LACTOSE SMEARING IN TRANSPORT LINES

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ABSTRACT

The smearing of lactose in pneumatic conveying lines, leads to cakes of lactose building up within the lines. This is an undesirable situation as it leads to reduced throughput, caused by the narrowing of the lines and the increase in downtime required to unblock and clean the pipes. This study was carried out to investigate the causes of smearing and identify solutions to this problem.

Impact testing was carried out, to look at the breakage behaviour of lactose. This identified that energy of impact is the main consideration for the breakage of lactose in pneumatic conveying. This is not only the energy contained before impact, but also the way in which the energy is dissipated during contact. The use of rubber proved an effective technique in lowering the amount of breakage , due to its ability to adsorb and disperse the impact energy during contact.

Testing was carried out looking at the ability of sliding contact to cause the adhesion of lactose to a surface. The results showed that combination of the frictional forces and the sliding velocity can provide enough energy to cause the lactose to adhere. The conclusion drawn was the same as that for impact testing, with energy being the main consideration in the breakage and adhesion of lactose to surfaces.

A link between amorphous lactose formation and the smearing was found, with the build up in the conveying lines having a higher amorphous concentration than was found on the free flowing lactose powder. An attempt to show a change in the amorphous concentration of α-lactose crystals after impact proved unsuccessful, although the use of a polarised microscope showed the formation of amorphous lactose on the impact surface. Calculations looking at the amount of amorphous lactose that would have formed after impact, identified that the concentrations were below the levels measurable using the methods available.

Following on from impact testing work, a rubber lined bend was placed in a section of the conveying line at Lactose New Zealand. Monitoring of this bend showed it to be successful in preventing the adhesion of lactose to walls of the conveying pipe. However, there was a small amount of wear observed at the entrance of the bend. This was concluded to be due to a design defect as the rubber was raised above the level of the main line. More testing needs to be done, with a change in design, to allow a conclusion on the applicability of rubber for preventing lactose buildup to be drawn.

ACKNOWLEDGEMENTS

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Chapter 1 Project Overview

1.1 Problem Definition

Pneumatic conveying is the transport of solid materials through vertical and horizontal pipelines by the conversion of kinetic energy in the air stream to dynamic pressure and aerodynamic lift (Kraus, 1980). As a method of conveying lactose powder the pneumatic option provides a transport system with the advantages that it is enclosed, dust free and enables easy distribution throughout the plant.

The problem to be addressed here is that, using pneumatic conveying lines to transport lactose often results in lactose depositing and building up on the pipe walls. This can lead to large cakes of lactose building up in pipelines, with the end result that, the lines eventually become blocked. This is an undesirable situation, as it results in decreased throughput, caused by the narrowing of the lines and the increase in downtime required to unblock and clean the pipes.

A review of literature the highlighted the following points that are of interest in attempting to understand the parameters involved in causing the smearing to occur. The details of this literature review are expanded further in chapter two.

- How a material behaves in a pneumatic transport line is dependent on the properties of its particles.
- Lactose cakes often form in areas where significant impacts between crystals and surfaces occur.
- High particle velocities in pneumatic transport systems cause particle degradation to occur and alter the physical properties of the materials being conveyed.
- Where moisture and amorphous lactose are present, caking in lactose is increased.
- Amorphous lactose is formed during intensive mechanical breakage of lactose crystals.
- Buildup in pneumatic conveying lines is often prevalent at bends, an area where impact is more likely.

- The type of bend used in a pneumatic transport line has a strong effect on how the system behaves.

The above points highlight that understanding solids deposition requires that a knowledge of the properties of the powders being conveyed, combined with a knowledge of the conditions that occur within pneumatic conveying lines. This means that this work has two areas of interest. The first requires that, an understanding of the properties of lactose and the conditions under which it will adhere to itself and other surfaces is developed. The second requires that the conditions that particles are exposed to, during pneumatic conveying, are studied to determine the role they play in causing solids deposition.

1.2 Adhesion of Lactose

Adhesion of lactose has been showed in previous studies to be a function of both, the physical properties of the powder, and also the conditions to which it is exposed (Bronlund, 1997, Brooks, 2000). The physical properties relate to factors such as, particle size, shape and the condition the powder is in.

1.2.1 Amorphous Caking

Amorphous caking can occur when some or all of the lactose powder is in the amorphous form. The caking occurs as a result of the conditions enabling the amorphous lactose to transform from the solid glass phase into a less viscous rubber state. In this less viscous rubber state the molecules are capable of flow. This permits bridges to form between the particles and other surfaces. The formation of these bridges generates strong adhesion forces that cause the particles to lump together.

Amorphous caking is of particular interest to this work, as previous work has shown that amorphous lactose can be formed when crystalline lactose undergoes intensive mechanical treatment (O'Donnell, 1998, Lerk *et al.*, 1984). The mechanical processing typically involves impact and breakage of the lactose crystals. It is in the areas where significant impacts between crystals and other surfaces occur that lactose cakes tend to form. This suggests that there is a link between impact, breakage and the buildup that occurs in pneumatic conveying lines. The observation also suggests that these conditions may result in the formation of amorphous lactose, and also allow for transition of the amorphous lactose from the glass phase into the rubber phase.

1.3 Problem Areas in Pneumatic Conveying

In order for changes in particle properties to occur certain conditions have to be met. The two factors which seem to effect particle properties in pneumatic conveying the most are, velocity and bends.

1.3.1 Particle velocity

Achieving the optimum particle velocity in a pneumatic transport pipeline is a difficult task. Too fast and you risk high levels of particle degradation. Too slow and particles fall out of the gas stream, deposit at the bottom of the pipe and cause blockages. Velocity is a critical variable influencing the way a material behaves in a pipe, with high velocities resulting in, not only particle degradation, but also having an effect on the way the solids interact with the walls of the pipe line (Thorn *et al.*, 1998).

1.3.2 Bend Structure

Examination of where the buildup occurs in the pipelines at Lactose New Zealand, identified that it is at the bends where a large amount of the problems occur. Due to the changing flow direction, the bend is the point where the particles are subjected to the greatest impact. Work in the literature identifies that the type of bend used in a pipeline has a role in the level of damage done to the particles. Also a bend in a pipeline can also have the effect of lowering the flowrate of the material being transported, causing particles to fall out of the gas stream, deposit and result in a blockage.

1.4 Overall Project Aims

The overall aims of this research were to:

- Develop an understanding of what mechanism(s) resulted in lactose depositing on the pipe walls.
- Investigate the effect of impact on the physical properties of lactose crystals

- Provide solutions which allow pneumatic transport systems to be designed and operated in a manner that prevents lactose buildup occurring in the pipes.

Chapter 2 Literature Review

2.1 Introduction

The objective of this work is to identify what causes lactose to deposit on the inside of pneumatic conveying lines. To do this requires that a knowledge of the physical properties of lactose be developed. It also requires that certain aspects of pneumatic conveying are understood. The purpose of this chapter is to introduce these two concepts in preparation for the chapters which follow.

2.2 Chemistry of Lactose

Lactose is a sugar found in the milk of most mammals. It is the major carbohydrate source obtained from animals, in the human diet. It forms over 50% of the solids in skim milk and an even greater proportion of the solids in whey (Nickerson, 1979). Lactose sugar is a disaccharide molecule that yields D-glucose and D-galactose upon hydrolysis (Zadow, 1984).

In the powder form, lactose can exist as amorphous lactose, crystalline lactose, as either α-lactose monohydrate or β lactose or as a mixture. The crystalline lactose forms all have a very ordered molecular structure, unlike the amorphous form that, as the name suggests, has a predominantly disordered structure.

2.2.1 Crystalline Lactose.

The bulk of commercially produced crystalline lactose is α-Lactose monohydrate ($C_{12}H_{22}O_{11}.H_2O$). The monohydrate term refers to the one water molecule incorporated into the crystal lattice for every lactose molecule. It is prepared through crystallisation from an aqueous solution below 93.5°C. The shape formed by crystals is dependent on the conditions of crystallisation, although the most common are the prism and tomahawk shapes (Van Krevald & Michaels, 1965). When crystallisation is carried out above 93.5°C, β-lactose is formed. Crystalline β-lactose has different properties to α-lactose including greater sweetness and solubility. α- and β- lactose also differ in specific rotation, melting point and hygroscopicity (Pritzwald-Stegmann, 1986)

2.2.2 Amorphous Lactose

Amorphous lactose can be visualised as a very concentrated lactose solution, where the solution has been dried so quickly that the mobility of the molecules becomes insufficient for crystallisation to occur (Brooks, 2000). The common method for manufacturing amorphous lactose is to dry a solution of lactose rapidly so that crystallisation can not take place. This is typically done by spray drying. The resulting dry lactose is in the same disordered state as it was in the solution, except the mobility of the molecules is very limited

There are other methods of manufacturing amorphous lactose. Roos & Karel, (1990) made amorphous lactose by freeze drying a 10% solution of α-lactose monohydrate in distilled water. α- Lactose was shown to lose its water of crystallisation and change into an amorphous state on undergoing intensive grinding Lerk *et al.*, (1984). (Otsuka *et al.*, (1991), also showed that crystalline lactose could be transformed into non-crystalline lactose by mechanical stress.

It is the transformation of α-Lactose to amorphous lactose through mechanical stress, that is thought to have be one of the primary causes of the buildup that occurs in the pneumatic conveying lines. Of particular concern to this study, is how amorphous lactose can, through breakage in pneumatic conveying lines, be inadvertently formed.

2.2.2.1 Glass Transition Temperature

The glass transition temperature (T_g) has been shown to play an important role in the caking and sticking of amorphous lactose powders (Brooks, 2000). Below the glass transition temperature amorphous materials exist in a stable glassy and highly viscous state. When the glass transition temperature is exceeded the amorphous material enters the much less viscous rubber state. In this state, the previously immobile molecules can begin to move past each other within the amorphous matrix (Aguilera *et al.*, 1995).

It is suggested that at temperatures exceeding T_g viscous flow will be promoted and the potential for caking therefore exists (Lloyd *et al.*, 1996). The amount that T_g is exceeded by determines the viscosity of amorphous lactose in the rubber state, and so determines how fast and to what extent it becomes sticky. It was found that where ($T-T_g$) was exceed by 25°C amorphous lactose became sticky almost instantaneously (Brooks, 2000). The value of T_g for amorphous lactose is dependent on the moisture content of the powder. As the moisture content is increased, lower temperatures are required to transform amorphous lactose from the glassy to the rubbery state (Slade & Levine, 1991, Sebhatu *et al.*, 1994b). The humidity of the surrounding conditions can have a significant effect on the moisture content of the powder.

Slade & Levine, (1991) state that due to moisture sorption or a temperature rise, T_g may be exceeded and as a result amorphous materials enter the rubbery state and the decreasing viscosity induces flow and deformation. T_g values of 100% amorphous lactose were reduced as the moisture content increased, i.e. lower temperatures are required to transform amorphous lactose from the glassy state to the rubbery state with increasing moisture content (Sebhatu *et al.*, 1994b).

2.3 Solids Deposition

Solids deposition is the buildup of powder deposits in pneumatic conveying lines. As the buildup occurs, the lines become blocked and capacity is reduced. The deposition of lactose on the walls of pneumatic transport lines is the main focus of this work.

The type of deposition observed within pipelines can be one of the following:

- Annular deposition, shown in Figure 2-1(a), is the formation of a thin layer of material on the inside surface of the pipe and is due to the adhesive nature of the powder being conveyed (More common in lean phase).
- Gravitational deposition, shown in Figure 2-1(b), is the buildup of solids at the bottom of the pipe. It is typically associated with low conveying velocities, and occurs in both lean and dense phase transport (Yan & Byrne, 1997).

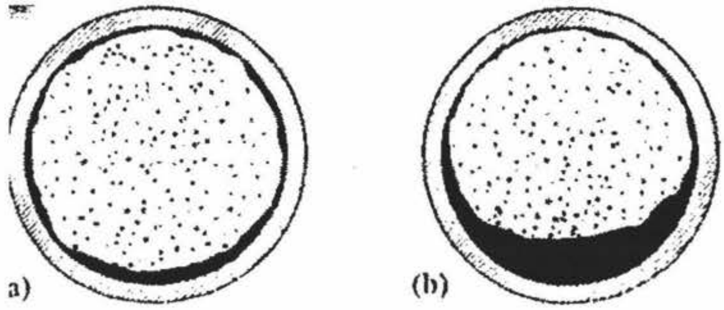

Figure 2-1 Solids deposition in a pipeline (Yan & Byrne, 1997)

Solids deposition is a function of the properties of the powder being conveyed and the conditions that exist within the conveying lines. Of the large number of conditions encompassed by the above definition, four have been identified as being the major factors influencing the buildup of powders in pipelines (Kalman, 1999).

- Properties of the solids
- Velocity
- Impact of Particles
- Mode of Conveying

2.3.1 Properties of the Solids

Particle size can effect the amount of deposition observed in a pneumatic conveying line. Fine particles often show higher levels of deposition when compared to coarse particles of the same material. Yan, (1995) found the solid deposition rate of coal to be an unstable mechanism, related to the cohesiveness of the material and the size of the particles, with fine particles being more likely to deposit. It was also suggested that deposition was enhanced by the presence of moisture. Decreasing the particle size or increasing the moisture content increases the cohesivity and adhesivity of a material (Rhodes, 1998).

Small particles can also be more prone to deposition due to their increased propensity to cake. The presence of fine particles was found by Roge & Mathlouthi, (2000) to make sugar more likely to cake. This was concluded to be due to the small particles being more readily dissolved by moisture in the air. The dissolved fines were then able to form inter-particle bridges between the sugar crystals. The presence of the bridges increased the cohesiveness of the crystalline sugar and yielded irreversible caking.

It is also possible that large particles will deposit in lines. Large particles have more inertia and so impact on surfaces with more energy. They are also more difficult to fluidise and are likely to fall out of the gas stream and deposit on the bottom of the pipe (Coulson *et al.*, 1999).

The cohesiveness of a powder can influence how or even if it can be conveyed pneumatically. Highly cohesive powders may bind to themselves or other surfaces forming buildups in the pipes. Cohesive solids tend to coat walls of pipes and may, over time, reduce the solids throughput by reducing the effective flow area and ultimately leading to the blockage of the pipeline (Venkatasubramanian *et al.*, 2000). The causes of bonding in these powders will be looked at in more detail in the section on cake formation

2.3.2 Particle Velocity

Velocity in a pneumatic conveying system needs to be maintained around the minimum safe value to achieve optimum operating conditions. Excessively high particle velocities cause high-energy consumption, pipeline wear and particle degradation. This has to be balanced against excessively low velocities, that can result in particle stratification and can cause pipeline blockages (Yan, 1995; Cabrejos & Klinzing, 1994).

The desired velocity for operation of a pneumatic conveying system is the minimum gas velocity that will provide enough force to transport the particles along the pipeline, and for lean phase transport to ensure they remain in suspension. This minimum conveying velocity is known as the saltation velocity.

The saltation velocity is defined as the superficial gas velocity below which the particle begins to separate from the gas phase and to slide or roll along the bottom of the pipe (Hong *et al.*, 1995). The saltation velocity is also a function of the solids loading factor, with the saltation velocity being higher, at higher particle concentrations (Purutyan *et al.*, 2001).

Predicting the minimum conveying velocity is a complex process. It is influenced by a number of factors, including the material characteristics, gas properties, and the configuration of the conveying system (Cabrejos & Klinzing, 1994). For this reason a conveying system is often operated based on experience and above the minimum velocity.

It is hard to maintain a constant gas velocity in the conveying line with pressure drop causing gas expansion and leading to higher gas velocities, which can correspond to higher particle velocities. This increase in gas velocity in a pipeline can be overcome by increasing of the pipeline diameter at strategic points along the line to reduce the gas velocity; a technique known as stepping. The ideal design of a pipeline is one where the diameter increases gradually to maintain a constant gas velocity. The design of such a pipeline would be expensive and unrealistic, so stepping is used instead (Thorn *et al.*, 1998).

It is also important to understand how a particle behaves in a pneumatic conveying line at different velocities. As it may be possible to transport powders at higher than their minimum conveying velocities without having any negative effects occur. Higher conveying velocities provide a safety factor above the saltation velocity and increase the throughput of a system. The average thickness of the settled layer at the base of the pipe was lowered with increasing air velocity and decreasing the solids loading factor (Mckee *et al.*, 1995).

For α-lactose at low impact velocities, 5m/s to 11.2m/s, very little breakage was observed (Arteaga *et al.*, 1996). As the velocity was increased from 11.2 m/s to 35.9m/s, a steady increase in the amount of particle degradation was observed. Bridle *et al.*, (1999) looked at conveying velocities for barley grains, rice and sugar. The study found that at air velocities above 30m/s particle degradation increased significantly, but below 20m/s degradation was relatively small. The conclusion that can be drawn, from this work, is that the conveying velocity most suited to minimising breakage and at the same time ensuring efficient conveying is material dependent. To correctly operate a pneumatic conveying system requires an understanding of how the conditions will affect individual powders.

2.3.3 Bends

Pneumatic conveying is designed to get solids from one location to another. It is rare that these points can be connected using a straight section of pipe. This creates a need for bends to be used in order to change the direction of the flow. Bends add complexity to how a pneumatic system behaves, they alter flow patterns, change velocity and are a significant point of impact and blockage.

During a pneumatic conveying cycle the particles experience extensive impact loads. These occur mainly at the bends, due to the changing flow direction (Kalman, 1999). Upon exiting a bend, the suspension velocity can be reduced enough to cause a large deposit downstream of the bend (Venkatasubramanian *et al.*, 2000). As solids pass round a bend, they are slowed by friction and impact, and energy is expended as they are reaccelerated towards their equilibrium velocity (Davies *et al.*, 1998).

Typically three types of bends are used in pneumatic conveying lines, the blinded Tee, the long radius bend and the short radius bend. The type of bend used is influenced by the material being conveyed, with the different bends having varying effects on how a system will perform.

The long radius bend is designed to reduce pressure drop and particle attrition through providing a gradual change in the direction, rather than the sudden change that occurs with the blinded Tee and short radius bend. It has the disadvantage that it is longer and takes more space to change the direction of flow. Kraus, (1980) states that the longer the radius, the less abrasion, the lower the possibility of compaction in the bend and the lower the pressure drop.

The Blinded Tee is designed around the assumption that the Tee section allows a pocket of the material being conveyed to build-up. The conveyed material then impacts on itself as opposed to impacting on the pipe wall. This serves two main purposes; the first is that through having the powder impact on itself, rather than the pipe wall, the level of impact is reduced. The second is, for particles that cause high levels of abrasive wear, the amount of contact with the pipe wall is reduced in an area of high impact.

A study of the three bends discussed above was carried out using coal as the particulate material. The level of build up observed over time was greatest for the long radius bend, then the short radius and then finally the blinded tee. At the primary point of impact for the long radius bend, a deposit had become firmly adhered to the walls of the pipe (Venkatasubramanian *et al.*, 2000).

The type of bend used will also effect the impact between a particle and the wall. A 90° bend means the particle hits straight on. As the bend radius increases, the impact becomes more tangential so rather than hitting the wall straight on the particle may slide along the wall (Kalman, 2000). This increased contact time may explain why more build up occurred on the long radius bends studied by (Venkatasubramanian *et al.*, 2000).

When different bend types were compared for conveying of barley and sugar, the level of attrition observed in a long radius bend was equal to or less than when a short radius bend or blinded tee was used. However, this same study showed that when conveying rice at suspension densities above $5kg/m^3$ the blinded tee performed the best (Bridle *et al.*, 1999).

The literature on the best bend to use for a pneumatic conveying system indicates that the best bend is dependent on the material and conveying conditions. This may be due to the way different materials handle different impact conditions. The tight bends, such as the short radius and blinded tee have higher initial impacts but the change is abrupt. The long radius bends may reduce the initial impact levels but carry the instability over a longer distance (Venkatasubramanian *et al.*, 2000). They also increase the amount of time the solid is in contact with the surface, increasing the potential for bonds to form.

It is often assumed that the pressure loss will be generally greater in blinded Tees and short-radius bends than in long-radius bends. However, it has been observed that after a certain point the increase in pipe length required to increase the radius of the bend negates any benefit that may be gained through providing a more gradual turn (Purutyan *et al.*, 2001).

The material of construction used in bends has been shown by Kalman, (2000) to influence the level of particle breakage. More flexible rubber bends exhibited much lower attrition rates than the rigid stainless steel bends.

2.3.4 Fluidisation

From experimental observations it has been seen that the fluidising behaviour of a gas solid system changes drastically within the broad range of particle sizes and different fluids used in industrial applications (Qian *et al.*, 2001). How a powder fluidises may determine how it functions in a pneumatic conveying line. Geldart, (1973) provided four groups, A,B,C and D, to distinguish between the different behaviour of solids fluidised by gases. The groups are characterised by the density difference between the solid and the gas (ρ_s-ρ_f) and their mean particle size.

Geldart's observations were as follows

- Powders in group A exhibit dense phase expansion after minimum fluidisation and prior to the commencement of bubbling. Particles in this category have a small mean size and/or a low particle density (less than 1.4 kg.m^{-3})
- Powders in group B bubble at the minimum fluidisation velocity. Bed expansion is very small. Particles in this category fall in the mean size and density range of 40μm<d_{sv}>500μm, 4g/cm^3>ρ_s<1.4g/cm^3.
- Powders in group C are difficult to fluidise at all, since the interparticle forces are greater than the force the fluid can exert on the particles. Attempts to fluidise result in the powder forming a plug or rat holes being formed. Particles that are in any way cohesive fall into this category. These powders are generally finer than the powders in group A

- Powders in group D form stable spouted beds. Particles in this category are large and dense. Solids mixing is poor and the turbulent flow regime around the particles can result in particle attrition.

The category a powder falls into can change depending on the fluidising conditions. For example, a powder that falls into group C may evince group A powder behaviour when gas of higher viscosity is used (Rietema, 1984). It is also possible for the properties of powders to change as they undergo processing . Where powders are subject to factors such as attrition the material properties may change, leading to a change in the fluidisation behaviour of the solid.

Rietema, (1984), thus proposes that rather than classifying powders into the groups A,B,C and D it better to talk about A,B,C and D behaviour. This idea becomes important when designing a pneumatic conveying system as it means the way a powder will behave when fluidised is not fixed. A knowledge of how the powder behaves under different conditions may allow the system to be operated in such a manner that problems with fluidisation can be reduced.

2.3.5 Modes of Conveying

There are three modes or phases of transport employed in pneumatic conveying. These are shown in Figure 2-2. The differences between the phases are the gas to solid ratio and how they move. These are phases are;

- Lean or dilute phase
- A moving bed
- Dense phase

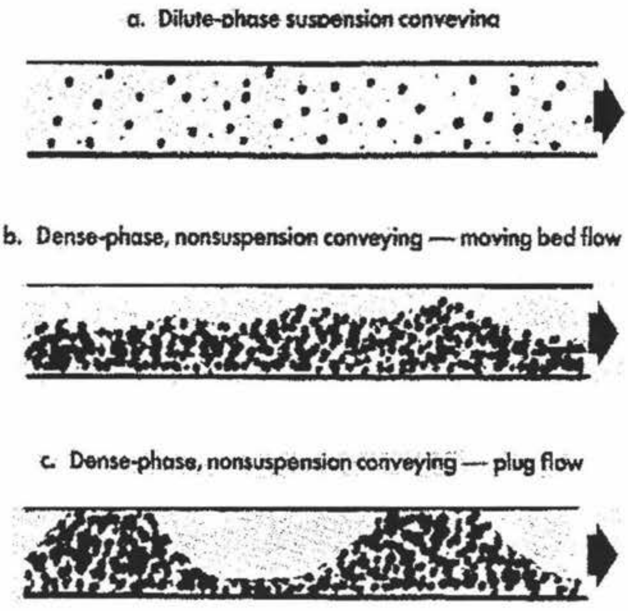

Figure 2-2 Pneumatic conveying modes (Reed & Bradley 1992)

The best flow mode for a bulk solid is largely determined by the material properties, in particular those which involve particle air interaction (e.g. air retention, de-aeration, permeability). These characteristics are usually a function of the basic particle properties, especially particle size, size distribution, density, shape and hardness (Pan, 1999).

2.3.5.1 Lean Phase

Lean phase conveying is the transport of solids through a pipe in a suspension. It is possibly the simplest method to design as it is less limited by the fluidising properties of the materials being conveyed. As long as a solid can be transported pneumatically it is possible to transport it in the dilute phase (Pan, 1999).

Keeping the solids in suspension requires that a low solids loading ratio is maintained. What constitutes lean phase transport is only loosely defined . Lean Phase is a state where the concentration of solids does not exceed 0.05 and a high proportion of the particles spend most of their time in suspension (Coulson *et al.*, 1999). The volumetric concentration of solids in the pipe for lean phase conveying has to be quite low, typically less than 10% (Konrad, 1986).

A lean phase system also requires relatively high gas velocities to provide enough lift for the particles to remain suspended. This is where a large number of the difficulties associated with lean phase conveying arise. The high gas and ultimately high particle velocities required, lead to problems like particle degradation and pipe wear.

2.3.5.2 Moving Bed

Moving bed conveying is a form of dense phase conveying, with the difference being that the particles are swept along the pipe in a bed formation rather than the conventional plug flow of dense phase conveying. The solids loading ratio in a moving bed system may be as high as 0.6. This phase of transport is only relevant where the pipelines are horizontal (Coulson *et al.,* 1999).

2.3.5.3 Dense Phase

Dense phase conveying is the conveying of solids along a pipe that is filled with solids at one or more of its cross sections (Konrad, 1986). Dense phase allows a lower airflow velocity to be used than a lean phase system. This, along with the particles being partially supported by other particles and reduced contact with the walls, can reduce the level of pipe wear and particle attrition (Reed & Bradley, 1992)

A study done by (Bridle *et al.,* 1999) found that dense phase conveying resulted in less particle degradation. A possible reason for this is that the proportion of particles impacting against the wall is decreased Whilst inter-particle collisions would have increased, the effect of these on degradation would have been much less severe than the wall impacts.

Dense phase conveying is not suited to all types of materials and pneumatic conveying systems. Pan, (1999) looked at a different range of materials and how they behaved in different modes of conveying. Heavy granular and crushed products, along with some fibrous spongy materials, were found to be unsuitable for dense phase transportation. These materials were found to have a tendency to interlock together, affecting flowability.

It is also possible that in a dense phase system, a high pressure drop may occur across the length of the conveying line. The high expansion caused as a result of this can result in high velocities occurring at the end sections of the line (Reed & Bradley, 1992).

