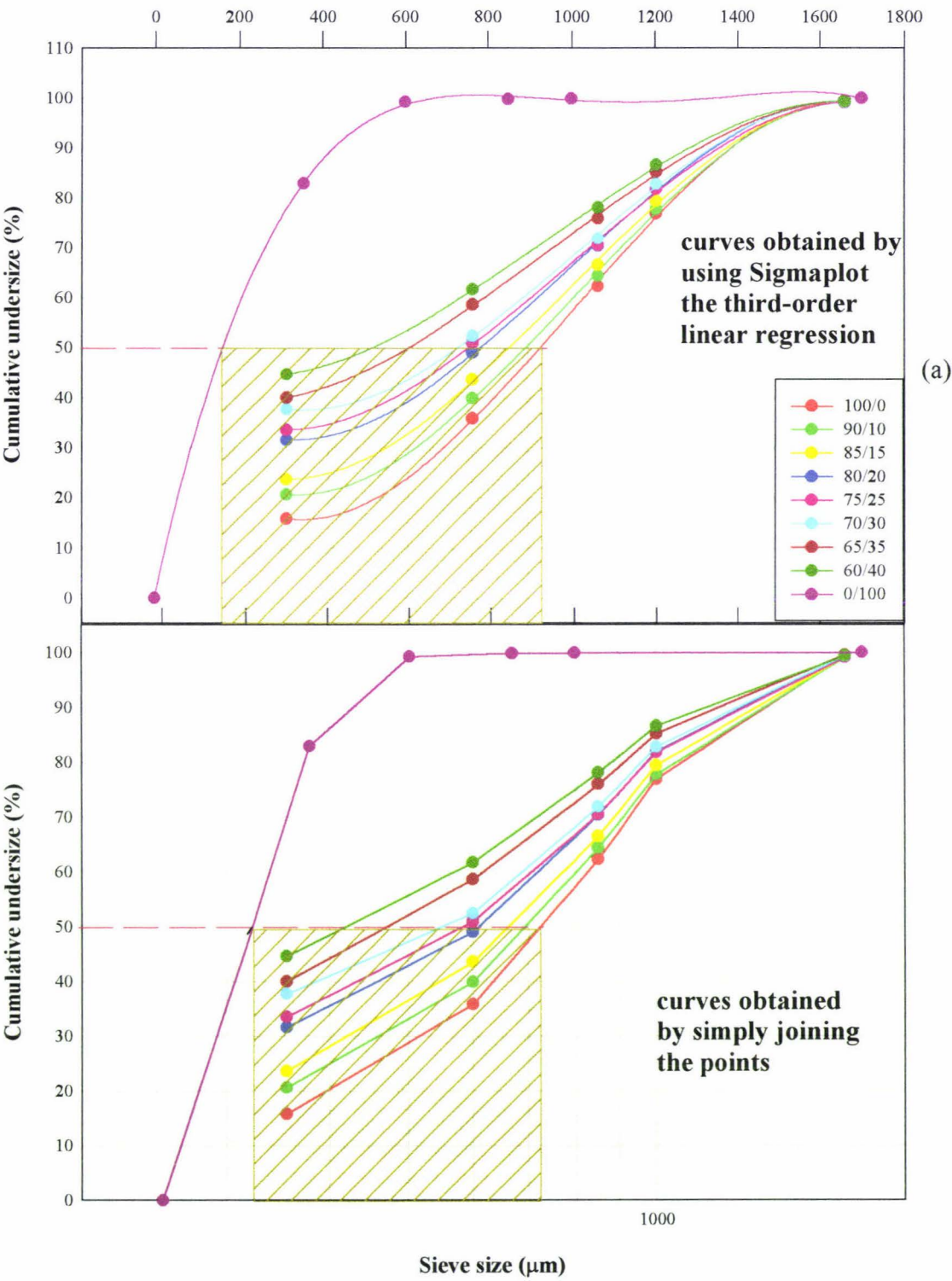
Cumulative undersize against sieve size of 50017, 69430 and Kraft



(a) 50017



(b) 69430



(c) Kraft