2.4 Cake Formation Mechanisms.

To understand why lactose forms deposits in pneumatic conveying lines it is necessary to identify what causes free flowing lactose particles to lump together, and to adhere to pipe walls. This section examines the literature on the mechanisms of adhesion that play a role in the particle-particle and particle-wall interactions exhibited by lactose powders.

These mechanisms of adhesion may be grouped into the two categories of caking and sticking. The difference between the two categories is not always clearly defined and the words are often used interchangeably. It may be assumed here that stickiness refers to the tendency of a material to adhere on a surface of similar or different type. Such adhesion may occur temporarily and does not necessarily involve caking. Caking may be considered as a collapse phenomenon that occurs due to stickiness of particles that form permanent aggregates and harden into a mass, which results in the loss of the free flowing properties of powders (Roos, 1995).

Using the definition provided by Roos (1995), it would appear that in the case of pneumatic conveying, sticking is the most important phenomenon, as it is the reason for particles adhering to each other and thus building up in the pipelines. Caking also needs discussion, as it is the mechanism that leads to permanent bonds and deposits forming. This has implications regarding problems with cleaning and the possibility of the blockage being self clearing.

2.4.1 Interparticle Interactions.

A number of interactions play a part in the bonding of particles to surfaces. These are listed as follows;

- van-der Waals forces
- Electrostatic and magnetic forces
- Liquid bridges

- Solid bridges
    a) Sintering, i.e. solidification after melting
    b) Crystalline or amorphous solidification of dissolved substances after drying

(Okuyama & Kousaka, 1991, Roth, 1976, Rumpf, 1990).

Particle size and size distribution also affect the tendency for caking, they can alter the number of contact points between particles. A minimum caking tendency is shown by, relatively large, well formed and uniformly sized and shaped particles due to there being relatively few contact points per unit volume (Mullin, 1972).

2.4.2 Sticking

Stickiness has been defined above as the tendency for a particle to adhere onto a surface. If the right conditions exist, it is a phenomenon that can occur instantaneously.

A lot of the literature on caking and sticking of powders relates to storage. The major difference between caking during storage and caking during manufacture is the time caking takes to occur. During storage it may take many months, while during manufacture it may take hundredths of a second (Crofskey, 2000). Caking during storage is a result of the conditions allowing the particles to form bonds, and is dependent on the environment and condition of the powders. During manufacture these factors are subject to rapid changes. This along with the movement of the powders causing forced interaction, allows for much lower caking times. Another difference is that when looking at the storage of powders it is only the particle-particle interactions that are considered. During manufacture it is also important to consider the interactions between the particles and the processing equipment and the effect this has. This idea highlights the fact that two different interactions need to be examined when looking at sticking during pneumatic conveying; particle-particle sticking, (cohesion), and particle-wall sticking, (adhesion).

2.4.2.1 Cohesion

In powders the cause of caking and sticking is almost always liquid bridging between particles, as opposed to electrostatic or molecular attraction forces (Bronlund &

Paterson, 1997, Peleg, 1993). For this reason, liquid bridging will be the major focus when looking at the cohesion of powders. For liquid bridging to proceed, the surface of the crystals must be fluid, at least at certain sites (Peleg, 1993).

Peleg, (1993) gave the following mechanisms by which liquid bridging may proceed;

- Accidental Wetting
- The transformation of an amorphous material into a highly viscous material
- Liberation of adsorbed water
- Melting of the surface

In pneumatic conveying, it is unlikely that accidental wetting or liberation of absorbed water will play a roles in liquid bridges forming due to the nature of the process. For this reason, it is only melting and the transformation of an amorphous material into a viscous material that will be considered further.

Discussed in section 2.2.2.1 is the glass transition temperature and its effect on the viscosity of amorphous lactose. It is proposed that upon impact amorphous particles in pneumatic lines adsorb enough energy to cause T_g to be exceeded, thus lowering their viscosity to a point where they become sticky. It is not required that all the mass undergo a phase change. Provided the surface material is transformed, bridging will still occur, even if the average particle temperature lies well below T_g (Peleg, 1993). Where amorphous lactose is not present in the feed it is proposed that, due to the mechanical stresses imposed by pneumatic conveying, crystal breakage occurs. As a result of this molten lactose is formed on the surface of the particles. This cools forming amorphous lactose. This will be further discussed when looking at particle degradation.

Under conditions that may be encountered by particles during pneumatic conveying melting of the crystals is a possibility. Impacts of the crystals on walls and other crystals focuses a large amount of energy of one point. Weichert, (1976) estimated the temperatures generated when one sugar crystal was impacted against another to be more than 2000°C. This compares 180°C which is the melting point of sucrose. Fuller *et al.*, (1975) observed that large temperature rises occurred in the fracture crack of polymers, and concluded that the rise was large enough to have an effect on the properties of the surrounding material. As the crystals impact, the crack formation may have the same effects as observed in polymers and melting may occur.

2.4.2.2 Adhesion

The deposition of powder from a pneumatic suspension to substrate (wall) results from contact between a particle and the wall. It will occur if the adhesion force can overcome the inertial forces or re-suspension if the adhesion force is not strong enough (Rennie *et al.,* 1998). The adhesion of powders to a wall only provides the initial buildup. After the wall has been covered, the bonds formed are powder-powder, and cohesion rather than adhesion is occurring. Consequently, the powder wall bond determines how well attached the buildup is to the pipeline and thus the ease with which it is removed. The adhesive forces that cause particle wall bonds to form have already been discussed. Unlike cohesion, molecular forces such as Vander Waals forces and electrostatic forces also play a part. Electrostatic forces are stronger in materials that are conduct (Rennie *et al.*, 1998). This makes them more important when talking about particle wall interaction due to the nature of materials commonly used in pipe wall construction. However, static electricity is easily discharged, thus the likeliness of electrostatic forces causing permanent bonds is low. A more likely mechanism is that the electrostatic forces play a role in the causing the initial pipe-wall contact; from this more permanent bonds are able to form, causing buildup. The surface of the material also plays a part in the level of adhesion observed. Polished stainless steel was observed by Taylor, (1999) to exhibit less build up than unpolished stainless steel.

The mechanisms of melting and liquid bridging are the same as they were for cohesion and for this reason they are not readdressed in this section.

2.4.2.3 Moisture Adsorption

Generally, the most important force, from an industrial point of view, is adhesion resulting from moisture. This often results from beds that are in contact with a humid atmosphere where moisture transfer leads to water being adsorbed onto the surface of the particles. The problem is less obvious with porous particles, as the moisture often transfers to the internal surfaces, thereby allowing a greater weight of moisture uptake before any effect on flowability is noticed (Wright & Raper, 1998).

2.4.3 Caking

Caking is more of a time dependent process than sticking as it requires some change in the properties of the particles to occur. Caking requires that the bridges that form between the two surfaces are solid. Solid bridges can form during casual contact between particles. The solid bridge starts out as a liquid bridge, this subsequently solidifies, crystallises or reacts chemically with the solid. The precursor may be a local melt which cools, a solution which solidifies or a slurry which becomes more viscous (Tardos & Gupta, 1996). Solid bridges can occur as a result of sintering, chemical reactions, melting, hardening agents and crystallisation of dissolved material (Rennie *et al.*, 1998). It is unlikely that under the conditions encountered during pneumatic conveying that chemical reactions or hardening agents will be involved in the formation of solid bridges. For this reason, they will be ignored and only melting, sintering and crystallisation will be discussed further.

2.4.3.1 Melting

Most of the energy that goes into permanently, or inelastically, deforming a material is dissipated thermally. Bending a paper clip back and forth until it breaks, and then putting the broken piece to ones lips easily demonstrates this phenomenon (Zehnder *et al.*, 2000). In the collisions that occur in pneumatic conveying, the energy involved in the impact occurs over a short time frame and a small area.

In situations where the impact is high, the temperature generated may be enough to melt a material, causing it to stick, or to undergo physical changes that may make it more prone to sticking in the future. Solid bridges were observed to form between particles of sodium chloride in local "hot spots" of a ball mill (Krycer, 1980). It was proposed that these bridges were formed due to pressure and friction, which caused localised melting at the points of contact between adjacent particles. The melting temperature of sodium chloride is approximately 800°C, compared to the melting point of lactose which is approximately 200°C (Washburn, 1927).

For melting to play a role in caking, only the surface of the lactose crystals would need to melt. This would allow the lactose to flow and provide an area for bonding to occur. The low thermal mass of the melted area would require very little time for cooling and would return to a solid state almost instantaneously. It has been discussed above how the formation of cracks can generate high temperatures.

In pneumatic conveying, where particle velocities are high enough, it is possible that the energy released on impact will be enough to cause localised melting of the particles. This localised melting of the particles and then sudden cooling could provide a cause of caking. In a study looking at the buildup and solids flow behaviour in bends, Venkatasubramanian *et al.*, (2000) noted a deposit of coal had adhered to the wall of the pipe. It was suggested that the fine material had some special surface characteristic that made it adhere to the pipe wall. It is also possible that the volatility or moisture content could cause such behaviour in coal. In addition, the high velocity impact could cause temperatures to rise enough to bond the coal physically or chemically to the pipe surface.

2.4.3.2 Sintering

Sintering is bonding at a temperature below the melting point of a material, by application of heat. The sintering mechanism of amorphous materials such as glass, is usually controlled by plastic flow (Toyama, 1991). Visco-plastic sintering is one mechanism by which glassy materials adhere. It occurs as temperature is increased and the ability of the material to flow is increased. When two particles touch, the point of contact forms a neck, as flow increases the size of the necks increases until at some point they are able to overcome the forces required for fluidisation (Seville *et al.*, 1998). Sebhatu *et al.*, (1994b) suggested that even at very low moisture contents, a material that has regions in an amorphous glassy state before compaction, may, during the compaction phase, convert to the rubbery state due to the increased local temperature and pressure. Sintering would occur where the temperature was at a point that allowed flow to proceed. As a mechanism for sticking due to impact, it does not differ much from melting. The time of the temperature increase would be so small, that flow would need to be rapid, much the same as if melting had occurred. Where sintering becomes a consideration is when the temperature in the pipeline is high enough to allow bond formation but not melting.

2.4.3.3 Crystallisation

Crystallisation in lactose can result in the formation of solids bridges between the particles. Amorphous lactose is a hydroscopic substance that when the conditions are correct, can draw in water. This lowers the glass transition temperature, and can lead to the molecules becoming mobile enough to crystallise. Upon crystallisation the water is expelled and the neighbouring molecules become saturated causing them to crystallise (Darcy & Buckton, 1998a). Amorphous sugar flow and crystallisation at temperatures exceeding the glass transition temperature have been shown to be the cause of caking in many spray-dried or freeze dried foods (Bronlund & Paterson, 1997).

Caking of a powder may occur due to the moisture in the bulk powder or through adsorption of moisture from the surroundings. This results in the formation of liquid bridges at the particle-particle contact . Changes in temperature or humidity can result in the evaporation and formation of solid bridges. Compressive forces generated during the process will compact the material to the point of particle surface deformation and will result in the creation of large lumps (Tardos & Gupta, 1996). A study looking at the adhesion of pharmaceutical powders to stainless steel showed that longer compression time resulted in higher adhesion. (Lam & Newton, 1993).

2.5 Attrition

Two major problem areas in pneumatic conveying are the effect of pneumatic conveying on particle attrition and the problem of deposition of materials on the walls of the pneumatic conveying pipe (Taylor, 1999). Attrition is the undesired generation of fragments from particles suspended in a fluid stream, caused by collisions, mutual, fixed and mobile parts of equipment. It is an area of concern on its own, but is looked at here as it plays a role in particle deposition.

The production of fine powder can be a problem when high levels of attrition occur in a pipeline. It can result in a solid that flows well normally, but becomes troublesome as the particle size distribution is reduced through breakage. When conditions are appropriate, pneumatic conveying has the potential to cause significant degradation and the fine particles generated by this process will generally be more cohesive (Marjanovic *et al.*, 1998). Attrition is not limited to pneumatic transport and is common in a wide range of processes where particles interact with other surfaces and sufficient energy exists to cause breakage. In pneumatic conveying operations, breakage of particles can occur due to the impact of particles on bends, impact of particles on walls of pipelines, particle to particle interaction and within the feeding mechanism (Reed & Bradley, 1992).

Attrition has deleterious effect on product quality and the reliable operation of process equipment because of the changes in particle properties such as size, distribution, shape, surface area and the material characteristics (Bemrose & Bridgwater, (1987), Zhang, (1994). Increasing the amount of fines in a particulate material usually decreases the flowability and increases the tendency for caking (Kalman, 1999). Under conditions of repeated impact against a surface, the lactose particle size distribution showed a decrease in the mean particle size. The solid state structure of the lactose particles also appeared to be altered due to the impact. The change in structure was shown through examination of the particles using a Differential Scanning Calorimeter (DSC) (Bentham *et al.*, 1996). The results do not, however, clearly demonstrate that amorphous lactose formation occurred.

Various mechanisms are responsible for attrition depending on the particle mechanical properties, particle shape and mode of loading. In general, particle degradation or attrition may be classified in to two groups.

- Breakage or fragmentation occurs when the collision energy is significantly higher than that required for particle fracture. Cracks generate in the areas with high stress and then develop through the whole particle. The result is that two or more similar-sized particles are formed.
- Abrasion is the result of the collision energy being sufficient to remove small quantities of material, causing surface damage to the particles. Abrasion is characterised by a lot of fines being produced and the size distribution of the original particle changing slightly (Reppenhagen & Werther, 1998, Kalman, 2000, Bravi *et al.*, 1999, Zhang, 1994).

Attrition is mainly caused by impact or shear loads. Particles break if the impact load in a single collision is greater than the particle strength. The collision velocity, the angle of collision and the elasticity of the collision are significant factors that effect the impact load. Damage can also occur at lower impact loads if collisions occur a number of times, through fatigue (Kalman, 1999).

The variables that control the level of attrition can be loosely classified into two groups, shown in Table 2-1. The physical properties of the particles, and the external factors that the particles are exposed to during of processing. The external factors are more easily controlled in the latter stages of processing and can be adjusted as a means of influencing the level of attrition.

Physical properties	**External Factors**
Size	Shear
Shape	Loading
Surface	Temperature
Hardness	Bends
Cracks (Particle Conditioning)	Velocity

Table 2-1 Variables affecting the attrition of particles

2.5.1 Physical Properties.

How a particle hits a surface has an effect on how it breaks up, due, in part, to how the force is distributed. $NaCO_3$ particles were observed to break via fragmentation and abrasion. Cleaver & Ghadiri, (1993) showed that the prevailing mechanism was related to the particle velocity and orientation. The shape of particles influences how they hit a surface. For example, a spherical particle will impact the wall surface over a larger surface area than if the particle is jagged. In a jagged particle, impact will be focused at a small point. The larger surface area will allow the load to be distributed more evenly through the particle.

Particle size plays a role in the susceptibility of a particle to attrition, with different sized particles having varying flow patterns and exhibiting different mechanical properties. It has been observed that as particle size decreases the size of the flaws and cracks becomes smaller and the breakage strength of the particles increases markedly (Hess & Schonert, 1981). Kalman, (2000) notes that smaller particles have lower inertia and they can more easily follow the airflow streams reducing the amount of impact undergone in bends. Salt particles were looked at by Ghadiri & Yuregir, (1991); the study showed that large particles (greater then 500μm) contributed the most to the attrition. Smaller sized lactose particles exhibited lower breakage than the larger particles, when compared at the same impact velocity (Bentham *et al.*, 1998).

The last three physical properties listed in Table 2-1 are similar in that they relate to the strength of the particle rather than the geometric factors of shape and size. These factors are harder to determine than the geometric factors, as they require more than the human eye to recognise and test for. Hardness of lactose particles was measured by Bentham *et al.*, (1998) using the nano-indentation method. Cracks and surface properties require the use of a microscope to identify structural flaws in the particles. All three factors may be influenced by the previous handling of the particles.

Particles that have been exposed to mechanical handing previously are more likely to have structural defects and weaknesses. This is referred to as fatigue and can occur within a pneumatic conveying line. The repetitive impacting causes the particles to break, when conditions would not normally result in breakage occurring. Fatigue is also a design issue, as factors such as the number of bends in a conveying line influence the number of impacts that a particle will be subjected to. Fatigue appears to be one of the main effects of attrition, and design of the pipeline should attempt to limit it through simple design and avoidance of unnecessary bends (Kalman, 2000).

Previous impacts may reduce observed breakage as they cause the weak particles to break leaving only the defect free particles. In a study looking at attrition of $NaCO_3$, the level of damage observed was largest during the first three impacts. This was the result of the weaker particles breaking and in the following impacts the level of attrition became more constant (Cleaver & Ghadiri, 1993).

Work Hardening is the hardening of the surface layers through mechanisms such as pinning of the dislocations by the precipitates within the crystal or by the boundaries.

Ghadiri & Yuregir, (1991), concluded that fluidised bed drying and lean phase pneumatic conveying can cause salt particles to become harder through work hardening.

2.5.2 External Factors Affecting Attrition

Loading is a design factor in pneumatic conveying that requires attention, as the higher the loading rate, the higher the gas flow has to be to move the particles, thus increasing the velocity of the particles, an undesired factor. However, as the loading rate increases shielding can occur. Shielding is where the particles interact with each other more than they do with the wall. This effect can decrease the attrition rate. The effect of loading factor and air inlet conveying velocity on the breakage of salt particles indicates that attrition increases with low loading factor and high conveying velocity (Mckee *et al.*, 1995).

The temperature of the gas stream used for conveying can play a role in what level of attrition is observed. This is mainly due to the effect temperature can have on the particle properties. In order to reduce brittleness, operation at higher temperatures is preferred as it makes the material more ductile (Ghadiri & Yuregir, 1991). Particle properties such as strength, hardness and elasticity are effected by temperature. For any specific material there may be an optimum temperature, that allows minimisation of attrition during conveying. At low temperatures, particles may become brittle and easy to break. Higher temperatures however, may cause the particles to soften, agglomerate or melt, resulting in problems not with attrition but with caking (Bemrose & Bridgewater, 1987).

The effect of velocity on attrition has been briefly discussed above, and one general conclusion can be drawn. The higher the velocity, the higher the level of attrition. Lactose crystal breakage was found to increase in a power law relationship to the velocity of impact (Bentham *et al.*, 1998). For this reason it is desirable to keep the velocity of particles in a pneumatic conveying line as low as possible when trying to minimise the level of attrition.

Higher velocities are advantageous for other operating conditions in a pneumatic conveying line such as saltation and throughput. This requires that a trade-off is made in an attempt to provide the most desirable solution. There is not a set particle velocity that proves optimum, as the level and type of attrition that occurs is material dependent. It was found that to reduce attrition in salt particle velocities should be kept below $10ms^{-1}$ (Ghadiri & Yuregir, 1991). Complete fragmentation of $NaCO_3$ was observed for all impact velocities above $20ms^{-1}$, below this chipping but no fragmentation took place.

2.5.3 Mechanisms of Breakage

The sections above illustrate that understanding attrition, requires an understanding of the physical properties of the particles. It is also necessary, when investigating attrition, to determine the failure mode of a material, as this helps identify the conditions that are important in contributing to breakage. The characteristics of attrition are dependent on the interaction between the material properties and the process of application of the load (Zhang, 1994).

- Brittle Failure is caused by fracture with little or no plastic deformation, where the damage zone is under elastic response regime.
- Semi-Brittle Failure is characterised by plastic deformation preceeding the elastic plastic response.
- Ductile Failure is dominated by extensive plastic flow, which is responsible for the rupture of the material. (Zhang, 1994)

Studies have shown that ductile and brittle materials exhibit distinctly different erosion behaviour. Ductile materials are subject to plastic deformation and cutting by the impacting particles. Brittle materials are fractured and are more resistant to erosion when the particles impact at low angles. At high impact angles, the resilient, ductile materials are more resistant (Bodner, 1982). Angle of impact was shown by Wellman & Allen, (1995) to influence the type of failure. High angles of impact, 90° and 60° showed lateral fracture and chipping. With lower impact angles, 45°and 30°, the damage observed was predominately plastic in nature with cracking occurring only after fatigue.

As a material, lactose, when compacted, has been shown to deform by a mixed mechanism of plastic flow at the contact points as well as particle fracture (Roberts & Rowe, 1986). This type of behaviour indicates that lactose was undergoing a process of semi-brittle failure.

2.6 Conclusions

This review of the literature undertaken in this chapter has identified that the build up of lactose in a pneumatic conveying line can be influenced by two main variables; the properties of the powder, and the conditions of transportation. Particle size, amorphous content and previous handling, are properties of the powder that, have all been identified as factors that influence the rate and level of build up.

The literature review has shown that the design and manner in which a conveying system is operated can affect the level and rate of build up. Of these design and operating conditions there are two major factors which stand out. These factors are the conveying velocity and the bends; both are involved with the impact of the particles, and both need further investigation.

What happens to a particle during impact plays a significant role in the build up. The mechanical disruption of the lactose, as a result of impact, produces amorphous lactose, which leads to build up in the conveying lines. It can also exaggerate any existing properties of the powders that may lead to build up occurring.

One of the major forms of mechanical disruption to the lactose crystals that occurs during impact is breakage. The study of the effect of impact on the breakage of lactose crystals will be the focus of the next chapter.

Chapter 3 Impact Testing

3.1 Introduction

It was identified in chapter two that impact plays a significant role in the rate at which solids deposition occurs in pneumatic transport lines. The bends are typically the areas where the most buildup occurs and they are the area where most impact occurs. Increased velocity leads to increased impact force, as the energy contained by the particle is increased. It is the combination of these two elements of pneumatic conveying that is the focus of this chapter which will look at how lactose responds to various impact conditions.

3.2 Contact Mechanics

Impact occurs during the period when the lactose crystals come in contact with the pipe walls or each other. It is during this period of contact that any buildup will occur as the cohesion forces are at their greatest. Contact mechanics is concerned with the stresses and deformation that arises when the surfaces of two solid bodies are brought into contact (Johnson, 1985). It will be used here to try to provide an insight into the factors that play a significant role in the breakage that occurs when lactose is conveyed through a pneumatic transport line.

3.2.1 Deformation Mechanisms

There are two types of surface interaction that occur when two or more solids are contacted with each other, and three main mechanisms which cause loss of energy during this interaction. The interactions are adhesion and material displacement, and the energy loss may occur through the processes of elastic deformation, plastic deformation and fracture (Halling, 1978).

- Elastic Deformation is said to have occurred when a body returns to its original dimensions after having been deformed, upon the release of the stress.
- Plastic Deformation is said to have occurred when a body undergoes permanent deformation without failure.

- Fracture is the final stage and results in cracks and the removal of material. (Ivanoff, 1996)

At what point a material will enter any of the above stages is dependent on its physical properties and the type of conditions to which it is subjected. Crystalline lactose is, as discussed in section 2.5.3, a semi-brittle solid and will undergo plastic flow before fracture, where as glass is a brittle solid and exhibits almost no plastic flow before fracture.

3.2.2 Properties of Materials

Two main elastic properties of a material are used when looking at the contact of two surfaces; these are the modulus of elasticity and Poisson's Ratio.

The modulus of elasticity or Young's Modulus (E) is defined as the ratio of direct stress, (σ), to the strain, (ε), produced (Ryder , 1977).

$$E = \sigma / \varepsilon$$

Equation 3.1

As stress is the force per unit area and strain is the ratio of the deformation , it can be seen that the higher the Young's Modulus of a material the greater the resistance it has to deformation under a load.

Poisson's ratio, (v), is shown in Equation 3.2 to be the ratio of lateral strain, (ε_{lat}), to longitudinal strain, (ε_{long}), produced by a single stress.

$$v = \varepsilon_{lat} / \varepsilon_{long}$$

Equation 3.2

The value of Poisson's ratio for a material is relatively constant under conditions of elastic deformation, under plastic deformation the value of Poisson's ratio becomes 0.5 (Ryder , 1977).

3.2.3 Hertzian Contact Theory

An impact occurs when two or more bodies collide. It is characterised by the generation of relatively large forces at the points of contact for a relatively short period of time. These forces are referred to as impulse forces (Hoppmann, 1995). In order to calculate how large the force is, it is necessary to know the length of the time, (t_I), over which the impact occurs. The average force, (F_{av}), can be calculated from the momentum, (J), using Newton's second law, shown in Equation 3.3 (Young & Freedman, 1996).

$$F_{av} = \frac{J}{t_I}$$

Equation 3.3

The time period over which a collision takes place can be solved using the Hertz theory of impact. Hertz's theory relates the compressions of the two surfaces and the forces between them, to the radii and elastic properties of the two bodies.

The time of contact t_I is shown in Equation 3.4 to be equal to the α value calculated in Equation 3.5 using Young's modulus, (E), the Poisson ratio, (v), the mass of the particle, (m), and the particle radius, (r), divided by the velocity, (u), (Hoppmann, 1995).

$$t_I = \frac{\alpha}{u}$$

Equation 3.4

$$\alpha = \left[\frac{15}{16} v_1^2 \left(\frac{1-v_1^2}{E_1} + \frac{1-v_2^2}{E_2}\right) m\right]^{2/5} r^{-1/5}$$

Equation 3.5

The theory also allows the change in momentum, (J), on impact to be calculated using Equation 3.6.

$$J = mu(1+e)$$

Equation 3.6

Where e is the coefficient of restitution calculated as shown in Equation 3.7

$$e = \frac{h\,\rho_1\,a^2 - 0.56m}{h\,\rho_1\,a^2 + 0.56m}$$

Equation 3.7

a equals the area on the plate over which the disturbance is observed; this is calculated using Equation 3.8.

$$a^2 = \pi\,t_l\,h\sqrt{\frac{E_1}{3\rho_1 * (1 - v_1^2)}}$$

Equation 3.8

where h is equal to half the thickness of the impact surface.

3.2.3.1 Limitations of the Theory

The theory has limitations when applied to this work. The main one is, that it was formulated to deal with elastic impacts. After a certain force is reached in a collision, elastic flow will cease and, depending on the properties of the solid, either plastic flow or fracture will occur. As lactose is a semi-brittle solid, failure will be preceded by plastic deformation.

Plastic flow reduces the intensity of the contact pressure pulse and adsorbing energy (Johnson, 1985). Fracture adsorbs the kinetic energy and dissipates it as heat. This is illustrated by the high temperature rises in the fracture of sugar crystals observed by (Fuller *et al.*, 1975).

Another limitation of the theory is that it assumes that the two surfaces involved in contact are perfectly smooth and continuous. In consequence the stresses are finite everywhere (Johnson, 1985). Distortions in the surfaces provide a localised area of contact that becomes subjected to higher forces than the rest of the body, making it more prone to breakage. This is the case with lactose crystals, which have imperfections in their surfaces. This is shown in Figure 3-1 where even a smooth crystal has raised points where localised contact can occur The surface of a lactose crystal is also not continuous like the spheres dealt with by Hertzian theory.

The tip of the tomahawk shaped crystal, like that shown in Figure 3-1, forms a wedge. In the case of a wedge, the contact pressure at the apex becomes theoretically infinite. It must not be assumed that even a light load will cause deformation, as on impact there will be localised plastic flow that will allow the stresses to become more evenly distributed (Johnson, 1985). This concept is of little importance in this work, as it has already been concluded that as fracture is occurring plastic flow will also be proceeding. The reason for the discussion of Hertz Theory here is to provide an illustration as to how velocity relates to impact.

Figure 3-1 Washed Lactose Crystal

What the theory does show is that the force on impact under elastic conditions will increase as a function of the velocity squared. This is illustrated by rearranging Equation 3.1 and Equation 3.4 to give Equation 3.9.

$$F_{av} = \frac{mu^2}{\alpha}$$

Equation 3.9

3.3 Impact Testing Methods

In looking at the effect impact has on lactose crystals it is necessary to use a method that can simulate the conditions in a pneumatic conveying line closely. Yet, at the same time, allow individual parameters to be studied with out the complexity created by other interactions.

A lot of the work looking at impact testing has its focus based around particle breakage or attrition. It was discussed in chapter two that there is a link between the attrition of particles and buildup.

Testing can be divided into two areas:

- Single particle testing
- Multiple particle testing

A review of the types of tests, which look at particle breakage, has been carried out by Bemrose & Bridgwater, (1987) and also by Zhang, (1994).

Multiple particle testing provides a more realistic representation of the processing conditions experienced during pneumatic conveying, as it allows the interparticle interactions to proceed to at least some degree. The major problem with using multiple particle testing is that the number of interactions that occur make the results obtained complex for analysis (Zhang, 1994).

Single particle testing reduces the complexity of the system, and allows individual particles to be studied under more controllable conditions. This permits the effect of variations to the system to be observed more easily. However, in a pneumatic conveying system factors such as the interparticle interactions are unavoidable and are likely to play a role in breakage and smearing in the lines. This means that to get a true representation of how different factors influence the level of deposition, the results obtained from single particle test need to be compared with those obtained in a pneumatic conveying system.

3.3.1 Single Particle Testing

Two main test methods can be used to examine the breakage behaviour of single particles. These two testing methods can be divided into impact testing and compression testing.

- Compression testing is used to measure the load bearing capabilities of particles. It involves applying a gradual force to the particle and observing how the particle behaves.

- Impact testing shows how a particle will behave when subjected to an impulse force.

It is the latter that more closely represents the conditions in a pneumatic conveying line. Single particle impact testing will be used as the initial method for studying how varying different factors influences the breakage of lactose crystals.

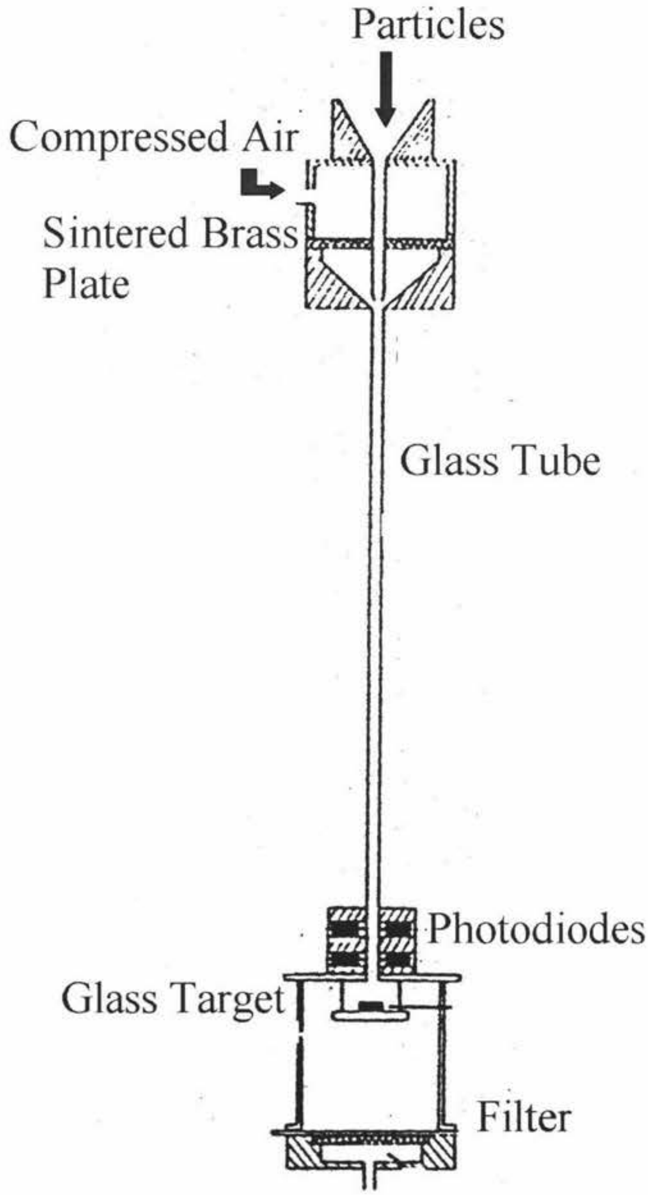

Figure 3-2 Impact testing device

3.3.2 Testing Device

The impact-testing device used in this work was built and based on the design described and used by (Yuregir *et al.*, 1986). This device is illustrated in Figure 3-2.

This impact-testing device allows the impact velocity of individual particle to be measured.

The velocity of a particle was measured using two sets of three photodiode sensors 10mm apart. These were mounted just above the target plate in order to get an accurate reading of the impact velocity. The mounting for the sensors is shown in Figure 3-3. A set of photodiodes are opposite an infrared light source. When an interference of the light source occurs each set of sensors is designed to generate a five-volt pulse. This triggers a 1MHz timing device. The time that occurs between each pulse is measured and from this the velocity of the particles was calculated.

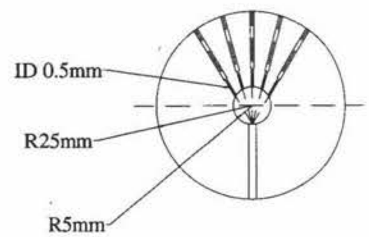

Figure 3-3 Sensor Housing

The particles are accelerated down a one-metre glass tube using compressed air. The funnel at the top is used for feeding the particles. The funnel has a tapered end and is positioned at the entrance to the main glass tube. A narrowing at the entrance to the glass tube, generates a pressure drop due to a change in velocity. The vacuum created allows the particles to be feed in to the device. The target plate is mounted in the chamber at the end of the glass tube. This is removable to allow different materials and angles to be used in the testing process. The collection chamber consisted of an aluminium top and base separated by a polycarbonate cylinder. The particles are removed from the air stream by passing the air through a sintered bronze plate at the base of the chamber.

3.4 Measuring Breakage

To measure the effect that the different conditions created in the impact-testing device, have on the attrition of lactose, a method for quantifying each experiment was required.

3.4.1 Sieve Analysis

In order to quantify how much breakage was occurring after each impact test a sieve analysis was carried out. The lactose crystals were collected for each run and the total mass of crystals impacted [M_{BS}] was determined by weighing them on a balance. The crystals were then hand sieved through a sieve with an aperture halve the size of the smallest sieve used for generating the initial size fraction. i.e. for the crystals in the 425-500μm size range a sieve of 212μm was used and for the 212-350μm crystals a 106μm sieve was used. The crystals were then re-weighed to determined the mass left after sieving [M_{AS}]. The percentage difference in weights was then calculated as shown in Equation 3.10. This number was then used as a method of determining how much breakage had occurred.

$$\%Difference = 100\left[\frac{M_{BS} - M_{AS}}{M_{BS}}\right]$$

Equation 3.10

The method is based on one used by Arteaga *et al.*, (1996). The variation between the two is that in Arteaga's method a sieve two sizes smaller was used.

3.4.2 Crystal Preparation

In order to study the amount of breakage that occurs when lactose crystals undergo impact it is necessary to prepare the crystals so that only the mass of crystals being broken is measured. The lactose crystals were sieved out into selected size ranges from commercially available lactose crystals, supplied by Lactose New Zealand.

These crystals were then washed with distilled water to dissolve any surface debris. This debris would have provided fines that may have become detached during the final sieve analysis and provided a source of error. After washing with distilled water, the crystals were washed with ethanol to prevent them forming liquid bridges and caking. The crystals were then dried using a vacuum filter to remove any liquid.

After washing and drying, the crystals were then resieved to remove any crystals that the washing process had reduced to below the desired size range. The two stages of before washing and after washing can be seen in Figure 3-4 and Figure 3-5. The images were taken under polarised light. In the "before" image, Figure 3-4, small fines are attached to the surface of the crystal. These are not present in the "after" image, Figure 3-5.

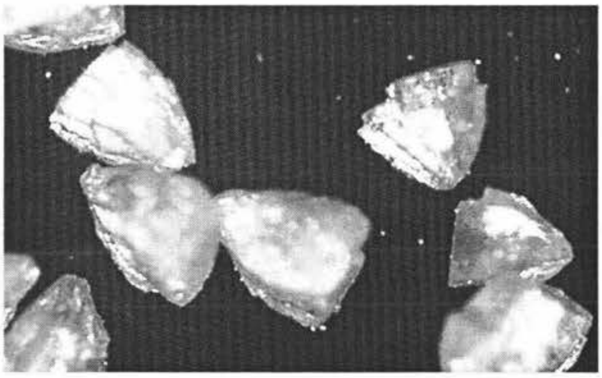

Figure 3-4 Lactose Crystals Before Washing

Figure 3-5 Lactose Crystals After Washing

3.5 Velocity Testing

In a pneumatic conveying line, forces act on each particle of a bulk material being conveyed. These forces, caused by kinetic energy, are expressed in terms of friction, pressure and thrust, act among the particles and between the particles and the pipe walls (Klinzing *et al.*, 1997).

Increasing the velocity of particles travelling through a pneumatic conveying system has the effect of increasing the amount of kinetic energy of each particle by the power of two. This increased quantity of energy results in higher impact forces acting on the particles. The increased energy is also available for release in other forms of energy such as heat, when a particle undergoes an impact against another surface.

The increased velocity also provides the particle with a greater amount of momentum. This means that when the particles collide with other surfaces the average force observed on impact increases. This increased force places more stress on the crystals and increases the chances of failure or breakage occurring. All of this is shown by the Hertzian theory above.

To test the influence of velocity on the breakage of lactose crystals the impact-testing device was used. Lactose crystals in two size ranges, 425-500μm and 212-350μm, were prepared as described in section 3.4.2. The crystals were then feed individually into the impact testing device and fired against a glass plate at velocities ranging from 12m/s to 30m/s. The particles were then collected and the amount of breakage determined.

Literature shows that, increasing the particle velocity in a conveying line increases the rate at which breakage will occur. In the case of lactose crystals, work done by Bentham *et al.*, (1998) showed that the loss per impact varied with velocity to the power of 2.4-2.7.

The results obtained from the experimental work completed here show a high level of agreement with the work done by Bentham *et al.*, (1998). The results can be seen in Figure 3.6, where the 550 and 350μm particle data is from Bentham *et al.*, (1998). It is observed that the slopes of the data sets are almost parallel to each other. This indicates that the pattern of breakage is consistent with what is reported by literature.

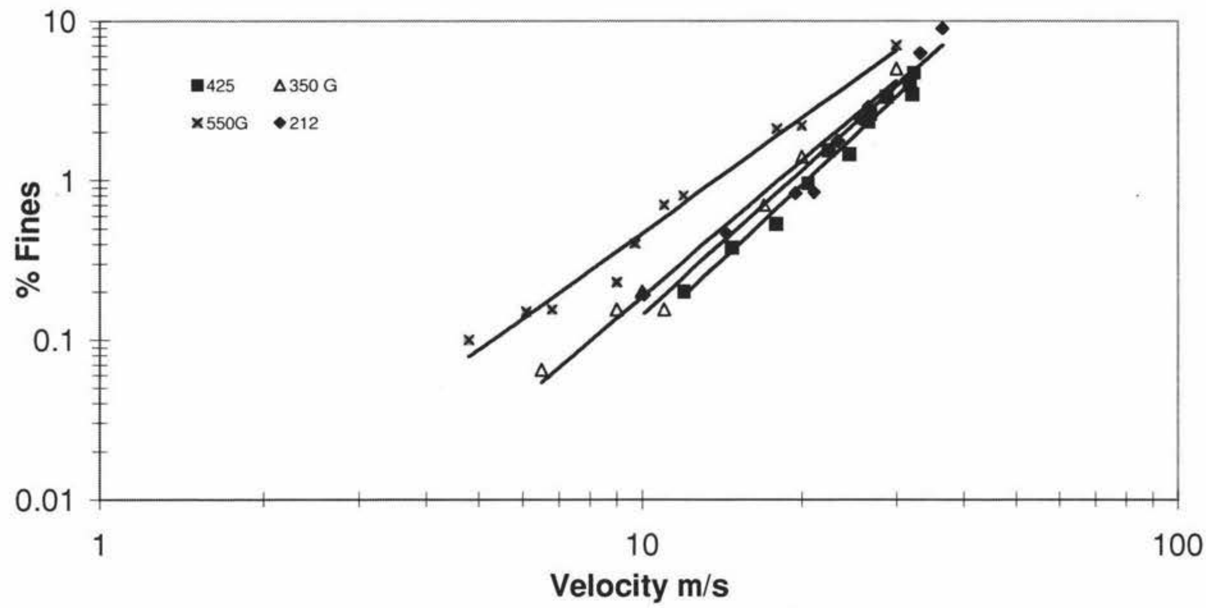

Figure 3-6 The Effect of Velocity on Breakage

(G =Data from (Bentham *et al.*, 1998))

3.5.1 Variations from the expected results

The experimental results vary from what is predicted by the contact theory. It was discussed how the amount of kinetic energy increases with velocity by the power of two. This squared relationship is also the rate that the Hertzian contact theory calculates the increase in the size of the impact force.

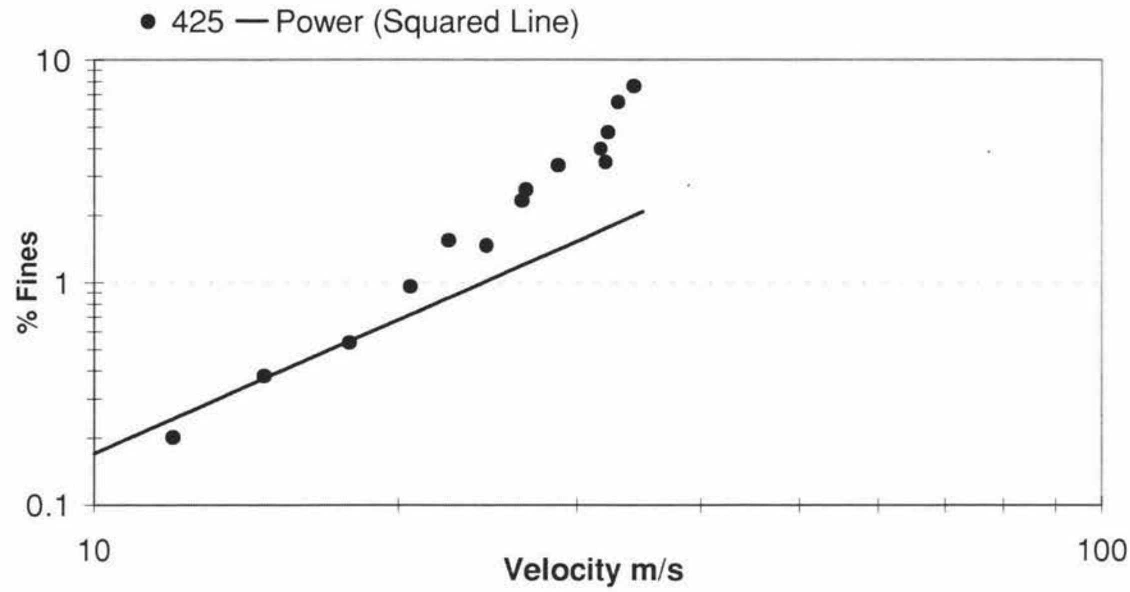

Figure 3-7 The effect of velocity on the breakage of 425μm lactose crystals fitted to a squared line.

Figure 3-7 shows the experimental data collected from the 425μm particles alongside a line with the slope to the power of two. The line has been drawn in to fit the data as closely as possible. It is observed from this, that up until about 20m/s the experimental data follows the expected trend, but above this a steeper slope is observed..

An explanation for this deviation from the predicted results is that above 20m/s there are two mechanisms of attrition occurring. These two mechanisms are abrasion and fracture; these were discussed in chapter two.

- Abrasion is the removal of fines causing only surface damage to the particles and requires only small quantities of energy.
- Fracture or breakage occurs at higher levels of collision energy and results in two or more similarly sized particles being formed.

Above 20m/s both fracture and abrasion are occurring and below 20m/s only abrasion is occurring. Once fracture starts to play a role the rate at which the weight fraction of fines collected increases. This increase in weight is due to the larger particles coming through in the sieve analysis.

To check this mechanism, a particle size analysis was carried out on four sets of samples from the velocities 0, 20, 25 and 30m/s using a Malvern Mastersizer. The particles were impacted against the glass plate as described above but rather than sieving for the fines the whole sample was run through the Malvern Mastersizer.

It can bee seen in Figure 3-8 that for the zero velocity impact the distribution is very narrow and no fines are present. In Figure 3-9, impact at 20m/s, there is a mass of fines outside the main distribution. These are randomly spread and are what would be expected when abrasion is occurring. At 25m/s, Figure 3-10, there are particles in the size range about half the original particle size. This compares with the previous two distributions that had no particles in this range. In Figure 3-11 30m/s impact, the distribution has become wider and the fines present in the 20m/s impact are present here. In Figure 3-11, a second peak on the distribution is also apparent. The particles in this second peak are about quarter the size of the original particles, showing that large pieces of original crystal have been fractured off. This is similar to the results in Figure 3-10 at 25m/s, but the peak has becomes more defined, due to the increased amount of energy available, resulting in a higher amount of fracture.

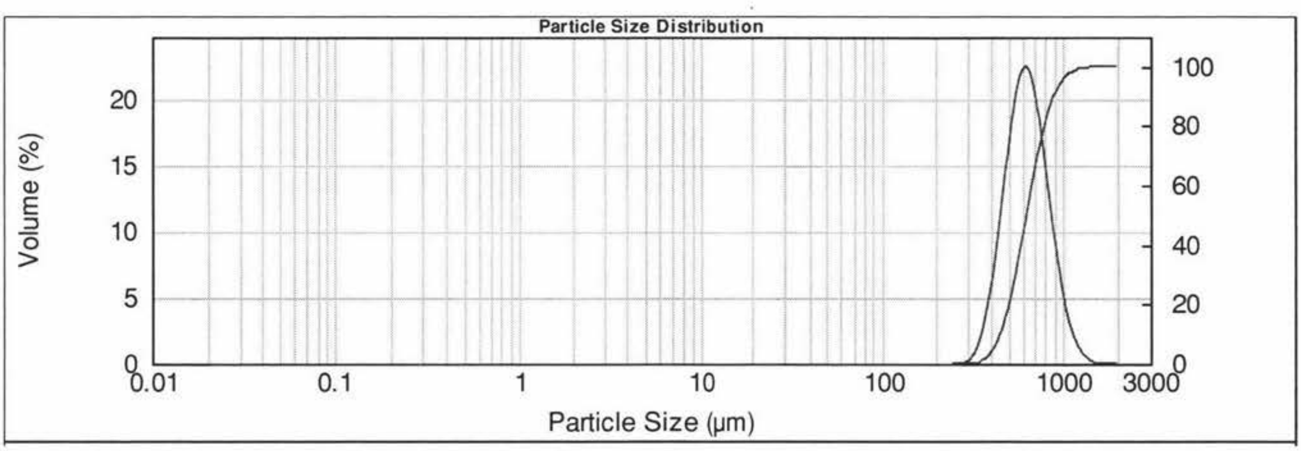

Figure 3-8 Particle Size Distribution from 0m/s

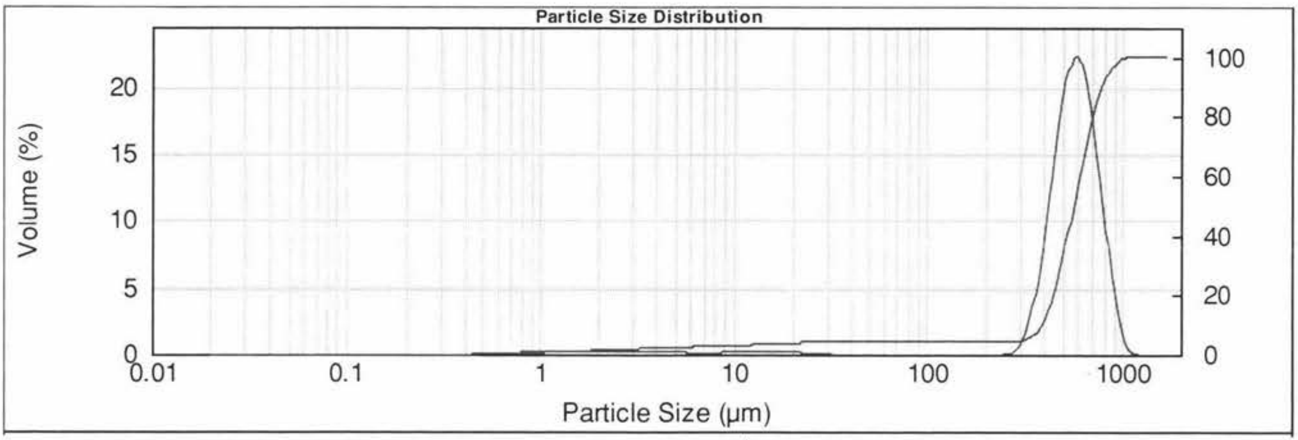

Figure 3-9 Particle Distribution from Impact at 20m/s

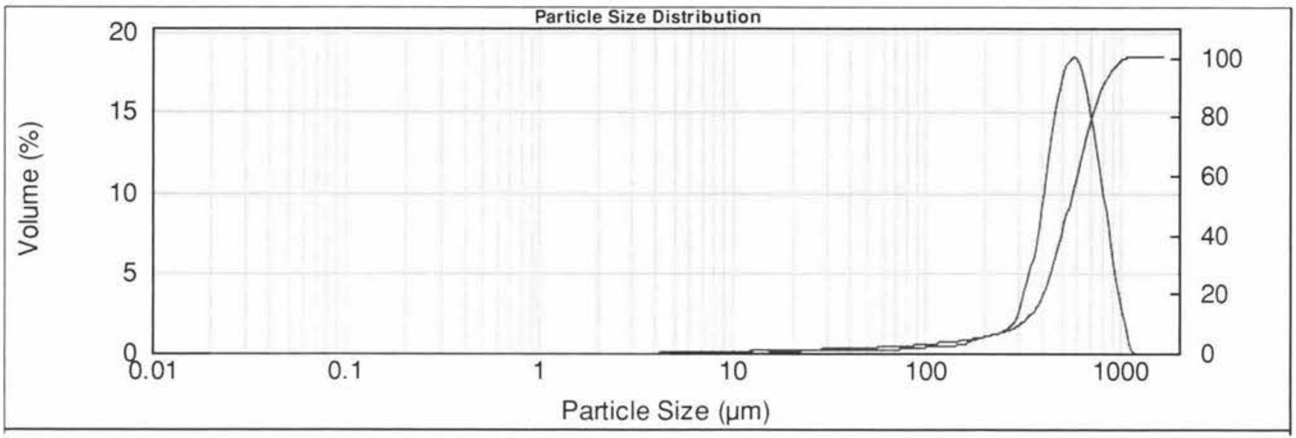

Figure 3-10 Particle Size Distribution from Impact at 25m/s

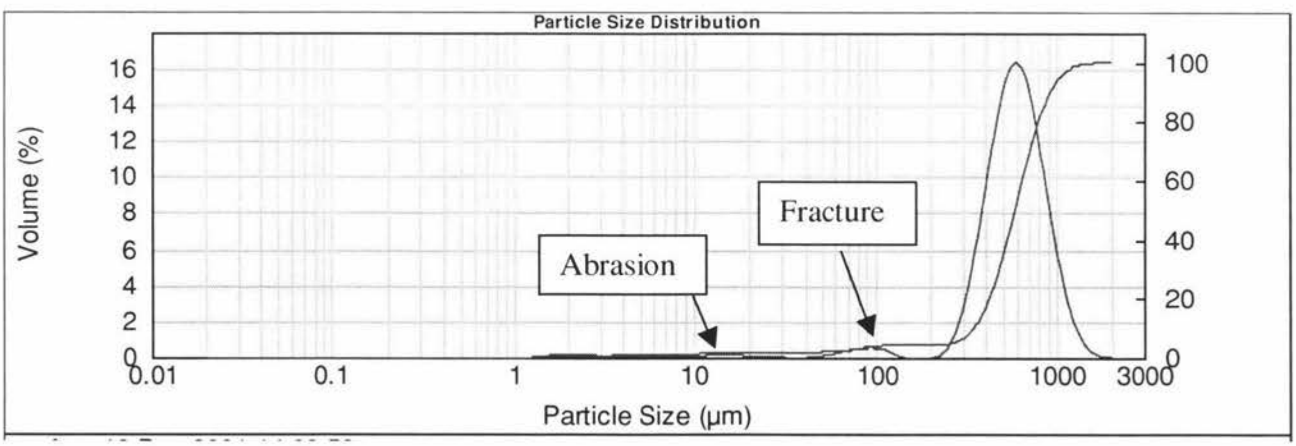

Figure 3-11 Particle Size Distribution from Impact at 30m/s

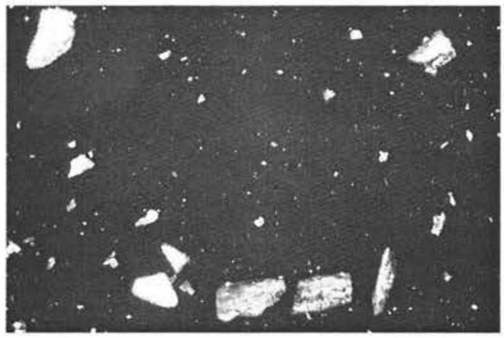

Figure 3-12 Image of the Fines from 25m/s Impact

The results shown by the particle size distributions are further illustrated by Figure 3-12, which shows the fines collected from the sieve analysis after impact at 25m/s. It can be seen in the image that two types of fines are present, large particles from fracture and small particles from abrasion.

At the velocities lower than 20m/s the impact is more likely to be elastic in nature and thus fit with the Hertzian theory. Once fracture begins to occur, as has been discussed in section 3.2, the energy becomes dissipated through both plastic flow and released in crack formation. Where this energy dissipation does occur it might be expected that the slope of the line above 20m/s should be close to four, the sum of both the losses from fracture and abrasion. It is instead 3.6, the reduction resulting, it is assumed, from the energy losses due to plastic flow and fracture.

It was observed by Hess & Schonert, (1981), that sucrose particles showed a higher fracture strength than quartz particles of similar size. This phenomenon was explained by the fact that quartz is a brittle solid and under impact exhibited little or no plastic deformation. The sucrose, however, is like lactose, semi-brittle in nature and under impact, fracture was preceded by plastic flow. This increased the contact area of the crystal and thus reduced the internal stresses of the crystal.

3.6 Particle Size Effects

It was shown in section 3.2.3 that the average force a particle is exposed to on impact is a function of its momentum and the impact time. As momentum is a function of mass and velocity, it is predicted that smaller particles will exhibit less breakage than a larger particle when conveyed at the same velocity due to the reduction in impact force, assuming impact time stays the same.

The reduced particle size mean smaller flaws in the particles. Higher stresses are therefore needed to satisfy the differential energy conditions for crack releasing (Hess & Schonert, 1981).

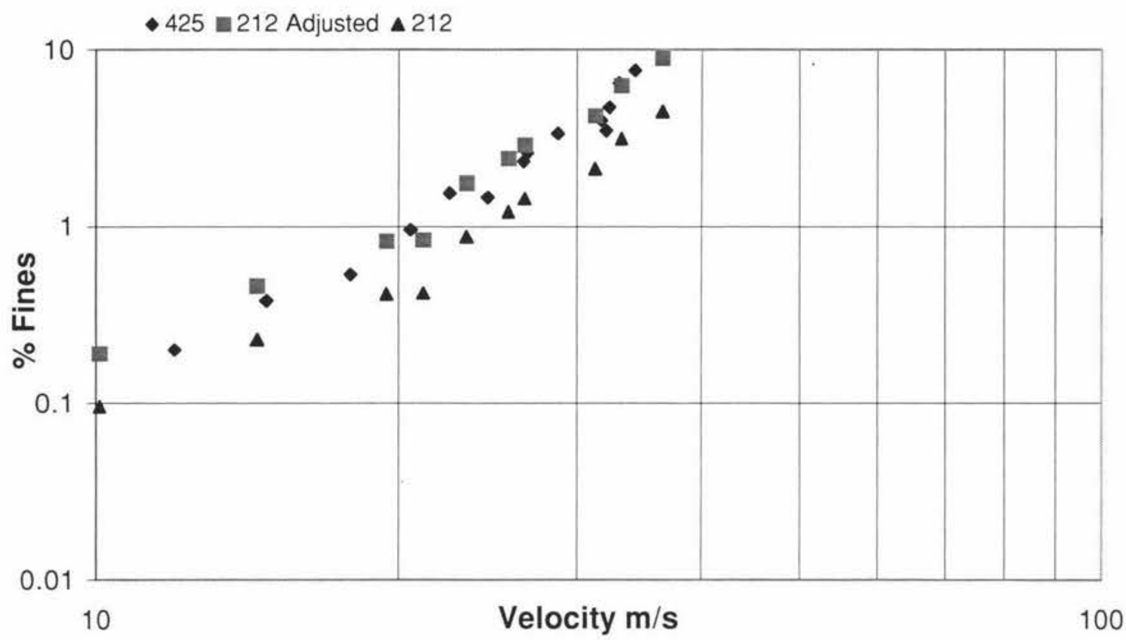

Figure 3-13 The relationship between the attrition observed for the 425μm and the 212μm particles, with the 212μm particles adjusted to account for the difference in size

Looking at the work done by Bentham *et al.*, (1998), and also the experimental work done here, shown in Figure 3-6, the breakage is linearly proportional to the diameter of the particles.

The amount of breakage shown by 212um particles is multiplied by two it shows a close similarity with the results obtained for 425μm particles. This is illustrated by the results in Figure 3-13, where three data sets are shown. The particle breakage data collected for two size rages of crystals, 425-500μm and 212-250μm, and also the 212-250μm data multiplied by a factor of two (425/212). The adjusted data fall closely along side the results obtained for the 425-500μm crystals, whereas the actual 212μm data falls below it.

This relationship can be explained if the particles are assumed to be spherical. The kinetic energy contained by a particle at a set velocity is proportional to its mass. The mass of a particle has a cubic relationship to its diameter. Therefore, as the diameter increases, the energy changes to the cube of the diameter. However, the area over which the impact occurs increases to the power of two. These two relationships cancel out, with the result being that the breakage observed varies linearly with the diameter of the particles, as is seen in Figure 3-13 .

The change in mechanism of attrition for the 212-250μm particle occurs between 20-25m/s per second. Examination of the data in Figure 3-13 shows that it is in this region that a change in slope occurs. At this point, the 212-250μm particles also follow the 425-500μm particles more closely in the amount of fines produced, when corrected for size.

3.7 Angle of impact

Section 2.3.3 discussed how bends are one of the main problem areas influencing the quantity of breakage and buildup that occurs in pneumatic conveying lines.

There are typically three types of bends used in pneumatic conveying, the long radius bend, the short radius bend and the blinded tee. The variation in design of the bends is in the angle at which the particles hit the surface and in the case of the blinded tee, the level of cushioning that is provided.

One of the main aims of this work is to reduce the level of buildup that occurs in conveying lines. Part of doing this requires an understanding of what conditions effect the breakage of lactose crystals. Studies looking at the erosion of conveying pipe walls showed that the way in which the impact angle effected the level of wear observed was material dependent (Bodner, 1982).

It is presumed that the effect of impact angle on breakage will also be material dependent. This makes it necessary to test how lactose behaves when it undergoes impact at various angles. Through knowing what the effects of various impact angles are an assists in determining the most suitable bend type to use when conveying lactose, in order to minimise the breakage and buildup.

Tests were carried out looking at three angles 90°, 45° and 60° to see how these angles influenced the breakage of lactose. The tests used lactose crystals in the size range of 425-500μm. These were prepared with fines removed using the method described in section 3.4.2. The crystals were then fired at varying velocities, at a glass plate mounted on a aluminium block, cut to the desired angle. The amount of fines produced was determined using the sieving analysis described in section 3.4.2.

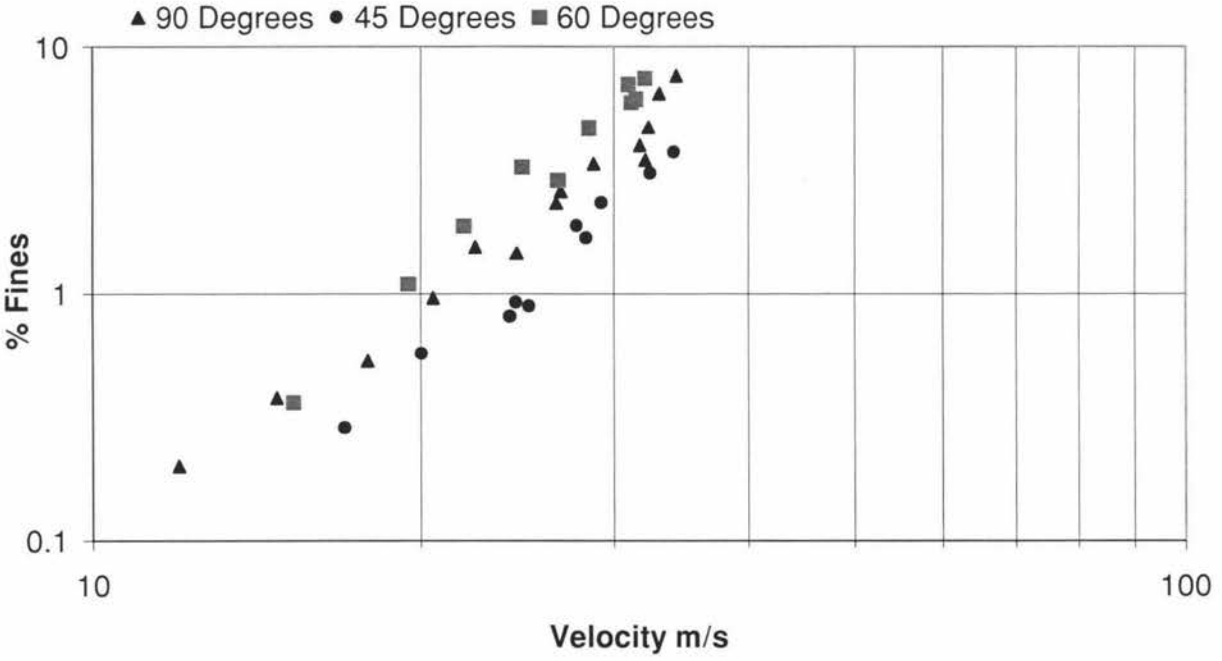

Figure 3-14 Angle of impact results

3.7.1 45 Degrees

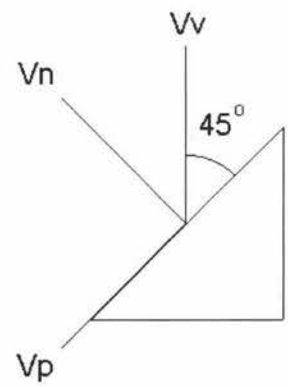

Figure 3-15 45°: The normal (Vn) and parallel (Vp) components of the vertical velocity (Vv)

The results obtained for the angle of impact testing are displayed in Figure 3-1. It can be seen from this that the breakage for the 45° impact is lower than is shown for the head on, 90° impact. This result can be explained through looking at that the way the velocity is distributed upon impact. The velocity has been split into two components, the normal component and the component parallel to the plate. These are shown in Figure 3-15. The normal velocity component has the major effect on breakage, as this is the direction an opposing force is provided.

The amount of force that is being applied to the particles upon impact is dependent on the velocity in the normal direction. This velocity is equal to the vertical velocity times the sin 45° (the angle of impact). Figure 3-16 shows the 45° and the 60° results fitted against the 90° results after the velocity has been multiplied by the sin of the angle of impact. The 45° data now lies above the data for a 90° impact. It can be concluded from this that it is not only the normal impact that is causing breakage.

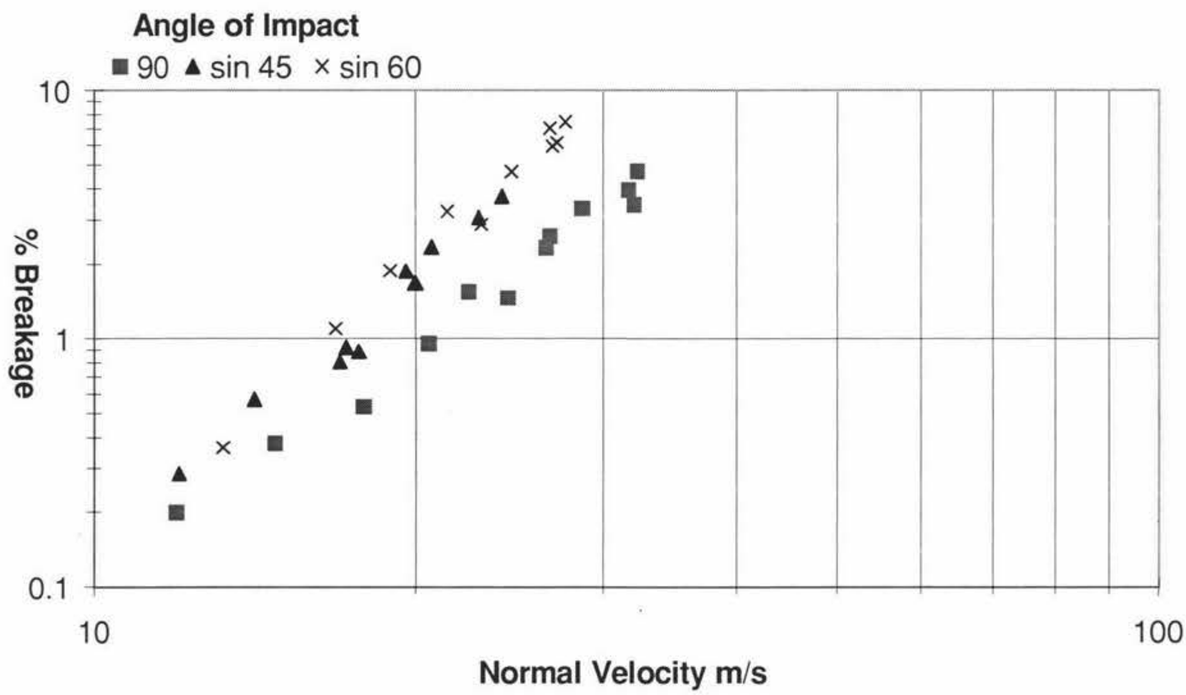

Figure 3-16 Results adjusted for the angle of impact

3.7.2 60 Degrees

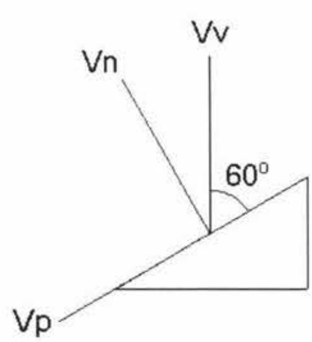

Figure 3-17 60°: The normal (Vn) and parallel (Vp) components of the vertical velocity (Vv)

Figure 3-14 also shows results collected for impact against a 60° slope. These results for the 60° do not fit with the concept that the breakage is reduced due to a component of the velocity being projected in the direction parallel to the plate. The results show that the breakage is higher at 60° than for a straight on impact. The trend-line moves further away from the 90° slope, as the velocity is increased.

Figure 3-16 also shows that adjusted data for the 60°. The data for the adjusted 60° impact and 45° impact to follow almost the same line. It is concluded from this that the same phenomenon is causing the particle breakage to be higher than predicted from the normal component of impact. This result is attributed to secondary impact and is discussed in the next section.

3.7.3 Secondary Impact

When particles collide with an angled target not all the force is directed in a normal direction. A percentage of it is directed parallel to the plate. This results in the particle rebounding with a significant horizontal component. In the case of the device used in this work, the distance between the centre of the impact target and the walls of the collection chamber was 0.04m . The closeness of the wall and the fact that the particles are being projected off the target with a horizontal velocity component, means there is potential for secondary impact, causing further breakage to occur.

In tests using 45° and 60° targets, an area on the clear polycarbonate used as the housing to the collection chamber, had a smearing of powder on it. This was not observed with the 90° target. In addition to the smearing, particles in the base of the collection chamber built up predominately on the opposite side the target would have directed them. These two factors indicate that the particles were hitting the target, then rebounding to hit the collection chamber wall. This double impact may have resulted in a higher level of breakage than would have been observed had the particles only under gone the impact against the target.

The impact of NaCl crystals showed the formation of subsurface cracks; these formed during the unloading phase from the elastic/plastic deformation (Yuregir *et al.*, 1987). These cracks would provide weak areas on the crystals so that it would become easier for attrition to occur in subsequent impacts. For lactose crystals impacting at 60° and 45° angles, breakage would be higher than predicted using a force balance for a single normal impact. As these crystals would experience damage from two impacts and from the unloading stage. Whereas those crystals that are impacted at 90° do not have a secondary impact, where the damage from the unloading phase can be exploited. This provides an explanation for the adjusted breakage values in Figure 3-16 being higher than the values for a single normal impact.

To test to see if the secondary impact was indeed resulting in higher levels of breakage a piece of soft rubber was glued on the wall of the chamber where the smearing was observed. Three impact tests were then done with the rubber lining. The data were then plotted in Figure 3-18, with the velocity corrected to the normal velocity. The rubber wall data fits with the 90° impact results, demonstrating the effect of secondary impact.

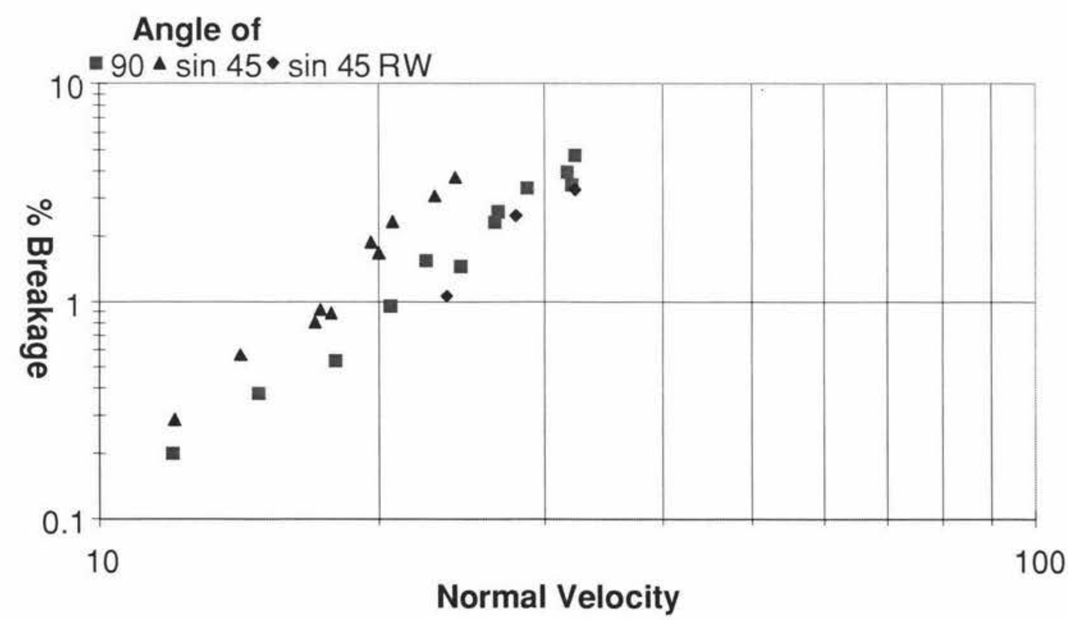

Figure 3-18 Test for secondary impact on 45° Impact (Corrected for Normal Velocity)

The effect of secondary impact on breakage was also significant when particles were impacted against a 60° target. Data for velocities at approximately 30m/s shows that

that addition of a rubber lining on the collection chamber reduced the level of breakage considerably.

Velocity m/s	Percentage
30.9	7.04
32.0	7.4
31.6 (R Wall)	6.1
32.1 (R Wall)	5.9

Table 3-1 Results for 60° Impact: demonstrating the effectiveness of a rubber lining

Table 3-1 shows that the addition of a rubber lining to the wall of the collection chamber reduced the amount of breakage observed by about one percent, which corresponds to 15% of the total crystals fines collected after impact. This suggests that the particles were rebounding off the target plate and impacting with the wall with a force high enough to result in further breakage occurring. The notion that lactose was colliding with the wall after impacting on the target plate was further supported by a distinctive area of buildup on the wall. This was in the direction that the plate would have projected the particles.

These results from the impact angles reveal that not only is it important to reduce the amount of energy contained in a particle on impact when trying to reduce breakage. It is also important to reduce the number of collisions that lactose crystals under go. This conclusion poses an interesting result in terms of bend design. The best bend may, not be the one that give the smallest the initial impact, but instead being the one that exposes the particles to the least number of impacts. Both the normal impact velocity and the number of impacts are important, and bend design aimed at reducing the normal impacts, has the potential to increase the number of impacts.

3.8 Impact Surface Effects

It was shown by Shipway & Hutchings, (1993) that fracture of lead glass spheres was influenced by the material against which they impacted. Hard, stiffer targets required lower impact velocities to cause fracture, than soft targets. This observation can be explained using the contact theory discussed above and outlined again briefly below.

Hertzian Contact theory can be used to show that contact time in a collision is a function of the elasticity of the two materials. It shows that when a collision occurs, the more elastic a material, the greater the contact time between the two objects. The impulse-momentum theorem states that: The change in momentum of a body during a time interval is equal to the impulse of the net force acting on the body during that interval (Young & Freedman, 1996). This means that when the time of impact is longer the maximum force experienced by the object involved in the collision is less. An Illustration of this is provided in Figure 3-19.

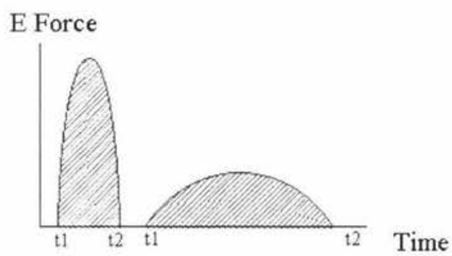

Figure 3-19 How Impulse Force is affected by Contact Time

Rubber has a modulus of elasticity in the order of 0.02GPa. This compares with glass or steel which have modulus of elasticities of 60-120GPa. Thus the damage observed upon impact of a lactose crystal against glass should be significantly higher than when the object was impacted against a rubber surface. The applicability of Hertz's theory of impact to the case of rigid spheres colliding with rubber was studied by Southern & Thomas, (1972) and agreement was found over a range of severities of impact.

Rubber also has the useful attribute that it is able to dissipate energy thermally during stretching, a phenomenon known as elastic hysteresis. During the elongation of rubber, the segments of the chain molecules slide against each other. As a consequence of the interaction of adjacent molecules frictional heat is developed. This is the irreversible part of the energy of stretching , that is hysteresis (Houwink & De Decker, 1971). Some rubbers exhibit large irreversible energy losses due to elastic deformation, elastic hysteresis (Halling, 1978) . This means that when impacted against the rubber surface the particles rebound with a lower energy level than before impact. This reduces the potential effect of secondary impact, that may occur after the initial collision.

To determine whether the rubber could be used to reduce the breakage of the lactose, crystals of in the size range of 425-500μm were subjected to a range of velocities using the impact testing device. The lactose crystals were impacted at 90° against a Nitrile rubber disc, 25mm wide and 4mm thick mounted on a glass plate. The preparation of the crystals before the impact test, and then the analysis for breakage was the method used previously, described in section 3.4.

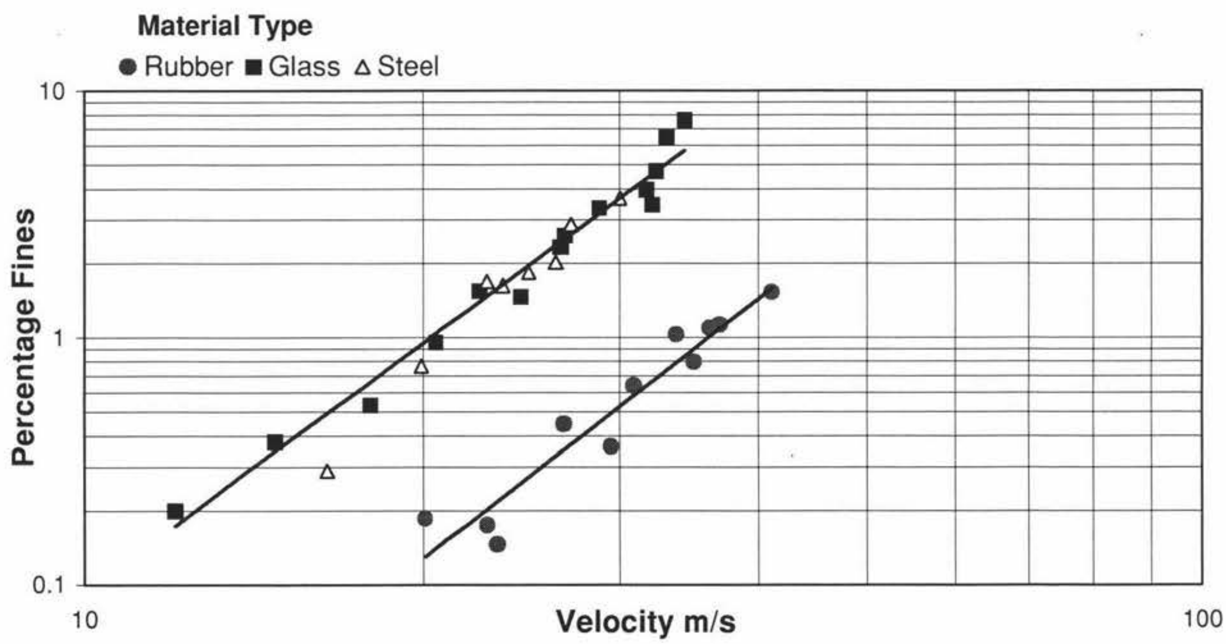

Figure 3-20 Impact Surfaces

The results obtained are shown in Figure 3-20. Data is shown for three surfaces, rubber, glass and stainless steel. It can be seen the breakage pattern for glass and stainless steel is almost identical. This is consistent with what was predicted by Hertzian contact theory. Table 3-2 shows the contact time and average force for the three surfaces, predicted using the Hertzian contact theory, when spherical 400μm particles travelling at 25m/s are impacted against them. The data generated for velocities ranging from 5 to 35m/s are shown in Figure 3-21. This also shows that the force a particle is subjected to at impact is almost identical for stainless steel and glass.

Material	Young's Modulus	Poisson Ratio	Contact Time	Average Force
Glass	60 GPa	.23	$1.68*10^{-8}$ s	157 N
Rubber	0.016 GPa	.48	$2.49*10^{-7}$ s	10 N
Stainless Steel	220 GPa	.28	$1.93*10^{-8}$ s	137 N

Table 3-2 Force at 25m/s

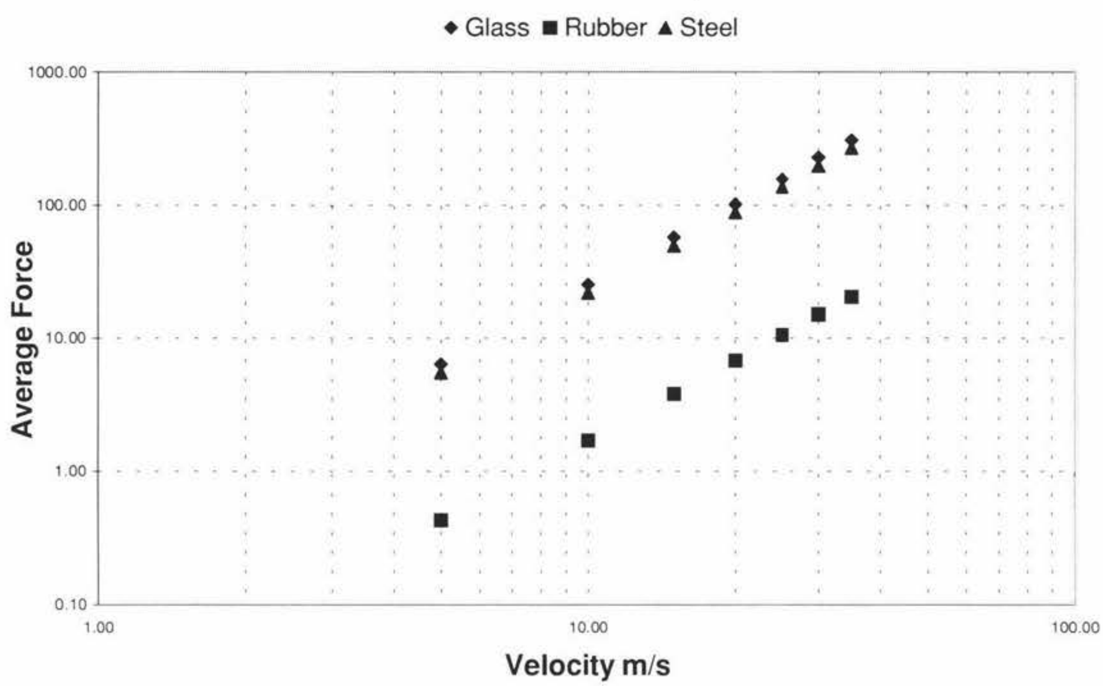

Figure 3-21 The effect of material properties as predicted by the Hertz calculation

The breakage pattern shown for rubber follows the slope shown for the other two substances, but the amount of breakage is reduced by a factor of approximately 10. This same pattern is seen in the predicted curves, shown in Figure 3-21. The experimental reduction is lower than the factor of fifteen to be the reduction in force due to the increased time over which the impact occurred, calculated using the Hertzian contact theory. The difference in the observed and predicted results is possibly due to the fact that rebound occurred. As only the target plate was covered with rubber, when secondary impact occurred it was with one of the other surfaces in the collection chamber, all of which have a much higher Young's modulus than the rubber used, causing secondary impact abrasion or breakage.

3.9 Pneumatic Conveying Test

In section 3.3 it was stated that single particle testing is limited in terms of how the results obtained can be interpreted for pneumatic conveying. Interactions such as particle-particle contact and the repeated loading-unloading that occur as particles contact the wall, are more difficult to study using a single particle testing device. To allow a comparison to be developed, between the single particle testing device and a pneumatic conveying line, experimental work was carried out looking at attrition of lactose on a small scale pneumatic conveying line.

3.9.1 The conveying line used

The equipment used was located at Industrial Research Limited (IRL), Lower Hutt, New Zealand. A diagram of the system can be seen in Figure 3-22. It consists of three straight sections of conveying pipe, each 5m long and 0.025m in diameter; two horizontal and one vertical. The system was run on positive pressure, with air flow being provided by a Nu-Con Blower. The flow was controlled by a bleed valve, located before the entrance to the conveying line.

To change the direction of the flow, two short radius bends were used. The powder was separated from the gas phase using the cyclone. After removal from the gas stream, the powder was collected in the first and second cans, or allowed to flow back into the system. This was controlled using ball valves located at the base of the first and second cans.

The powder was fed into the system via the downcomer. The feed into the system was controlled by adjusting the level of fluidisation of the powder bed in the downcomer. An increase in fluidisation resulted in an increase in the volume of powder entering the conveying line. The driving force for the powder flowing into the conveying system was the pressure head of the powder bed. This provided a limitation on the system, as in order for powder to flow into the system, the combined pressure from the main air flow and the fluidising air had to be kept below the pressure of the powder bed. This restricted the testing of gas velocities above 25m/s, and solids loading was limited at high gas velocities.

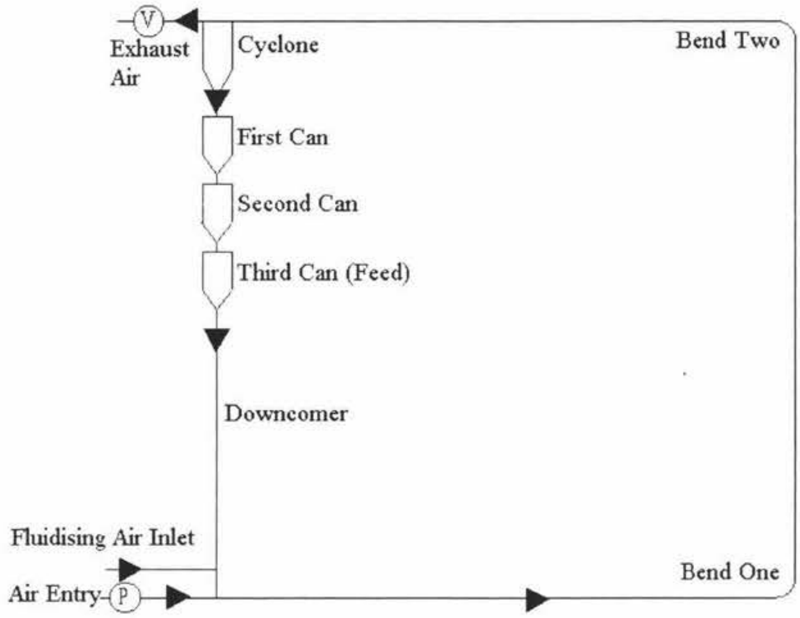

Figure 3-22 Diagram of the conveying system used in the testing

The pressure of the system was monitored using a water manometer attached to a pressure tapping located just before the feed entry point. The gas velocity was calculated from the volumetric air flow. This was measured using a rotameter located after the cyclone. The assumption was made in the calculations that no air leaked from the pipework.

3.9.2 The experimental work

Four experimental runs were carried out at the conditions shown below in Table 3-3. The system allowed the control of two main variables, solids loading and gas velocity. Both of these conditions are known to have an effect on the attrition of powders in a pneumatic conveying line (Mckee *et al.*, 1995). The work done here was concerned with the effect of velocity and so an attempt was made to keep the mass loading ratio as constant as possible throughout the experiments.

Test Number	Av. Gas Velocity m/s	Av. Mass Loading (SLR)	Inlet Air Temp C°	Outlet Air Temp C°	Cycle Time (min)	Number of Cycles
One	24.06	0.83	26.0	23.5	15.74	9.54
Two	19.64	0.87	27.0	23.0	18.38	9.46
Three	24.059	0.89	27	24.5	14.72	8.15
Four	14.23	0.97	25.5	23.5	22.38	9.41

Table 3-3 The conditions for the conveying tests carried out

The aim of this experimental work was to study the effect that the conveying velocity has on the attrition of lactose crystals, with the purpose of relating it back to the experimental work carried out using the impact testing device. The volume of lactose required for each trial prevented the use of lactose within the small size ranges used with the impact testing device. The lactose used in each conveying run was "Special Dense", a commercially available lactose from the Lactose Company of New Zealand.

At the start of each trial a mass of lactose (approximately 10kg) was loaded into the downcomer and feed can. The weight was measured and, using the volume of the downcomer, the bed density was calculated. All four trials gave this as 827kg/m^3. The air flow was then turned on and air was allowed to flow through the system without solids for about 20 minutes. This was done to allow the system to reach steady state. The air flow into the bed was then turned on to allow the solids to flow into the conveying system. Following this, the conveying gas flow and the fluidising air were adjusted so that the desired solids loading rate and conveying velocity were established. Solids loading was determined by measuring the time it took for a measured volume of lactose to enter the system. This was calculated as shown in Equation 3.11.

$$SLR = \left(\frac{\rho_B / V_L}{\Delta t} \right) * \frac{1}{m_A}$$

Equation 3.11

Where SLR = Solids load ratio, ρ_B= density of bed (kg/m^3), V_L= change in volume of lactose (m^3), Δt = time (s) for volume to change and m_A = mass flow rate of air (kg/s).

Using the loading rate and the known volume of powder, the time for one full cycle was calculated. This was used to give an approximate time for the desired ten conveying cycles to occur. This gives approximately 30 impacts per run, with the two bends and the cyclone combining to give three major impact points per cycle. As the conveying method is lean phase, the actual number of impacts is likely to be higher, due to the potential for particle-particle and particle wall interactions to take place any where in the conveying line.

After the completion of each run the lactose was removed from the system and a sieve analysis was carried out on a sample of the powder to determine its size distribution. This was then compared to the particle size distribution of the Special Dense lactose before any conveying had occurred.

3.9.3 Results and Discussion

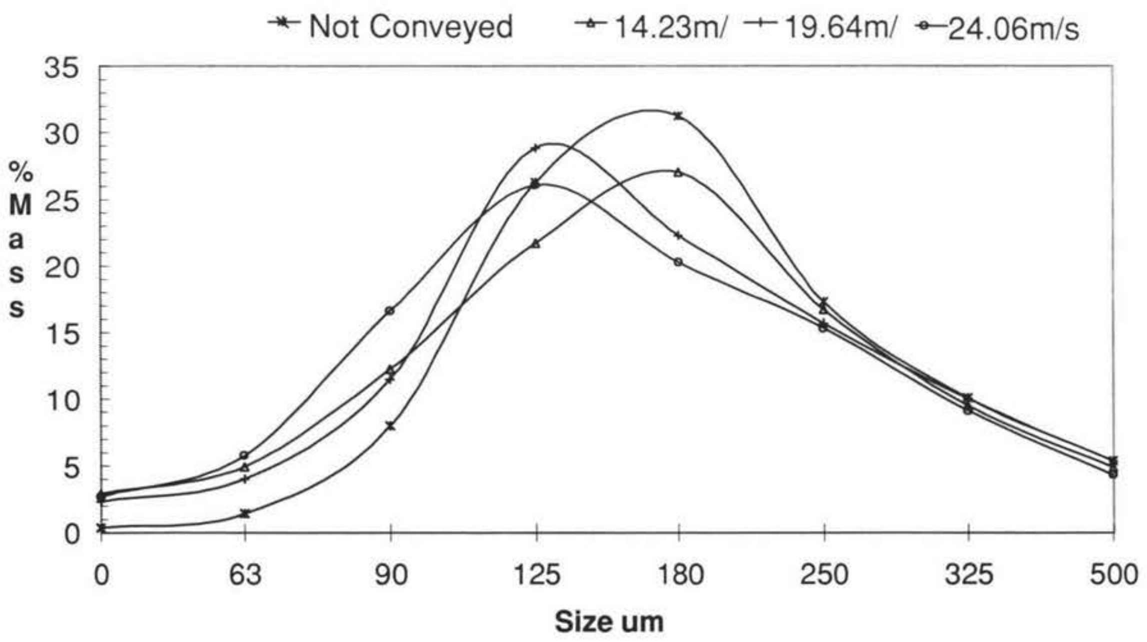

Figure 3-23 The Particle size distributions of the conveyed lactose powder

Figure 3-23 shows the particle size distributions for three of the lactose powder conveying trials, compared with the original particle size distribution of the special dense lactose. It is clear, that conveying results in the attrition of the lactose crystals. This is illustrated by the increase in fines, shown in Figure 3-23, for all the powders that have been conveyed, compared to the original material. Table 3-4 emphasises this effect as it can be seen that for the conveyed lactose the mean particle size decreases and the standard deviation increases.

Velocity	Mean Particle size	Mode Particle size	Standard Deviation μm
0m/s	200.05μm	180μm	99.05
14.23m/s	187.18μm	180μm	105.86
19.64m/s	187.45μm	125μm	106.69
24.06m/s	176.9μm	125μm	104.11

Table 3-4 The mean, mode and the standard deviations of the lactose crystals

What is more difficult to interpret from the results is the effect of the two attrition mechanisms, abrasion and fracture, in the breakage patterns. It is apparent that both mechanisms are having an effect. If abrasion was the sole factor influencing the size reduction, as attrition increased, the small fines produced would become much more significant. What is observed from the particle size distributions, is in fact the opposite, the amount of small fines increases only at a very gradual rate compared to the overall attrition. A portion of the fines are likely to have been lost through the cyclone and this may explain this phenomenon to an extent. A mass balance carried out at the end of each run found losses consistently of 1-1.2% had occurred, a portion of this was attributable to spillage that occurred during the unloading of the feed can , meaning that the fines losses were below the 1–1.2% of total losses recorded.

The particle size distributions for the 19.64m/s and 24.06m/s velocities, in Figure 3-23, show a distinctive shift to the left. This is not seen in the 14.23m/s results. This is demonstrated also by the change in the mode for the two higher conveying velocities. The shifting of these two distributions suggests that there is a change in the type of attrition occurring, as was seen in the single particle impact tests for the higher impact velocities.

The results from the single particle testing work done in sections 3.5 and 3.6 show that this change in the mechanism of attrition should only be occurring to the larger particles; 20m/s for particles in the 425-500μm size range, and approximately 25m/s for particles in the 212-250μm size range. Particles smaller than this, will, according to the impact testing results, not contain enough impact energy at the velocities tested here to cause breakage. However, Tavares & King, (2002) note that in a grinding mill, relatively little control exists over the individual impact events, so that in many instances, the energy input to a particle may be lower than the minimum required to cause fracture. This energy, however, may be able to make the particles more amenable to breakage so that it will require less energy to fracture in subsequent impacts.

This is the case for the attrition that occurs within a pneumatic conveying line, with little control existing over the individual particle impacts. It has already been shown that lactose breakage is increased as a result of secondary impacts, due, it is assumed to the loading-unloading of the particles weakening the crystal. Weakening of the crystal structure due to repeated impacts means that particles in the size range below that where a single impact would have caused fracture at 24m/s are likely to have exhibited some degree of fracture.

3.9.4 Understanding the experimental results

In an attempt to predict the attrition patterns, the data from the impact testing were used to generate curves for the velocities of 14m/s and 24m/s. The curve from the 14m/s pneumatic conveying trial was, assumed to be solely generated from abrasion. This allowed a distribution pattern of the fines produced from the abrasion of lactose in the pneumatic conveying line to be calculated. The distribution of the fines was obtained, by trying to as closely as possible, approximate the 14m/s size distribution.

To obtain the mass of fines produced by the abrasion of a particular particle size Equation 3.12 was used. The P_u values were obtained from breakage curve, Figure 3-6, for the 425-500μm lactose crystals developed using the single impact testing device .

$$M_{nac} = C_F * P_u * I * M_{In}$$

Equation 3.12

where M_{nac} mass of mass of fines produced at sieve size (n).

C_F = correction factor for particle size = n/425μm (425μm is the base value obtained from impact testing)

P_u = fraction of fines produced at velocity (u)

M_{In} = mass of fines in initial sample at sieve size (n)

I = The number of impacts undergone by the particles (3 times number of cycles)

$$M_T = \Sigma M_{nac}$$

Equation 3.13

In the calculations, to redistribute the fines, all losses that became fines from abrasion were assumed to fall into the size range of 0-125μm. To do this the fines were summed together (M_T), as in Equation 3.13, and then redistributed amongst the size fractions 0-63μm, 63-90μm, 90-125μm and 125-250μm as 40%, 30%, 20% and 10% respectively. As discussed above, these percentages were obtained from trying to approximate the fines distribution of the 14m/s experimental curve.

The new mass of particles at a particular sieve size (M_{An}) was then calculated using Equations 3.14-3.18.

$$M_{A(500-180)} = M_{In} - M_{nac}$$

Equation 3.14

$$M_{A120} = (M_T * 0.1) + (M_{I120} - M_{120ac})$$

Equation 3.15

$$M_{A90} = (M_T * 0.2) + (M_{I90} - M_{90ac})$$

Equation 3.16

$$M_{A63} = (M_T * 0.3) + (M_{I63} - M_{63ac})$$

Equation 3.17

$$M_{A0} = (M_T * 0.4) + (M_{I0} - M_{0ac})$$

Equation 3.18

This same set of equations was applied using the P_u value for the 24m/s velocity. It can be seen, by comparing Figure 3-23 and Figure 3-24, that the fines production is significantly higher than was observed in the experimental work. The predicted distribution for 24m/s, with only abrasion occurring, confirms the assumption that two mechanisms are occurring. The large mass of particles smaller than 125μm(fines) present in the predicted distribution is not present in the experimental work.

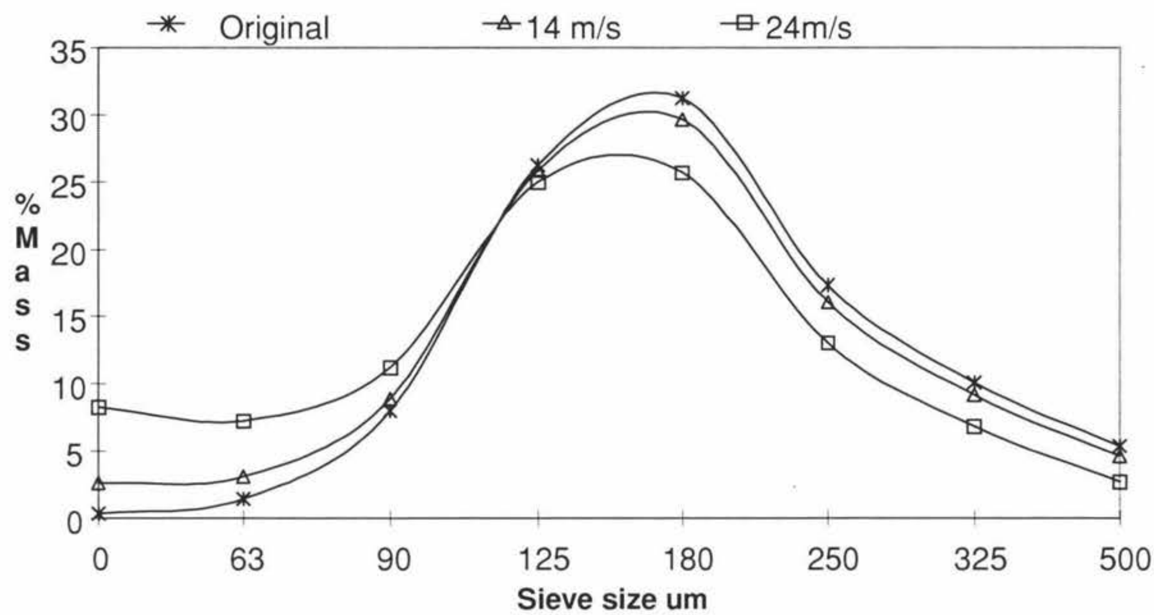

Figure 3-24 The predicted curves for attrition of lactose in a pneumatic conveying line conveying line at various conveying velocities

An attempt to predict the curve for the 24m/s the assumption was made, based on the work done in section 3.5, that attrition was equally attributable to both abrasion and fracture. This means that when applying the P_V values to the work done in the pneumatic conveying line at 24m/s, two mechanisms of attrition need to be considered. To get the new curve for abrasion a new M_{nac} value was calculated where the P_u value was divided by two as shown in Equation 3.19.

$$M_{na} = C_F * (P_u/2) * I * M_{In}$$

Equation 3.19

$$M_{nf} = C_F * (P_u/2) * I * M_{In}$$

Equation 3.20

The addition of fracture into the system was accomplished by attributing fracture to the other half of the P_u value. The mass of broken crystals was calculated using Equation 3.20. The redistributing of the fractured particles was done using the simplification that all fractured particles generated, were added to the sieve size directly below. Fracture of the fines was assumed to not occur due to their small size and the limited amount of impact energy, as discussed above. The drop, and then the respective rise to the left, seen in figure 3.23 for the 19.64m/s and 24.06m/s curves, is thought to be a result of the reduced kinetic energy available for fracture of 125 um particles.

To incorporate this into the prediction, the fracture mechanism was not included in the equations for the sieve sizes 125µm and smaller. To balance out the fracture, the mass of particles broken for 180µm and 250µm sizes was added to the 90µm and the 125µm sizes respectively. Equations 3.21-3.26 shown below are those used to predict masses for the new size distribution (M_{Dn}) with fracture included.

$$M_{D(500\text{-}250)} = (M_n - M_{na}) + (M_{nf+1} - M_{nf})$$

Equation 3.21

$$M_{D180} = (M_{I180} - M_{180a}) - (M_{180f})$$

Equation 3.22

$$M_{D120} = (M_T * 0.1) + (M_{I120} - M_{120a}) + (M_{250f})$$

Equation 3.23

$$M_{D90} = (M_T * 0.2) + (M_{I90} - M_{90a}) + (M_{180f})$$

Equation 3.24

$$M_{D63} = (M_T * 0.3) + (M_{I63} - M_{63a})$$

Equation 3.25

$$M_{D0} = (M_T * 0.4) + (M_{I0} - M_{0a})$$

Equation 3.26

Where M_{nf+1} is equal to the mass of fines created from fracture in the sieve size directly above. The use of the M_n value twice in the final mass allows the total effect of P_u to be included. The resulting predicted particle size distribution is shown in Figure 3-25.

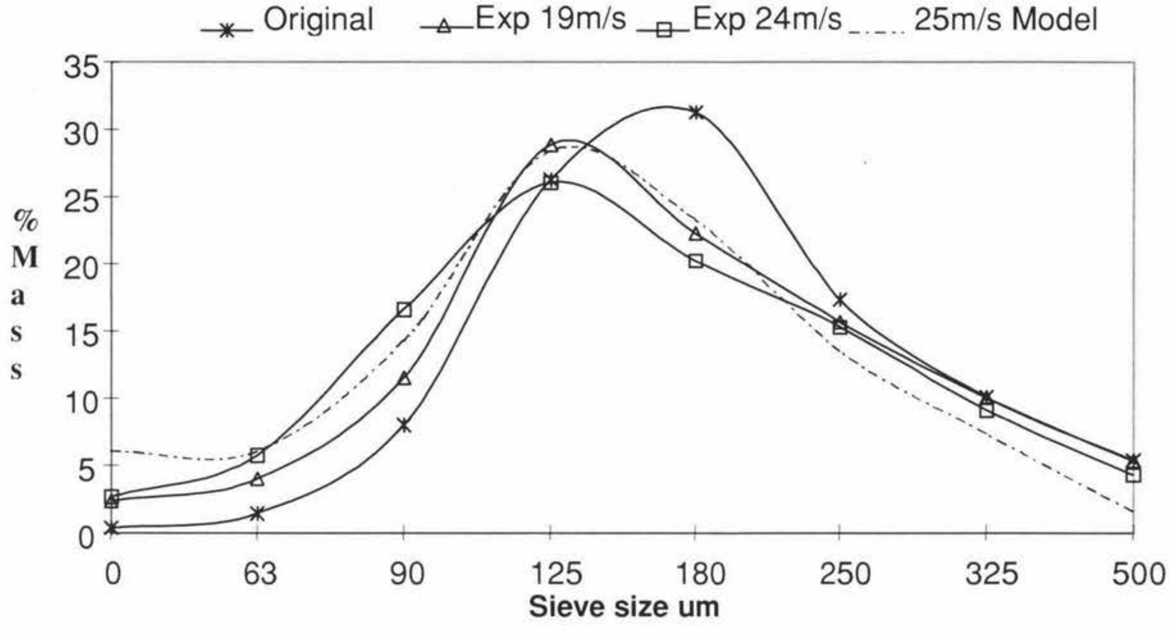

Figure 3-25 Particle size distribution shown for fracture only occurring above 125μm

The curve predicted by the new equations provides a good representation of the pattern given by the experimental results, in figure 3.23. One area where the curve does deviate is in its prediction of the fines production, the difference is to large to be explained by the 1-1.2% losses in recorded the mass balance. An explanation for this is that there is also likely to be a change in the type of fines produced by abrasion. As the energy increases the larger surface defects will be removed.

With each recycle the surfaces of the crystals are likely to become progressively smoother, meaning the potential for abrasion will diminish. This means that abrasion becomes a decreasing mechanism, reducing as the asperities from the surface of the crystal are removed. This compares with fracture where the loading-unloading of the crystals will result in an increasing potential for fracture to occur. Ultimately, as the lactose is recycled through the system, fracture will become the more dominant mechanism of attrition. This was not identified in the single particle testing as only single impacts were tested. However, it may provide another explanation for the slope above 24m/s in Figure 3-7 being lower than expected. If there is a limit to the amount of abrasion that can occur, then it may reach a maximum after which its effect decreases. This could then result in the total rate at which the overall attrition rate increases, decreasing.

This work shows that there is a relationship between the attrition that occurs in the single particle testing device and a pneumatic conveying line. Both sets of experimental work show that attrition increases with increasing the impact velocity. The two sets of work, also show that the two mechanisms of attrition, abrasion and fracture, occur, with fracture only occurring when impact energy permits.

Two main differences in the testing methods were observed. Attrition was shown to be a decreasing mechanism and the fracture appears to occur at lower velocities than was predicted. It is thought that these are a result of the differences in testing methods. In the conveying line trials had repeated impacts of the particles are thought to have caused the smoothing of the particles, allowing for the progressively decreasing effect of abrasion to be demonstrated. These repeated impacts also weakened the lactose yielding lower fracture velocities. This work is only an approximation to the attrition pattern in a pneumatic conveying line in an attempt to determine which mechanisms were dominant and to what degree. Further development would require more experimental work so that properties such as the actual size distributions of the fractured particles can be quantified.

3.10 Conclusion

The results in this chapter have shown that energy of impact is the main consideration for the breakage of lactose crystals in pneumatic conveying. This is not only the energy contained before impact, but also the way in which the energy is dissipated during the contact. This was illustrated with the amount of breakage being a function of not only the energy reduction from the reduced mass, but also, the change in the surface area over which the energy was spread. It was further confirmed, through the use of rubber to increase the time of contact between the lactose crystals and the impact surface, and thus lower the average force the particles were subject to at impact.

The results in this chapter also show that reducing the number of impacts is also an important consideration. The limited space in the impact testing device, meant that lowering the impact angle in an attempt to reduce the size of the impact force, resulted in higher breakages, as secondary impact then became a consideration when the particles were projected onto surrounding surfaces.

Two main ideas have come from this chapter. The first is the possibility that the use of other materials for the conveying lines can provide a method of lowering the breakage and thus the buildup. The second is that energy transfer has an effect on breakage. In using other materials to prevent buildup a knowledge of how they will behave, when subjected to the conditions of conveying line, is required. This, along with another mechanism of energy dissipation, friction, is the focus of the next chapter.

Chapter 4 Friction and Wear

4.1 Introduction

One of the reoccurring themes in the literature on pneumatic conveying, is abrasion and the detrimental effect this can have on conveying pipelines of a pneumatic transport system. This is hardly surprising when you consider that pneumatic conveying involves the passing of one solid across the surface of another solid at a considerable velocity, with air as the carrier medium. This work differs from that which looks at abrasion. Rather than being concerned with the transfer of material from the pipe surface to the conveying solid, it endeavours to provide an explanation for the transfer of a conveying solid to the pipe surface.

Impact forces are not the only forces that particles are exposed to during transport in a pneumatic conveying line. Another force, friction, is present whenever two surfaces come in contact and movement across the surfaces occurs. When two surfaces are in contact with each other there is a potential for the transfer of material from one surface to another; this is referred to as wear. Friction and wear are two closely related subjects and along with lubrication combine to make up the field of Tribology. They will be looked at here as a part of developing a further understanding of the interactions, which occur between the conveying pipeline and the lactose crystals being conveyed.

4.2 Friction

Friction is defined as the resistance encountered by one body moving over another (Hutchings, 1992). It is measured as the force required to move a solid object across another solid surface.

There are three empirical laws of friction; they may be stated as follows:

1) The frictional force is proportional to the normal load as indicated in Equation 4.1 Where F_F is equal to the frictional force, μ is the friction coefficient and W represents the normal force (Hutchings, 1992).

$$F_F = \mu W$$

Equation 4.1

2) The frictional force is independent of the sliding velocity. However, the sliding velocity effects the rate at which frictional energy can be dissipated and hence the temperature at the interface (Hutchings, 1992).

3) On the macroscopic scale, friction is independent of the apparent surface area of the two objects. This is because the large number of surface asperities make the actual contact of the two surfaces a small fraction of what it would appear on a macroscopic scale (Gang *et al*., 1999).

When examined on a microscopic scale the friction force becomes dependent on the contact area. This is the true contact area. The discrepancy between the two phenomena disappears when you consider the first law and the note that the downward force is going to increase the true contact area due to the deformation of the solids (Krim, 1996).

Friction occurs as a result of lattice vibration. This occurs during the sliding interaction of the solid surfaces. The atoms of one surface make the atoms of the other surface vibrate. Hereby part of the mechanical energy, which is required to move both surfaces, is transformed into sound waves and heat (Holinski, 2001). This heat generation gives friction an importance in this work. As lactose particles contact other surfaces, they are exposed to frictional forces, and there is the potential for heat generation. This may lead to the lactose becoming sticky and adhering to the other surface. This can occur like impact, during particle - wall contacts, and during particle - particle contacts.

The movement of a solid over another surface means that enough force has been provided to over come the frictional forces. This force can also be used to break the bonds within the solid and remove fragments of the particle from its surface. This is in effect the same as what is happening when impact forces cause the attrition of particles, except on a more macroscopic scale. When lactose is rubbed along another surface it is possible for small asperities in the surface to be broken away from the main crystals. This also provides the potential for large amounts of heat to be generated as the energy from the breakage of the bonds is released.

Friction also provides the potential for the removal of the material from the surface of the body the crystals are sliding across, causing abrasive wear.

4.2.1 Sliding Contact

Sliding contact occurs when two objects surfaces have a relative peripheral velocity at their point of contact. This compares with rolling contact where the two bodies have a relative angular velocity about axes parallel to their tangent plane (Johnson, 1985). It is when sliding contact occurs that friction becomes important. For contact to occur a normal force is required to press the two surfaces together. This force may simply be the weight of the particles, or it may be much larger, as in the case of an impact force. It is shown in Equation 4.1 that the magnitude of this force has a direct effect on the size of the frictional force that will be needed to be overcome in order for sliding to occur.

Friction becomes a consideration as even during normal straight flow, particle wall interactions are unavoidable and it must be considered that sliding will occur along the surface of the pipe. This is particularly true in sections just after the bends where the velocity has been reduced, as it is possible, at these lower conveying velocities for particles to fall out of the gas stream. In the pipe sections studied at Lactose New Zealand, a level of buildup due to smearing was observed on the straight sections of the pipes. This was evenly distributed over the entire circumference of the pipe wall. This compares with the buildup due to smearing at the impact points, which was focused on the outside of the bend and occurred much more rapidly.

Work done up until now has dealt with what happens to a material when lactose is impacted against it. It is apparent from the discussion above that it is also necessary to know what happens when a lactose crystal contacts a surface through a sliding motion.

4.3 Development of Test for Frictional Smearing

It was established during the impact testing work that lactose could be made to adhere to surfaces such as glass and stainless steel though impact. Contact between the particles and other surfaces occurs through impact, and as the particles slide along other surfaces. This sliding often happens after impact, meaning that the particles are provided with a significant normal force. The sliding behaviour, combined with the downward force, gives rise to the possibility that friction may play a role in the smearing of the lactose on the pipe walls. In order to know if it was possible for lactose to adhere to a surface due to frictional effects it was necessary to develop an experimental method that could provide a frictional force without any impact effects.

In the initial stages of testing to see if lactose could be made to adhere to the surface of a material, lactose powder was rubbed along a stainless steel sheet using another steel block to provide a downwards pressure. After rubbing a certain distance, the powder adhered to the steel surface and the horizontal force required to move the block providing the normal force became much larger. Examination of the surface revealed that the lactose was solidly caked on and removal could only be done by either washing the surface or through scraping the powder off.

This initial test indicated that lactose could be made to adhere to a surface through sliding contact. As an improvement to this test, the device shown in Figure 4-1 was developed. The device was designed with the aim of testing how lactose behaved when undergoing sliding contact. The advantage the device in Figure 4-1 has over the test described in the paragraph above is that it allows the downwards force and the sliding velocity to be more accurately measured, than the sliding block test described above.

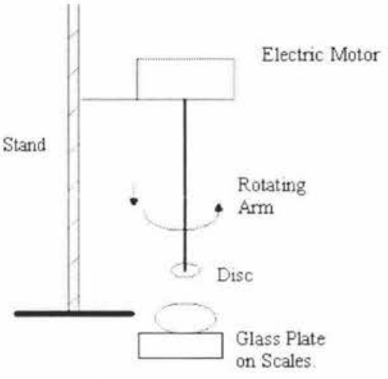

Figure 4-1 Sliding Contact Testing Device

The device consists of a 0.1kW laboratory stirrer (Chiltern Scientific) with a steel plate attached to the bottom. On the surface of the steel, "Selleys Araldite" was used to adhere lactose crystals in such a manner that the crystals were proud of the surface. The rotating arm had a maximum speed of 1050rpm (measured using a Line Seiki Hand Tachometer TM-2011) and the disc had a diameter of 3.1cm. The velocity at the edge of the spinning plate was calculated to be to 1.7m/s. The value the particles can be exposed to during testing is lower than this, as when a downward force is applied to the plate the resistance encountered reduces the operating speed. The glass plate against which the smearing was carried out, was mounted on scales (Camery ACS-6) using double sided tape. This proved effective for stopping the plate moving and allowed easy removal.

4.3.1 Buildup due to sliding contact

It is the normal force that is the controlling factor in the how much frictional force will be generated by the contact of two surfaces. The other factor influencing the buildup from the sliding contact in a pneumatic conveying line is velocity. Velocity controls heat dissipation. The sliding contact device shown in Figure 4-1 was used to test how these two variables influenced smearing. The surface used for contact was glass, as this allowed the smearing to be easily observed. A series of different velocities and normal forces was used and the plate was examined for smearing after each run.

It was difficult to quantify how much smearing had occurred after each test, as the device used removes material after it has been smeared. Consequently it was used to show whether smearing had occurred or not.

Speed (rpm)		**Weigh Force (N)**		**Smearing**	**No.**
133	Slow	12.65	High	Yes	1
931	Fast	2.96	Low	Yes	2
146	Slow	2.90	Low	Yes	3
865	Fast	12.72	High	Yes	4

Table 4-1 Testing of different effect for sliding buildup

It can be seen in Table 4-1 that smearing occurred in all four tests. Figure 4-2 shows these results. The explanation for this is that lactose crystals do not have perfectly smooth surfaces and there are a number of asperities. This means that even at the low downward force of 2.7N it was possible to find small regions of the crystal that could be broken, resulting in smearing. The 2.7N was close to the smallest force that could be applied using this test method. It is speculated that even at lower forces a small amount of lactose could be removed from the crystal surface and smeared on to the glass surface.

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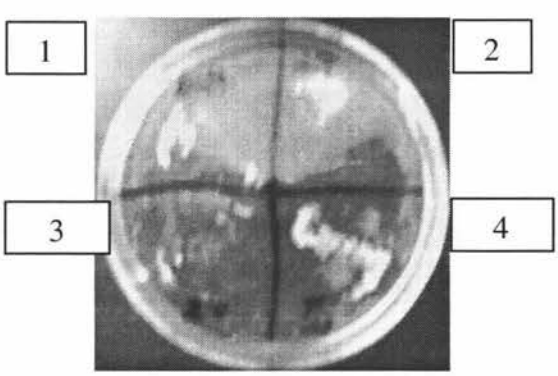


Figure 4-2 The results of the sliding contact test

A microscope was used to examine the smearing that has occurred during the sliding contact testing. Figure 4-3(1) is a digital image taken using the microscope. It shows the streaky nature of the adhered material. This compares with the impact image, Figure 4-3(2), where the adhesion is more localised as if the smearing occurs and then the main particle is removed.

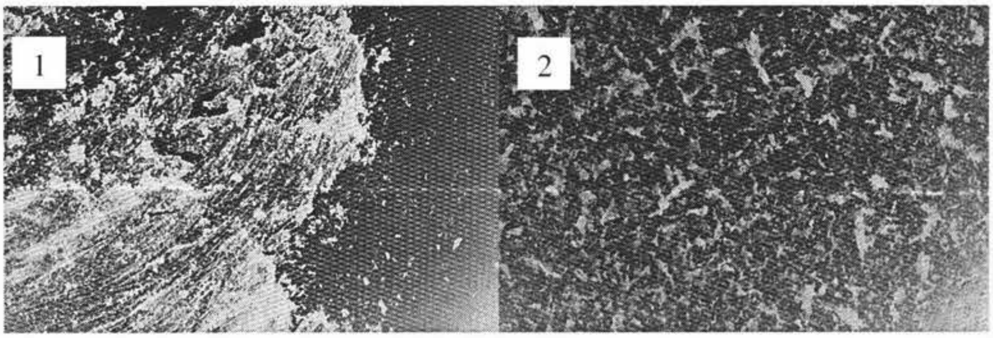


Figure 4-3 Microscope view of adhesion due to sliding contact[1] and impact[2]

4.3.2 Additional Tests for Smearing

4.3.2.1 High Velocity

One of the limitations of the above test is that it does not simulate the high velocities experienced by the particles in the conveying line. Frictional force is not effected by the sliding velocity. However, the amount of heat generation is, as the faster the particles slide across a surface the less time there is for heat dissipation. This means that at higher velocities the temperature rise is greater. Also, in the above experiment particles are being constantly ground, simulating a mill. A further possible difference in using the low velocities is that they allow a longer contact time between the particles and the surface. This may be more conducive to bond formation.

To eliminate all the possible problems presented above, a test was needed that would simulate a situation where the lactose crystals were not being subjected to a constant force and being ground over long periods. As well as doing this, it was required that a high velocity could be generated.

Lactose crystals were stuck to a flat disc 10cm in diameter, using "Selleys Araldite" glue. The crystals were glued around the entire circumference of the disc and covered the region of the disc 3cm from the centre and extending to the edge.

The disc was then mounted on an angle grinder (BOSCH PWS 600). This has an operating speed of 11000rpm. During testing, the crystals were moving at velocities ranging from 35 to 58m/s.

The grinder was then turned on and allowed to reach a steady speed. It was then contacted against a stainless steel plate for about 10 seconds.

The stainless steel plate had clearly visible smearing on it that could not be wiped off with out using water or through scraping. In order to show that the smeared material was not glue a test was done where the smearing was washed with hot water, this dissolved easily. This is what would be expected for smeared lactose. A sample of the glue was also washed with hot water and this did not dissolve. From these results, it was concluded that the material smeared on the surface of the stainless steel plate was lactose.

4.3.2.2 High Pressure

As it is the normal force that control the amount of friction between surfaces it is necessary to test for a large downwards force without the effect of velocity. Compression loading is known to lead to breakage of particles, which may lead to sticking. A run was carried out where lactose was subjected to a compression force of 200N and the surface examined for smearing. No smearing was apparent. Two possibilities are considered to explain this observation. The first is that the energy input from velocity is required to cause the lactose to adhere to the surfaces. The second is that the breakage of the crystals did not occur at the surface of the plates. As no breakage occurred at the surface of the plate no amorphous lactose was in contact with the plate surface and hence no adhesion occurred.

4.4 Wear

Wear is the damage to a solid surface, generally resulting in progressive loss of material, due to the relative motion between that surface and a contacting substance. It can be further defined as erosive wear; wear due to impact, and abrasive wear; wear due to the sliding of a material over the surface (Hutchings, 1993).

Following on from the work on impact and friction an understanding of wear becomes important in this work. A possible solution to reducing the adhesion of lactose to the walls of the conveying pipes is to use different materials in the areas prone to buildup. In doing this, it is important because of the physical characteristics of lactose and its applications as a product, that not only is the wear of lactose reduced but also the material being used in the pipeline exhibits minimal wear. High levels of wear lead to increased potential for foreign matter to appear in the final product and increase the frequency at which replacement of the pipes is required .

One of the controlling factors in the rate at which wear and abrasion will occur is the relative hardness of the particles. Particles with lower hardness than that of the surface cause much less wear than harder particles. There is a point where the particles become so much harder than the surface that the exact value of their hardness becomes insignificant (Hutchings, 1993)

In the case of the surface being significantly harder than the particles, the exact material hardness was shown in chapter three to be of little consequence in the amount of breakage observed. Stainless steel and glass, two of the materials tested, had a very similar effect on the amount of breakage observed. This similar breakage occurred even with; as shown in Table 4-2, glass having a hardness twice that of stainless steel, the governing factor was that both are significantly harder than lactose.

Material	Hardness GPa
Lactose	0.70[1]
Stainless Steel	200[2]
Glass	400[2]
Rubber	0.001[3]
Teflon	0.08[3]

[1] (Arteaga *et al.*, 1996, [2]Hutchings, 1992, [3]Brostow *et al.*, 1996)

Table 4-2 Hardness of Various Materials

4.4.1 Erosive Wear

It was stated above that erosive wear results from the impact of a material against another surface. Impact of particles against the surface of a conveying line is an unavoidable aspect of pneumatic conveying. Understanding the potential erosive effects that lactose has on different materials meant that a method of testing these materials had to be determined.

4.4.2 Testing for Erosive Wear

The test for erosive impact was done using the impact-testing device described in chapter three. The material of interest was stuck to a glass disc using super glue and then mounted in the collection chamber. A mass of between 5 to 6 grams of lactose particles in the size range of 412-500μm were then fired down at the material using a gas flow rate of 35m/s. This was seen to be the at the upper end of the typical conveying velocity. The gas flow rate equates to a particle velocity of about 34.9m/s, using the relationship obtained from work where particle velocities were measured, this relationship is shown in Figure 4-4. After each run the material of interest was inspected for wear and build up. Each material was also weighed so that any losses could be quantified.

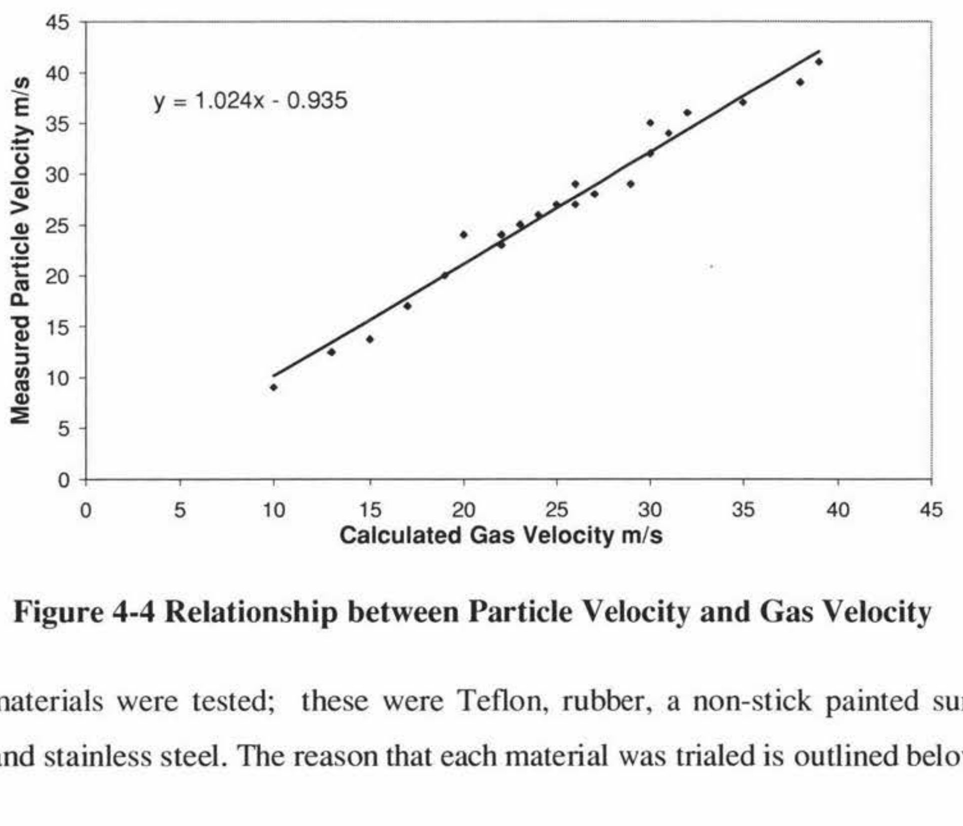


Figure 4-4 Relationship between Particle Velocity and Gas Velocity

Five materials were tested; these were Teflon, rubber, a non-stick painted surface, glass and stainless steel. The reason that each material was trialed is outlined below.

4.4.2.1 Glass

Glass was used in chapter three for the impact testing work and as such its behaviour was well known, this made it a good reference material. It also has the advantage that it is clear and this makes any buildup easily observable.

After the impacting of the crystals, examination of the glass disc revealed that lactose had adhered to the surface of the glass slide. This was as expected as it had been seen before in all the other tests carried out using glass as a target material. The smeared lactose powder had adhered to the glass surface. Removal of the lactose required the washing or scraping of the surface, as brushing was ineffective. No abrasion or wear of the glass was visible or could be felt by rubbing the surface. As shown in Table 4-3 no weight change was observed for the glass plate.

4.4.2.2 Rubber

Rubber was shown in chapter three to through its ability to adsorb some of the energy of impact, reduce the breakage of lactose crystals, a result that suggested that it may have a use in preventing lactose buildup. To further test the suitability of rubber in a conveying line, the resistance to wear was tested.

For this test three rubber types where used, a black rubber, a nitrile/SBR rubber (Skellerup reference number WCR930) and Linaplus FG. The black rubber was used as it made any build up more observable. The two other rubber products were white food grade rubbers and the buildup was difficult to see due to the similarity in colour if the materials with lactose.

After impact, powder was present on the surface of the black rubber, the buildup was removed through brushing the surface. No abrasion or wear on the surface of any of the rubber products was visible or could be felt by rubbing the surface. These results compare well with what has been observed with the white nitrile rubber used for the breakage tests.

4.4.2.3 Stainless Steel

Stainless steel is the material being used currently for the conveying pipelines at Lactose New Zealand. A visual examination of the stainless steel conveying lines revealed no significant wear, even in areas of high impact such as bends. A explanation for this is that buildup of the lactose powder on the surface of the pipelines provides a protective layer from any potential wear.

To test and see how well the results obtained from the particle gun related to the actual situation, stainless steel was included in this study. After the impact test was carried out, examination of the plate showed that lactose had adhered to the surface of the stainless steel disc. The powder could not be brushed off and removal required that it was washed or scraped off. This had been seen before in the other tests carried out using stainless steel as a target material and is also what happens in the conveying pipes. No abrasion or wear of the stainless steel was visible or could be felt by rubbing the surface.

4.4.2.4 Non Stick Pan Surface

A non-stick surface was thought to be an option in preventing the buildup, as it would provide a surface to which the powder would be unable to adhere. To test the usefulness of this type of surface a non-stick pan (Necessities Brand) was purchased . The pan was made of steel and had a black non-stick paint applied to the surface. An inquiry made to the supplier found that the non stick agent in the paint was a high molecular weight silicon compound.

For testing, a small disc was cut from the pan and glued to the glass plate. Lactose crystals were then impacted against the surface. Following this, the surface was examined for wear and buildup of powder. There was a distinctive region in the centre of the plate where the majority of crystals had impacted against the surface. In this area the non-stick surface had become removed, exposing the aluminium backing. As a non-stick surface the remaining surface worked well, with no buildup present in areas where the non-stick surface had not worn away.

The ease with which this surface was removed meant that is would be unsuitable for use as a method of reducing the buildup. The usefulness of this product in preventing buildup in the pipelines was further reduced by it colour, as the black material would be easily visible as foreign matter in a white lactose powder.

4.4.2.5 Teflon

Teflon is another non-stick product that it was thought could potentially be used to stop the powder adhering to the surface of the conveying pipes. A small Teflon plastic disc was glued to the surface of the glass plate. Crystals were then fired down at the plate. After the impact test, the plate was examined and a small amount of fine powder was on the plate. This was easily removed though brushing the surface of the plate, indicating that the powder had not adhered to the surface. However, where impact had occurred, the surface had become visibly worn and had gone from being very smooth, to being pitted and rough, indicating that the surface had been cut into by the lactose crystals.

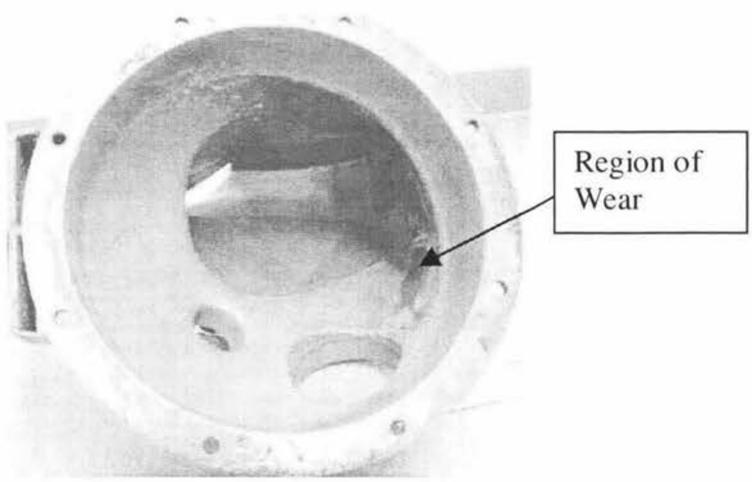


Figure 4-5 Chute showing wear of Teflon lining

This has been shown to be the case in the industrial environment. Teflon is used at Lactose New Zealand as a method of preventing buildup on the inside of the chute shown in Figure 4-5. It has proved in the industrial environment to be very effective at stopping the buildup occurring. The powder conveyed that passes through the chute has a 6% moisture content and before the Teflon coat was used in the process the powder built-up on the surface of the chute and prevented flow. With the addition of the Teflon coating the buildup has ceased to be a problem. It can be seen in Figure 4-5 that in areas of impact, wear does occur. The area with no coating on it is a high impact area on the chute and has worn off the surface. This is a regular occurrence and the lining has to be replaced at least once a year.

Material	Weight Before (g)	Weight After (g)	Percentage Change
Glass	4.7289	4.7289	0.00
Stainless Steel	8.9516	8.9517	0.001
Teflon	5.3899	5.3895	0.007
Non Stick Pan	7.5632	7.5611	0.028
Rubber (Lintex FG)	10.947	10.946	0.001
Rubber (Nitrile)	11.1535	11.1536	0.001

Table 4-3 Weight changes after erosive wear test

Table 4-3 shows the results of the weight change after each material had been tested for its resistance to erosive wear using the impact-testing device. It can be seen that only the "Non-stick pan" surface shows any significant weight change. Teflon, the other material where erosion was seen, shows only a minor reduction in weight. It is possible that this weight change is due to error and that the surface had not lost material but had been pitted by the impact giving the impression of wear. This pitting would, in an conveying environment, be likely to eventually lead to cutting of the material, essentially another form of wear.

The testing done using the impact-testing device is limited by the volume of powder that can be impacted against the various surfaces. In a pneumatic transport line, the volume of powder impacting against the surface is significantly higher and any effects of wear will be greatly exaggerated. So whilst wear may not have been observed in a material here, it is possible that it will still occur.

4.4.3 Wear of Materials due to sliding contact

Just as it is important to know how a material will wear due to impact in a pneumatic conveying line, it is also important to know how it will wear when it undergoes a sliding contact. To test the effect of sliding contacts on the wear of materials, the same materials that were examined for erosive wear, were tested using the sliding contact testing device.

The test involved applying a normal force in the range of 9.3-10.3 N and a rotational speed of 380rpm to the material. This test also gave the opportunity to see how well the different materials resisted the adhesion of lactose when sliding contact occurred.

The materials were subjected to the wear test for 30 seconds, after which they were studied so that any lactose buildup or wear could be noted. The materials were then washed to remove any powder that was present on the surface. After washing the material was dried and then reweighed to see what mass of material had been removed from the original sample. These results are shown in Table 4-4.

Material	Weight Before (g)	Weight After (g)	Percentage Change
Rubber (Linatex FG)	7.2209	7.2196	0.018
Rubber (Nitrile)	9.1900	9.1694	0.224
Stainless Steel	4.2505	4.2506	0.002
Glass	8.9126	8.9125	0.001
Non Stick Pan	4.5192	4.5182	0.022
Teflon	0.6258	0.6257	0.015

Table 4-4 Weight Change for Wear of Material Testing

4.4.3.1 Glass

As per Section 4.3.1 it was again demonstrated that lactose could be made to adhere to glass through sliding contact. The examination and then weighing of the glass showed no wear of the glass surface.

4.4.3.2 Rubber (Linatex FG)

Examination of the rubber surface after the sliding contact test had been carried out found that no observable wear or adhesion of lactose had occurred. The reweighing of the rubber after washing showed that a small change in weight had occurred. This compares to the result observed after impact where no weight change was recorded. The test shows that the rubber may be susceptible to abrasive wear.

4.4.3.3 Rubber (Nitrile)

The examination of the rubber surface after the sliding contact test had been carried out found no adhesion of lactose to the surface had occurred. What was shown was cuts in the surface of the rubber where wear had occurred. The reweighing of the sample confirmed that wear had occurred. The percentage weight change is shown in Table 4-4. It can be seen that the weight loss for this rubber is much higher than for any of the other materials tested. Compared to the Linatex FG, the nitrile rubber showed significant levels of wear.

An explanation for this abrasion is that rubber has an extremely high coefficient of friction. This means that in areas where sliding is likely there is greater amount of friction than would be experienced by the other surfaces. This results in increased heat generation and the potential for more wear. The Linatex FG is a much softer rubber and is more easily deformed than the Nitrile rubber. This ability to deform means it is able to counter the effect of increased friction.

4.4.3.4 Stainless Steel

The adhesion of lactose to stainless steel was confirmed here. The examination of the surface and the weight change in Table 4-4 shows that stainless steel is resistant to wear by lactose.

4.4.3.5 Non Stick Pan

The non-stick pan surface proved more resistant to sliding wear than it did to the impact wear test. The smooth surface of the pan seem to have the ability to allow the crystals to slide over the surface. No build up occurred on the non-stick surface. After a short time period, some of the surface was removed and the lactose adhered to the underlying aluminium.

4.4.3.6 Teflon

Teflon proved to be resistant to sliding wear. Some small indentations were observed in the surface, this appeared to be more a function of the downward pressure used in the test, than from the sliding of the lactose across the surface. It compares with the non stick surface which also showed better resistance to sliding wear than it did to erosive wear. In resisting buildup it was very good with no lactose adhering to the surface. These results suggest that in areas where impact is not a factor, Teflon has applications in reducing any adhesion to the surface.

4.5 Conclusion

The first section of this chapter was concentrated around looking at the effect that sliding contact has on the adhesion of lactose to surfaces. The results showed that the combination of the frictional forces and the sliding velocity can provide enough energy to cause lactose to adhere to a surface. The size of the downward force and velocity in the trials completed showed that the adhesion occurred at low velocities. The conclusion that sliding can cause smearing, is a factor that needs consideration in designing bends for the conveying of lactose. Work by Venkatasubramanian *et al.*, (2000), already discussed in chapter two, found long radius bends produced more adhesion. This may be due to the sliding of the powders across the surface and may mean that alternate bend designs will provide less buildup. In terms of adhesion this work comes to the same conclusion as chapter three, with energy being the main consideration in the breakage and adhesion of lactose to surfaces. The adhesion of lactose and the effect of energy are discussed in the next chapter.

The second focus of this work was the suitability of materials for use in lactose conveying lines. The main focus was their resistance to wear although the test also established their resistance to adhesion. Two types of wear were considered abrasive and erosive. A number of materials were considered with the aim being to find a material that could resist both wear and adhesion. A soft rubber (Linatex FG) was found to be the most promising in terms of being able to carry out both these demands. The ability of this material to do this was concluded to be a function of its ability to deform and dissipate energy. The testing had was limited in the time available to carry it out. To overcome this the suitability of this rubber was tested in a lactose conveying line. These results are presented in chapter six.

Chapter 5 Adhesion

5.1 Introduction

It has been shown in the previous two chapters that it is possible to cause lactose to adhere to a surface though both impact and sliding contact. The main link between these actions is that they involve the breakage of lactose crystals because of a force, be it impact or friction. Breakage was an area that has been identified in the literature as being one of the major factors influencing the buildup of powders in conveying lines. The main aim of this work was to provide an explanation of what causes the build up to occur and to provide possible solutions for preventing this. This chapter is focused around looking at what causes the lactose to adhere to surfaces.

5.2 Measuring Amorphous Lactose

A proposed explanation for the sticking of lactose powder is that breakage of the α-lactose monohydrate crystals leads to the formation of amorphous lactose. To identify whether amorphous lactose plays a role in the buildup it is necessary to show that breakage leads to the formation of amorphous lactose. To do this requires that a method of measuring the amorphous concentration of a lactose powder is available.

There are a number of different techniques listed by Sebhatu *et al.*, (1994a) available for measuring of the degree of crystallinity of a solid. The analysis has been performed using X-ray analysis, infrared spectroscopy, solid state NMR spectroscopy, density, differential scanning calorimetry (DSC) and isothermal heat conduction microcalorimetry. It is also possible to detect crystalline substances using a polarised light microscope.

The method chosen was isothermal conduction microcalorimetry. This was carried out on the Thermal Activity Monitor 2277 (TAM) (Thermometric AB, Sweden) at Lactose New Zealand. Isothermal conduction microcalorimetry is a technique in which a sample is held at one temperature. This means that any processes, which occur in the cell can be monitored by heat gain from, or heat loss to, a heat sink (Buckton *et al.*, 1995a).

The operation of a microcalorimeter is described by Buckton & Beezer, (1991) as follows. A sample cell ($3cm^3$ for this work) is surrounded by a semi-conductor thermopile, which is in turn, is located in a high heat capacity, large mass heat sink. The heat sink is maintained, via water thermostation, at a defined temperature, (25°C for this work) to +/- 0.0001°C. Any process which occurs in the cell results in a change in enthalpy and thus generates a rapid heat flow across the thermopiles. The output voltage of the sample is connected in opposition to the thermopile of the reference cell, to eliminate any external effects. The resultant voltage signal is amplified and recorded on a computer. The signal is the rate of change of heat with time (power). The stability of the signal is such that a base line fluctuation over a 24-hour period would be no more than 0.1μW.

It was shown by Sebhatu *et al.*, (1994a) that this technique could be used to monitor the recrystallisation of amorphous lactose. The technique was used by Buckton *et al.*, (1995b) to detect amorphous lactose at concentrations as low as 0.313% using an RH of 75%. However, at this point the adsorption and crystallisation peaks began to superimpose over each other.

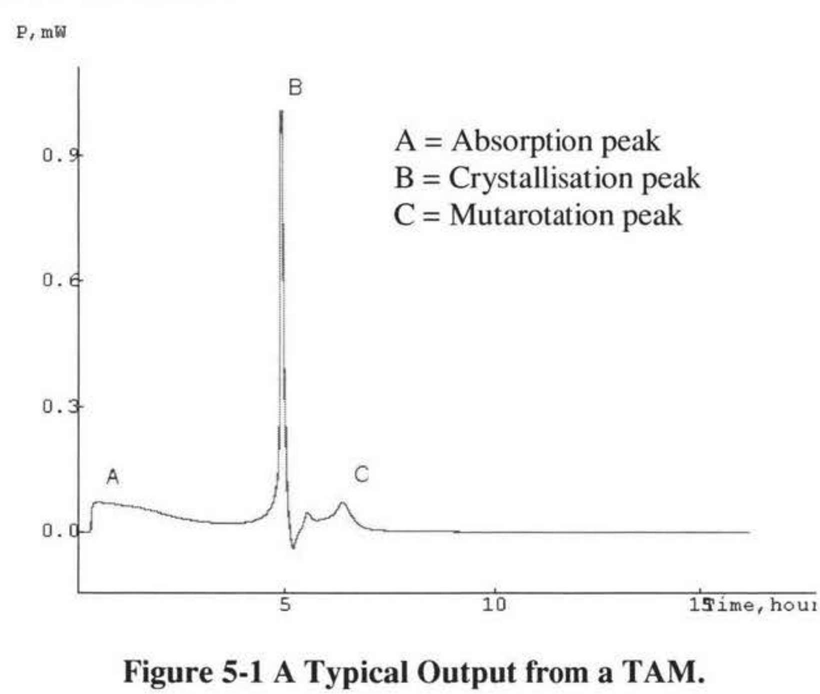


Figure 5-1 A Typical Output from a TAM.

Figure 5-1 shows a graph output from an isothermal microcalorimeter (TAM). It can be seen that there are three main peaks on the graph. These are labelled A, B, and C. The first peak Figure 5-1 (A) stage is a wetting response due to the absorption of water (exotherm) being slightly out of balance with the evaporation (endotherm) of the water from the salt solution. The second peak Figure 5-1(B) is due to the crystallisation of the amorphous lactose. The third peak Figure 5-1(C) is believed to be a result of the mutarotation of the lactose to a stable condition (Darcy & Buckton, 1998b).

The time taken between the peaks for crystallisation is dependent on the relative humidity used during the analysis; a higher humidity results in less time for crystallisation. A smaller sample size and a lower amorphous content, also reduces the crystallisation time (Sebhatu *et al.*, 1994a).

Bergqvist & Soderqvist, (1999) found that for detecting low levels of amorphous lactose in samples of lactose powder, microcalorimetry was better than DSC and X-ray diffraction. The detection level for microcalorimetry was 1-2%. This compares with DSC and X-ray diffraction, which require a level of more than 10% to give accurate results. It was expected that the concentration of amorphous lactose that would be present in the sample prepared in this work would be low, as it is only the surface layer of the broken crystal where amorphous lactose is expected to form.

5.3 Amorphous Formation

The basic theory of sticking due to amorphous lactose states that, when the glass transition temperature is exceeded the solid can begin to flow. This allows the formation of bonds (Brooks, 2000). For the sticking of lactose to occur as a result of amorphous bond formation, there needs to be amorphous lactose present in the powder. The powder being conveyed through the pneumatic transport lines at Lactose New Zealand is α-lactose monohydrate. This means that for amorphous lactose to be present conditions have to exist that allow the change in state to occur.

Lerk *et al.*, (1984) and Otsuka *et al.*, (1991) both showed that crystalline lactose could be transferred into non crystalline lactose through mechanical stress. O'Donnell, (1998) milled crystalline lactose and recorded a 2.8% amorphous lactose concentration in the final product. Roth, (1976) also showed that when crystalline sucrose was milled to form icing sugar a level of amorphous sucrose was formed on the surface.

The work done by Roth, (1976) estimates that an amorphous layer on the surface of icing sugar after it has been milled has a depth of 75A°. This mechanism of mechanical stress causing the formation of amorphous lactose is thought to be the cause of the buildup of lactose in pneumatic conveying lines. The formation of amorphous lactose requires a temperature rise to above the melting point of lactose. Once this occurs the molecules can enter a liquid phase then rapid cooling means they can not return to the crystalline state.

In an attempt to identify if amorphous lactose was a factor, in the buildup, a sample of the lactose that had smeared on the conveying line in the factory was taken. The sample was tested for any amorphous content using the TAM. It can be seen from the two raised peaks on the line labelled "lactose from pipe wall" in Figure 5-2 that amorphous lactose was present. It is assumed that long flat pattern of the two peaks is a result of the highly compacted nature of the sample. This compares to the Super-tab sample that has a porous structure, which allows the system conditions to be almost constant throughout the amorphous layer. As a result the crystallisation occurs quickly.

The compacted nature, meant that diffusion through the solid controlled how fast the crystallisation occurred. Calculating the amorphous content the from overlapping energy curves required that the crystallisation peak was extrapolated to determine the amorphous lactose concentration . An approximate answer of 2.3% was obtained from the crystallisation curve.

A sample of free flowing powder from the same section of the line was taken at the same time as the smeared sample was taken. This free flowing powder was taken to provide a comparison between the caked and non-caked powders. The sample was run through the TAM in the same manner as the caked lactose sample. The results are shown in Figure 5-2 as the "alpha lactose line". It can be see from this line that when compared against the other two samples, the amorphous content of this sample was insignificant and of a level that can be assumed to be zero.

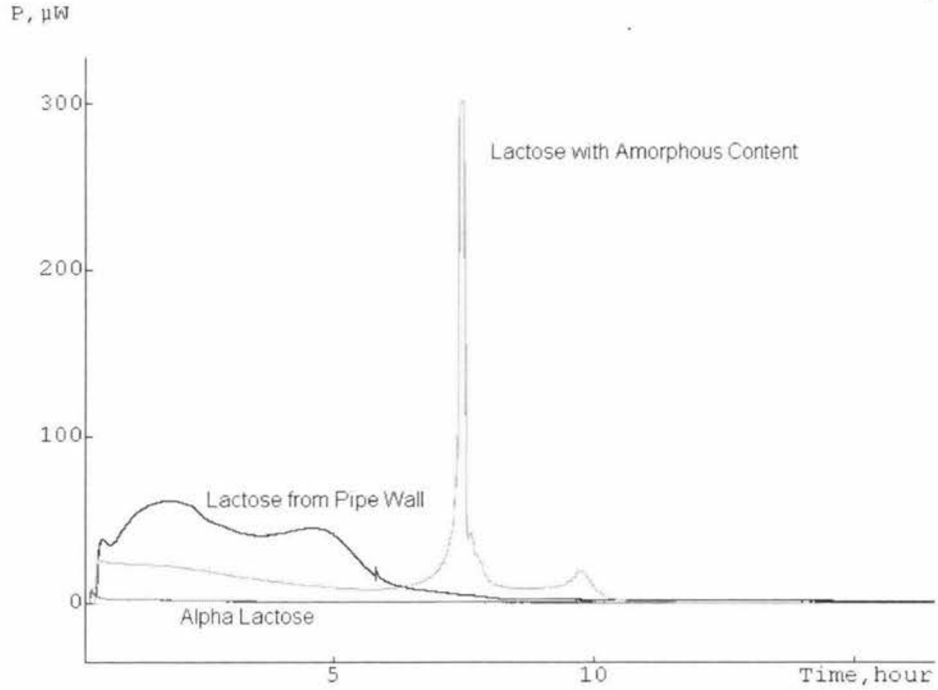


Figure 5-2 T.A.M graph showing the presence of amorphous lactose

Two tests were carried out with objective of showing, that amorphous lactose forms after crystalline lactose has been broken,. The first test took lactose crystals, and using the impact testing device, fired them at a glass plate at 30m/s. The second test used the friction-testing device, with 1.2kg of force applied to the particles and a rotating speed of 430 rpm. At the completion of each test, the samples of broken powder were collected and stored in a desiccator to stop any spontaneous crystallisation of the amorphous lactose. The samples were then analysed for amorphous content using the TAM.

The graph on the left in Figure 5-3 shows the output from the TAM for α-lactose monohydrate (A) against, lactose after impact (B), and lactose broken using the friction device. The graph on the right in Figure 5-3 shows these results compared against Supertab (D), a spray dried product produced by Lactose New Zealand with an amorphous content of around 8%. The results on the left in Figure 5-3 have a higher absorption peak than the α-lactose sample, indicating that it is possible that amorphous lactose has been formed as a result of the breakage. No crystallisation peak is visible. The low amorphous content of the samples, may mean that this peak has become superimposed onto the absorption peak.

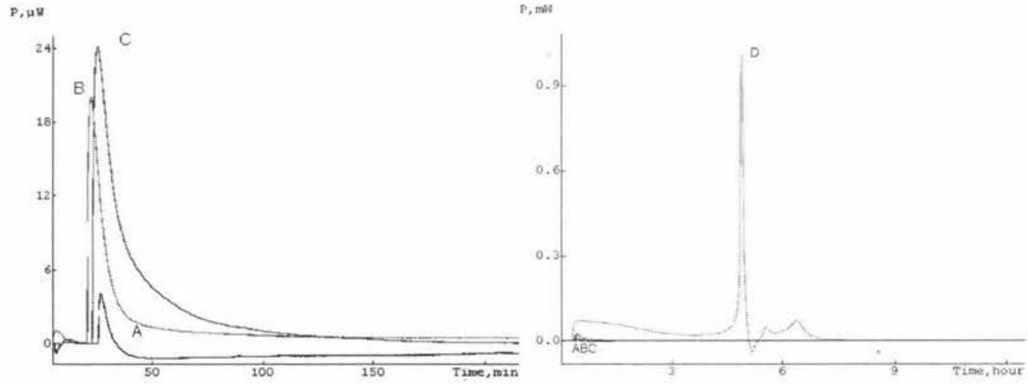
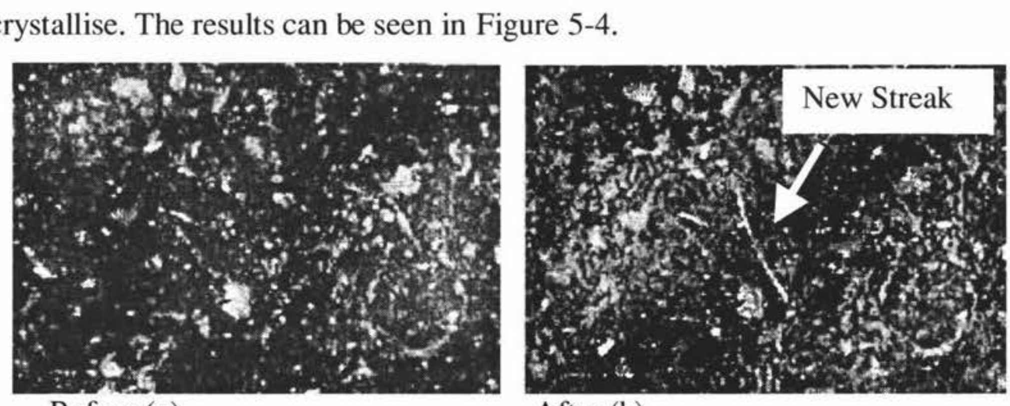


Figure 5-3 TAM output for breakage testing.

It can not be conclusively said that amorphous lactose is present in these samples due to the very low heat output values. The difference could by due to noise. The reason for the difficulty in detecting amorphous lactose in the two samples, is assumed to be because it forms in such small amounts that it is undetectable, using the methods available.

As an alternative method for determining if amorphous lactose is formed on impact, a polarised microscope was used to examine the surface of the glass plate the lactose crystals were impacted against. This plate was examined twice the first time was straight after the impact test had been completed. The plate was then placed in an environment of 100% humidity to allow the any amorphous lactose that had formed to re-crystallise. The results can be seen in Figure 5-4.



Before (a) After (b)

Figure 5-4 Polarised microscope of smeared lactose before and after exposure to a high humidity environment.

Ignoring the differences in focus that appear in the two images it can be seen that in Figure 5-4(b) that there is a new streak apparent. There is also evidence of other crystals that have grown and more general crystals. This suggests that recrystallisation has occurred and that amorphous lactose was present on the slide.

5.3.1 Calculating amorphous formation

In determining the ability of the TAM to detect any amorphous lactose that may have formed on the surface of the lactose, an approximation of the amount formed on the surface from a breakage event is required. Roth, (1976) calculated the depth for the formation of amorphous sucrose to be 75A°. This estimate was obtained through milling crystalline sucrose, determining the amorphous concentration (a_c), then using Equation 5.1 to equate the depth (d_a).

To gain an approximate depth of formation for amorphous lactose this same method was used in this work. Crystalline α-lactose was milled using a Cyclone sample mill (UDY, Model 3010-019). The sample was then analysed using the TAM and a amorphous content of 1.06% was determined. The Malvern Mastersizer was used to obtain the average surface area (S_{am}) for the milled powder sample, this was 0.701m^2/g. Using these results a depth of formation of 98.5A° was obtained, this is of a very close order of magnitude to that calculated by Roth (1976) for sucrose.

$$d_a = \frac{a_c}{\rho_L * S_{am}}$$

Equation 5.1

The result obtained from Equation 5.1 for the depth of formation was used in Equation 5.4 to estimate the percentage of amorphous lactose formation. The equation given calculates the percentage of lactose that may form after one impact, assuming breakage occurs across the centre. A crystal of 400μm in diameter has been used in the calculation.

$$CrystalVolume(V) = \frac{4}{3}\pi\left(200*10^{-6}m\right)^3 = 3.35*10^{-11}m^3$$

Equation 5.2

$$AmorphousLactose(V_A) = 2\left(95A^{\circ}*\pi\left(2.0*10^{-8}m\right)^2\right) = 2.46*10^{-15}m^3$$

Equation 5.3

$$PercentAmorphous = 100\frac{V_A}{V} = 0.0073\%$$

Equation 5.4

The detection device used for measuring the amorphous content of the two samples was the TAM. It has already been previously stated that, this has limits down to the order of 0.5% for accurate detection. The method is discussed in section 5.2. Based on the value calculated in Equation 5.4, it was not possible to show that amorphous lactose forms after a single impact, due to the small amount formed. In the case of the sample from the pneumatic conveying line, it is likely that detection was possible because the buildup is the result of particles hitting and smearing amorphous lactose against the surface. Amorphous lactose formed from mechanical disruption is most likely to form on the particles surface. When the particles smear against the pipe walls it is this surface layer that is left behind. This makes up only a small percentage of the total particle mass, but will contain a higher concentration of amorphous lactose.

Another way of looking at the potential amount of amorphous formation from a single impact, is to use the kinetic energy (KE_P) contained in a particle when it impacts against a solid surface. The working for this shown in equations 5.5-5.10

Assuming it is travelling at 25m/s, this is just higher than the point predicted for fracture to occur, for the 400 μm particle diameter crystals.

Mass of particle (m) = $5.159*10^{-8}$kg

$$KE_P = \frac{1}{2}mv^2 = 1.61*10^{-5}J$$

Equation 5.5

Energy required to Melt Lactose (E_M) = $\Delta T*c + \Delta F$ = 1829.2kJ/mol

Equation 5.6

Number of moles that can be melted (n_m) from assuming all KE_P goes into melting

$$n_m = \frac{KE_P}{E_M} = 8.88*10^{-12}$$

Equation 5.7

$$Moles\,in\,crystal(n_C) = \frac{m_C}{M_L} = 1.5*10^{-7}$$

Equation 5.8

$$Mass\,Melted = n_m * M_L = 3.17*10^{-9}\,g$$

Equation 5.9

$$Percent\,Melted = 100\left(\frac{n_m}{n_c}\right) = 0.0062\%$$

Equation 5.10

The actual amount of amorphous lactose formed will be less than this as not all the energy of impact goes into breakage. This number is lower than that estimated by using the calculated depth for the formation of amorphous lactose. The results are close enough, to indicate that the levels of amorphous lactose present can be estimated from the energy calculation. An error in the energy of impact calculation is that, it assumes that breakage occurs across the maximum surface area, where in fact the breakage will occur across a variety of surfaces areas.

5.3.2 Heat Generation

In order for crystalline lactose to become amorphous lactose, enough energy has to be supplied to cause the lactose to melt. It can be seen in Table 5-1 that the melting point of α-lactose monohydrate is 201-202 C°. The typical temperature of the conveying lines is 35-40 C°, so in order for the lactose to melt there needs to be another energy input.

Properties	**α-Lactose monohydrate**	**β-Lactose**
Melting point (MP)	201-202 C°	253 C°
Entropy	0.415 kJ K^{-1} mol^{-1}	0.386 kJ K^{-1} mol^{-1}
Enthalpy of Formation (ΔH)	-2481 kJ mol^{-1}	-2233 kJ mol^{-1}
Gibbs Free Energy (ΔF)	-1750 kJ mol^{-1}	-1564 kJ mol^{-1}
Specific Heat (c)	0.45036 kJ K^{-1} mol^{-1}	0.408 kJ K^{-1} mol^{-1}
Density (ρ)	1540 kg m^{-3}	1590 kg m^{-3}

(Roelfsema *et al.*, 2002)

Table 5-1 Physical Properties of Lactose

The main source of energy that a lactose crystal being conveyed in a pneumatic conveying line has, is kinetic energy. During impact, this energy is available for conversion to other forms of energy. For a perfect elastic collision, particle kinetic energy is converted into potential energy then back into kinetic energy on recoil. In a inelastic collision, only part of the potential energy becomes kinetic energy again, the rest is lost as heat due to the permanent deformation of the particle (He, 1999). The breakage of a lactose crystal represents permanent deformation of the particle, energy is required to break the bonds between the molecules and create new surfaces on the crystals.

When breakage occurs, it has been observed by Weichert, (1976) and Fuller *et al.*, (1975) that a large temperature rise occurs across the fracture zone. For sucrose this temperature rise was measured to be 2500K (Weichert, 1976).

It is stated above that energy is released from the breakage of the intermolecular bonds within the lactose crystal. Modelling of the intermolecular bond strengths of α-lactose monohydrate crystal, showed the total lattice energy (E_L) between one molecule and the 256 other molecules that surround it is 151.5kJ/mol (Clydesdale *et al.*, 1997). It can be seen from Equation 5.11 that even assuming that all this energy is released not enough energy is provided to generate the temperatures of up to 2500C° predicted by Weichert, (1976) for the fracture of sucrose.

$$\Delta T = \frac{E_L}{c_L} = 336K$$

Equation 5.11

Equation 5.11 only shows the potential temperature rise assuming that no melting occurs. For the formation of amorphous lactose to occur melting is required. To allow for the melting of the lactose, the 1750 kJ mol^{-1} of energy required for the heat of fusion (ΔF) needs to be included in Equation 5.11. When this energy for melting is included, Equation 5.12 shows that the breakage of the bonds between the molecules only provides enough energy for 8.28% of the surface to be melted.

$$\%Surface\,Melted = 100\left(\frac{E_L}{(\Delta T * c_L) + \Delta F}\right) = 8.28\%$$

Equation 5.12

The dimensions of a α-lactose monohydrate molecule are given by Beevers & Hansen, (1971) to be a = 7.815A°, b = 21.567A°, c = 4.844A°, the dimensions are shown in Figure 5-5. The 8.28% calculated is for the surface molecules with a maximum depth of 21.567A°. This is well below the predicted 98.5A° depth for the formation of amorphous lactose layer.

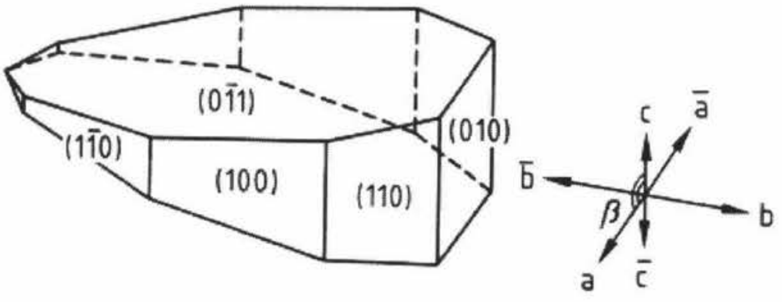


Figure 5-5 Diagram of α-Lactose Monohydrate Crystal (Roelfsema *et al.*, 2002)

This means that the energy released from the breakage of the bonds between the molecules does not provide enough energy solely for the amount of amorphous lactose predicted to be formed. In order for the predicted amount of heating and thus amorphous formation to occur, heat generation must result from other mechanisms.

5.3.2.1 Other Mechanisms of Heat Generation

The temperature rise across a crack was shown by Weichert, (1976) to be predicted by Equation 5.13.

$$T = k\left(\frac{\Psi}{c * \rho}\right)$$

Equation 5.13

Where k is a constant, Ψ the Vickers hardness, c the specific heat and ρ the density of the material.

This equation predicts that a harder material will have a higher temperature rise than a soft material. This means that rather than the temperature rise being dependent on the bond strength between molecules it is dependent on the physical properties of the material and its responses to stress. An example of this is rubber, which has a complex bond structure, but can easily dissipate energy. Harder substances may have less complex bond structures but are less able to dissipate energy and so energy across the fracture zone is focussed over a small mass, resulting in high temperatures.

Fuller *et al.*, (1975) showed that it is the crack velocity that determines how much heat is evolved per unit of surface area across the fracture zone. The kinetic energy provided by the velocity of the crack provides another source of energy for heat evolution.

When a particle impacts a surface, the kinetic energy is converted to strain energy, producing elastic deformation. Once a level of impact energy is provided, the strain energy will no longer just produce elastic deformation but will also induce internal cracking within the solid. When internal damage occurs part of the solid is relaxed and the strain energy converted to heat, in addition to that used to break the bonding between the fresh internal surfaces (Tavares & King, 2002). It is this conversion of energy across the crack that allows the generation of the high temperatures measured by Weichert, (1976)

An estimation of the temperature rise for a 400μm lactose particle travelling at 25m/s is shown in Equation 5.14 to be 7041K. This value assumes that all the heat is put into the layer of amorphous lactose formed.

$$\Delta T = \frac{KE_P}{n_c * c_L} = 7041K$$

Equation 5.14

This seems high when compared against the 2500K value measured for sucrose. If it is considered that a large percentage of this energy will be taken up by other energy sinks such as elastic and plastic deformation of the lactose or will remain as kinetic energy, the value becomes more realistic.

What is shown by this section is that it is possible for the amorphous lactose to form at the velocities being used in the pneumatic conveying line. The formation is a result of not only the breaking of the bonds between the molecules but also the way in which the kinetic energy is focused across the fracture zone. How this energy is focused and how much there is present will have a influence on the heat generation during breakage and thus the amount of amorphous lactose formed.

5.3.3 Particle Effects

The formation of amorphous lactose not only has the effect of making the particles more likely to adhere due to its sticky properties when T_g is exceeded, it also increases the tendency to remain on the surface in other ways. Amorphous lactose not only makes the particles sticky but is also effects their physical properties.

When lactose has amorphous lactose on the surface, if the heat energy generated upon impact becomes high enough to raise the temperature above the glass transition temperature, the surface becomes easily deformed. This reduces the recoil force and increases the possibility of sticking. Particles that are softer are more prone to agglomeration due to the fact that they are more deformable. The binding force becomes greater, not only due to the increased contact area, but due to the sticky nature of amorphous lactose in its rubber state.

The melting also provides a heat sink that means that it is less likely that a particle will rebound from the surface after impact has occurred. The more kinetic energy that is dissipated as thermal energy, the less is available for kinetic energy of recoil (Gugan, 2000). When breakage or plastic flow occurs, this results in the generation of thermal energy, leaving less available energy to break the forces of adhesion.

5.4 Other Adhesion Effects

Buildup on the surface of the conveying pipes is not solely be attributable to the formation of amorphous lactose. When a conveying pipe was removed for inspection of buildup, the entire internal surface of the pipe was coated. A large amount of this adhesion was not permanent and could be brushed from the surface and the powder still flowed as individual particles. This represents more of an electrostatic bonding, due to the small size of the particles.

This same effect was observed in the experiments using the impact-testing device. A fine build up of powder on the walls of the collection chamber was visible after a test had been completed. This powder was easily wiped from the walls, unlike the buildup on the target plate, which had to be scraped or washed off, due to the more permanent bonding that had occurred.

On contact, a large rough particle will generally lead to a higher number of contact points being established with a rough plane surface. Thus, due to a greater true contact area greater adhesion can result (Lam & Newton, 1992). Coarser particles were shown by Lam & Newton, (1992), to be more easily removed than finer ones, because as the particle size decreased the detachment force decreased at a greater rate than the adhesive force.

When powder contacts a surface, the greater surface area of the large particles makes adhesion more likely. The larger particle size also makes detachment easier. However, the impact can result in the detachment of asperities from the surface. These detached particles then remain behind when the larger particle is removed. A similar mechanism is likely when amorphous lactose is present on the surface of a large particle. The energy of impact may generate enough heat for the amorphous lactose to exceed T_g and flow, thus sticking the particle to the surface, the particle may then be removed, leaving the amorphous lactose behind. This is similar to the mechanism discussed in section 5.3, as an explanation for the higher concentration of amorphous lactose shown in the sample of smeared powder taken from the line.

5.5 Conclusion

It has been shown that the formation of amorphous lactose plays a role in the buildup of lactose on the walls of the conveying pipe. The sample taken from the pipewall confirms this, with a higher concentration of amorphous lactose appearing in the smeared lactose than was identified in the free flowing powder. The presence of amorphous on the target plate after impact testing also shows that amorphous lactose is smeared on a surface during impact.

Attempts to show this same result using the testing methods that have been used in the previous chapters have been only moderately successful. In an attempt to understand why, a series of calculations was carried out. These showed that it is likely that amorphous lactose does indeed form but only in very low amounts, below those that are detectable using the methods available. This lead to the conclusion the detectable levels in the built-up powder are a result of the amorphous lactose on the surface of the being smeared on the walls of the pipe. The main crystal then detaches, meaning the powder on the wall has a higher amorphous concentration.

Ultimately what has been demonstrated is that higher energy not only results in more breakage but it also appears from the calculations that when breakage occurs, it will result in more heat generation across the fracture zone, meaning more amorphous lactose will be formed. This leads to the conclusion that the best way to stop amorphous lactose forming is to reduce the amount of energy available at impact. This has two possibilities;

1) Use lower conveying velocities.
2) Provide a surface in the conveying lines that will adsorb most of the impact energy.

Lower conveying velocities means lower conveying rates, assuming the same loading is used. As a result the second possibility provides are more desirable option. This leads to the next chapter, where a rubber lined bend was tested to see how it performed in preventing the buildup of lactose.

Chapter 6 Using a Rubber Lined Bend to Prevent Buildup

6.1 Introduction

The effectiveness of rubber as a method of reducing the breakage of lactose crystals was shown in chapter three. Work in the chapters three, four, and five, shows that the breakage and buildup are a function of how the kinetic energy contained in a particle is dissipated during contact.

Rubber was identified as a material that has potential for use in preventing buildup. This is due to its ability to dissipate this energy, in a manner that reduces the amount of heat input into the lactose crystal. The only way to test the usefulness of the rubber was to trial it in an actual conveying line. This chapter provides a further look at the physical properties of rubber and presents a discussion of the results on how a rubber lined bend behaved when in an actual conveying environment.

6.2 Physical Properties of Rubber

A study of the ability of rubber to resist adhesion identifies that it is due to more than its ability to remove/adsorb the energy of the impacting lactose crystals. It is also a function of the way in which rubber behaves when it is impacted. Keuter & Limper, (2001) showed that in the conveying of Carbon Black the addition of a rubber liner to the inside of the pipe prevented adhesion of the particles to the pipe wall. Where adhesion did occur it was not present for long, due, it was concluded to be a result pulsating nature of the rubber.

Work carried out by Schallamach, (1971) found that the sliding of a solid across a soft rubber surface produced waves of detachment, or sections in which the contact area between the two solids is lost. These waves of detachment were in effect a fold in the rubber which travelled though the rubber in a ripple motion.

As a method of explaining the phenomenon discussed above the following analogy is presented. It can observed that when two objects, one hard and the other soft, are struck the resonation of the softer surface is more visible to the human eye. This is demonstrated by considering two examples at the extremes; a bowl of jelly and a lump of steel. When hit with a force, both objects are known to distribute the energy in the form of waves through the object. The waves on the jelly are large and comparatively slow and can be seen as the "wobble" typically associated with jelly. The waves on the steel move faster and are smaller such that they are difficult to detect by the human eye. They are present, however, a fact that is clearly illustrated in the form of sound when a tuning fork is held to your ear.

These larger, more visible waves may be one reason why the build up of lactose on the surface of the rubber is less. The waves moving through the softer rubber surface and the constant stretching of the surface cause any lactose that has built up to be removed. Where as with a stainless steel pipe wall the elasticity of the two materials is closer and so any built up lactose is able to adsorb any movement that may occur of the stainless steel wall.

The speed, (u_L), of a longitudinal wave in a solid rod is related to its Young's modulus, (E), and density (ρ) as shown in Equation 6.1 (Young & Freedman, 1996).

$$u_L = \sqrt{\frac{E}{\rho}}$$

Equation 6.1

The deformation leading to the detachment of adhered material from the surface of rubber is also illustrated by rubbers Poisson ratio. As stated in chapter three, Poisson's ratio, (ν), is the ratio of lateral strain, (ε_{lat}), to longitudinal strain, (ε_{long}), produced by a single stress. For rubber this is close to 0.5, which means that when a force is applied to the rubber it will distort in both the longitudinal and latitudinal directions. Harder substances, such a glass and steel, have much lower Poisson Ratios which means that when subjected to a force they distort much less in the side ways direction.

As a result, any particles stuck to them are able to adhere to a relatively constant surface area, when compared with rubber.

Not only does the constant deformation of rubber have the potential for removing adhered material from the surface of rubber. The initial deformation that occurs when a particle impacts against a rubber surface also serves to prevent adhesion.

As discussed in chapter three an elastically deforming surface has the ability to mould around the surface of a harder surface impacting against it. This has the effect of reducing the effect of any defects in the surface of the harder object. With the result being that the force of impact is more evenly distributed through out the impacting surface.

The result of the rubber moulding around the impacting object means there is the potential for bonds to form between the two contacting surfaces. Upon the removal of the forces the elastic nature of the rubber means that it has a desire to return to its original form . The compressed rubber relaxes and then flattens out. As this occurs bonds which have formed will be placed under stress. Unless the adhesion forces between the two solids are great enough, or equally able to deform elastically, it is likely that the bonds will be broken. When a solid such a rubber is squeezed rapidly, a larger amount of elastic energy is stored at the local deformation interface. It is this elastic energy that is used to return the rubber to its original form and break any bonds that may have formed (Persson & Tosatti, 2000).

The elastic response and deformable nature of rubber was found by Gay, (2000) through modelling and experimental work, to reduce the adhesion of a sphere to the rubber surface. When contact between an object and a rubber surface occurs the extension becomes an interplay between attractive forces, the external load and elastic restoring forces (Gay, 2000).

In the case of lactose impacting against stainless steel the majority of the deformation will be in the lactose crystal. Where amorphous lactose is present and enough energy is available, the deformation will occur as flow of the amorphous solid. As there is no stored elastic energy there is no driving force for the bonds between the two surfaces to be broken. Once the heat energy is dissipated to the environment the lactose returns to the solid state forming a permanent bond.

6.2.1 Wear

As this chapter is primarily focused around the potential for the use of rubber in a conveying line, it is considered appropriate to develop further the discussion on wear of rubber that was begun in chapter four. The preceding section focused on how the deformable nature of rubber gave it properties that make it less prone to adhesion. Mills, (2000) notes that resilient materials such as rubber and polyurethane are widely used in erosive wear situations. Although the hardness of the surface is lower than the material being conveyed and impacting against the surface, they derive their resilience from the fact that they are able to adsorb most of the impact energy by virtue of their resilience

An explanation of this resilience lies in the ability of elastomers to form huge numbers of combinations of the elastomeric chains. When a piece of steel is drawn, it will soon come to weakening and eventual destruction of the covalent bonds between the atoms. When rubber is drawn, rotations and other changes result in new conformations, but the primary bonds are preserved (Brostow *et al.*, 1996). When the energy is released the rubber is then able to return to is original form, due to the chain nature of the molecule, which due to energy considerations, would rather relax than fracture, as it takes about thousand times more energy to break the carbon-carbon bond that execute a conformational rearrangement (Brostow *et al.*, 1996).

In addition to the resilience from the conformation of the molecules, the Lake-Thomas effect shows that the fracture energy of polymeric materials is amplified. This is because in order for the fracture of one bond to occur, all the polymer bonds in the vicinity of the fracture need to be stretched to breaking point, even though only one bond breaks (Ghatak *et al.*, 2000).

However, rubber is not a perfect material and is prone to cutting where sharp hard materials can tear or cut the rubber (Reed & Bradley, 1992). Mills, (2000) notes that when fine sand and lump coke were conveyed through rubber lines bends, little wear was observed for the sand which is very hard when compared to rubber. Yet considerable wear was reported for the lump coke. The lump nature of the coke probably provided surfaces that can cut the rubber and the size gave it sufficient force to penetrate.

It also is possible for conditions such as sunlight and oxidation to lead to the formation of a brittle surface layer on rubber . This brittle layer is relatively easily removed by the stresses the rubber is exposed to under practical use (Persson & Tosatti, 2000).

This section further highlights that in terms of resisting and adhesion wear rubber has some unique properties that give it an advantage over other materials. However, as noted in the introduction to test comprehensively these properties requires that the rubber is trialed in a real conveying environment. This is the focus of the next section.

6.3 Rubber Conveying Trial

The conveying system studied, was the dry process conveying line (BM3) at the Lactose New Zealand factory in Kapuni. The conveying system is a vacuum conveying line. Lactose powder is feed from a hopper into the conveyed line, using a rotary valve. The system has been designed to operate as a slug flow system, with air injection being used to separate the slugs. As the lactose powder progresses further up the line and the conveying velocity increases, the powder leaves this slug formation and enters a more lean phase conveying mode.

Prior to entering this section of the conveying line the lactose has undergone a large number of processes. These processes take whey permeate and then using crystallisation and a variety of other unit operations, produce crystalline α-lactose monohydrate. The three unit operations directly preceding the entrance of the lactose into the conveying line are, in their respective order, drying, milling and sieving. These are mentioned separately as they are considered to have the most bearing on the properties of the powder that relate to this work.

The specifications for the conveying system are presented in Table 6-1. It should be noted that the measurements for the gas velocity were made during a shut down period with the line being open to atmosphere. The increase in velocity at the end of the line is due to the gas expansion that occurs due to the pressure drop across the line. When powder is conveyed this pressure drop is increased considerably. It is assumed that, because of this the actual conveying velocity will be much higher than the values displayed in Table 6-1.

System Type	Vacuum Conveying
Operating Air Temperature	35-40°C
Feed Rate of Lactose	Approx. 2.5-4.0 tons per hour
Gas Velocity	11-13m/s(start) 15-17m/s(finish)
Typical Particle Size	500-10μm
Pipe Diameter	0.1m
Relative Air Humidity	25-30%
Pressure in Line	-0.5 Bar (conveying) –0.8-0.9 (when not)

Table 6-1 Conveying line specifications

6.3.1 The Bends

Two bends were used in this study. Both were located on the BM3 conveying line, on the route from the mill to the main hopper before packing. One was an "S" bend in a vertical section of the conveying line. The other bend was a long radius 90 degree bend that went from a horizontal to a vertical section in the conveying line. The bends were identified by plant operators as sections of the conveying line where buildup has been a problem in the past.

In the first stage of the trial both bends were removed from the line during a shut down period . They were washed using hot water to remove all previous buildup. They were then dried to remove any excess moisture that may have been present. Each bend was then individually weighed using a balance (Toledo Model 8138) accurate to 0.01 kg. This weight was recorded and the bends were then returned into the conveying line.

The bends were removed from the line every three days to be examined for buildup and reweighed. The three day period coincided with the shut down cycle of the dry process section of the factory. This was the only time available when bends could be removed and examined freely. Towards the end of the trial some time periods between sampling were longer than three days. This was deliberate, and was done to ensure that the constant removal and examination of the bends was not having an effect on the buildup of the lactose.

As the study was concerned with powder that had become solidly adhered to the pipe wall, each bend was blown with compressed air to remove loose powder, before weighing.

After 21 days of observing the buildup of powder in the conveying line the 90 degree bend was removed from the line. This was so that it could be lined with rubber for the second stage of the study. An alternative bend was put in place to allow the plant to continue operating. The study of the "S" bend was continued with weight changes and buildup being observed.

The second stage of the trial was to compare the results of the buildup on a stainless steel bend against a stainless steel bend lined with rubber. The rubber used was 0.004m thick Linatex FG, as this showed, in Chapter Four, to be the most promising in terms of resisting wear. The "S" bend was left as a stainless steel bend, whist as mentioned above the 90 degree bend was lined with rubber. Both bends were cleaned with hot water and dried in the same manner as in the first trial. They were then both weighed and then placed in the conveying line.

The method of monitoring was the same as in trial one, with the bends being removed from the line and examined for changes every three days. The one exception to this was that, the rubber lined, 90 degree bend, was removed briefly from the line each day for the first three days. This was done to look for any signs of wear or detachment of the rubber from the stainless steel pipe.

6.3.2 Results

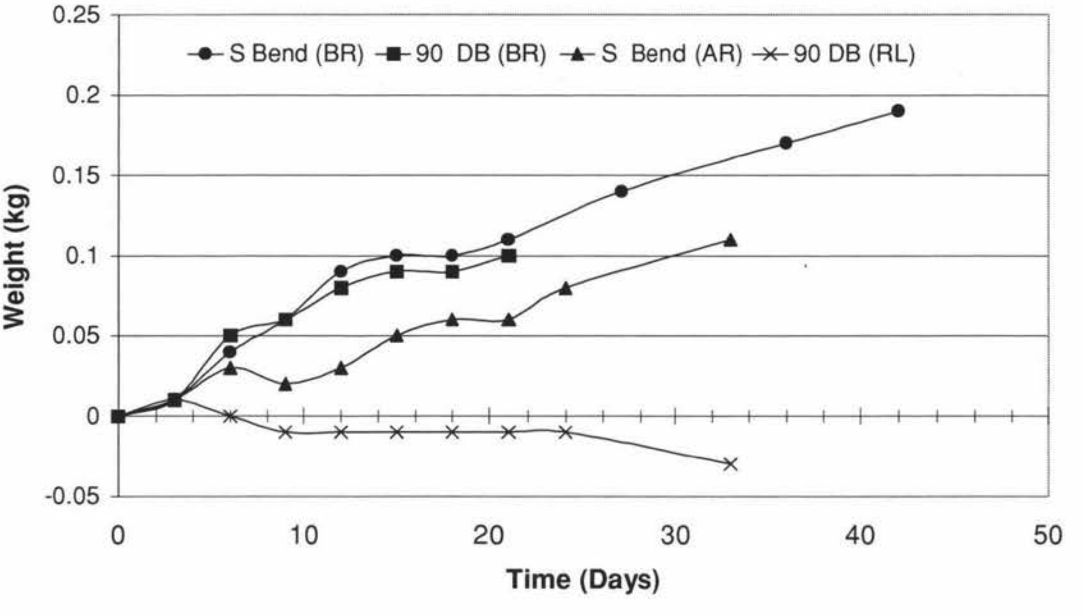
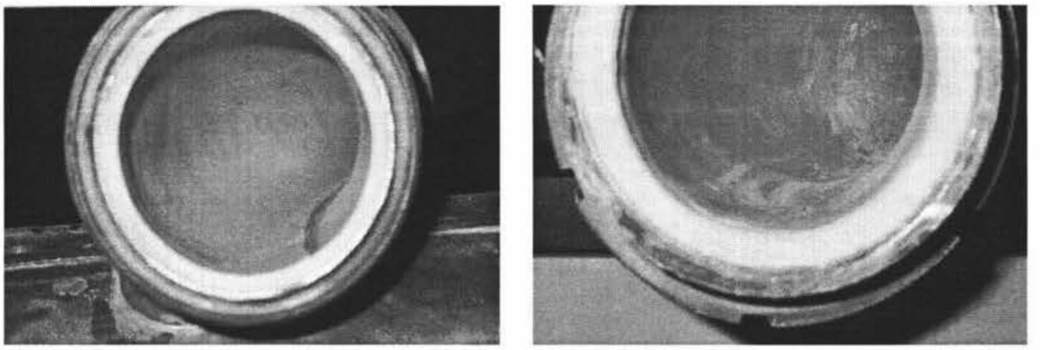


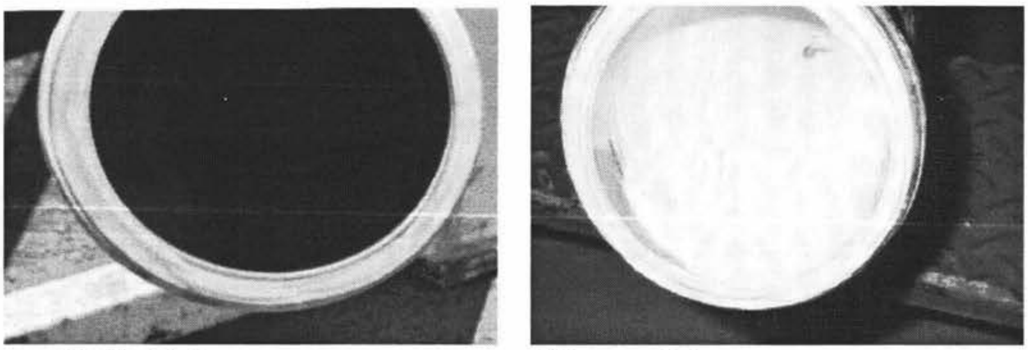
Figure 6-1 The buildup patterns of the two bends studied before rubber (BR), after rubber (AR) and with a rubber lining (RL)

It can be seen in Figure 6-1 that the weight change due to buildup follows a relatively constant path throughout the time that it was monitored. Also, the weight change for the "S" bend and the 90 degree bend are very similar in both mass and in their trend.



Before (a) After (b)

Figure 6-2 Images of the rubber lined bend before use and after 21 days in the line



Washed (a) After use (b)

Figure 6-3 Images of the "S" bend used at the same time as the rubber bend after washing and after 21 days in the conveying line.

The results obtained, shown in Figure 6-1 and Figure 6-2, for the rubber lined bend show it is effective at preventing the buildup of lactose. As predicted by the theory above, there was at no time during the trial period any lactose adhered to the surface of the rubber. A small surface layer of lactose was recorded on the surface of the rubber during the visual inspections, seen in Figure 6-2(b). This was easily removed through brushing the surface and had not formed any solid bonds. The weight change over the trial period confirmed this, with no increase occurring. What is of concern is the small decrease in weight, due to wear, that occurs at the end of the trial. This is discussed in more detail below.

The results seen in the rubber lined bend can be compared against the "S" bend, seen in Figure 6-3, where the buildup still occurred in a similar manner to the pattern shown by the first trial. The results from the weighing and visual inspection allow the conclusion to be drawn that it was the rubber that was preventing the buildup and not some change in conditions within the plant.

6.3.3 Wear of the rubber bend

It has already been discussed above that in the second trial, buildup in the rubber lined bend based on the weight change data is zero. What can also be seen in Figure 6-1 is a small increase in weight at the start, and then a small decrease in weight, the weight then remains constant. Then at the period between day 24 and day 33 there is a weight change of 20 grams. Visual inspection of the bend showed that at the entrance to the bend there had been a small amount of wear. This wear was due to a defect in the design of the bend and the position of the rubber lining.

All the wear that was seen was focused around a relatively small area of the bend at the entrance. In this section the thickness of the rubber meant that it was raised above the surface of the stainless steel and protruded into the flow path of the powder. The wear appears to have began at the corners of this section and moved inwards. A diagram of the protruding rubber and wear at the corners is shown in Figure 6-4.

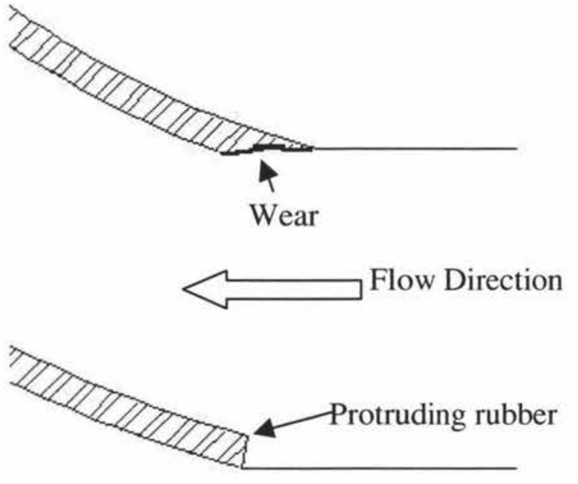


Figure 6-4 Diagram showing the rubber lining and wear that the corners

The corners of the rubber are, considering the wear resistance theory in section 6.2.1, at a disadvantage in terms of their ability to resist wear for two main reasons.

- The first is that they have a smaller area than the main body of the rubber to dissipate energy though. This means that a greater amount of energy is focussed on one section and more energy is provided for the breaking of the bonds between the rubber molecules.
- The second reason is that the molecules are surrounded by less molecules and as such there is less energy required to stretch the rubber and break the molecular bonds.

The combination of these two factors present a reason for the rubber wear shown in the trial. For future design two possible solutions have been considered. The first is to cap the corners with a stainless steel plate, this would be to prevent the exposure of the rubber. The second alternative is to use a slightly larger pipe so that the rubber lining becomes flush with the main pipe. These solutions of course would require further trialing to test their effectiveness, as they may generate unforeseen problems.

Section 6.2.1 also noted the rubber can form a brittle layer as a result of exposure to sunlight and other environmental conditions. The exact handling and storage of the rubber prior to its use in the bend are unknown. The removal of this layer may provide an explanation for the small decrease in the weight of the rubber bend observed in the early stages of the trial. This decrease may also have been an error in a previous weight measurement taken.

6.4 Conclusion

This chapter has demonstrated that the use of a rubber lined bend to prevent the buildup of lactose on the walls of a pneumatic conveying line has potential. In terms of preventing buildup the rubber was completely successful, with no lactose found sticking to the surface during the entire trial period. This confirms what the work in the earlier chapters, which found that the adhesion of lactose to a surface is effected by the way in which the energy at contact is dissipated, and the ability of the contact surface to form permanent bonds with the lactose. Both of these factors are a function of the way in which rubber deforms during the impact period.

Further testing and design of the rubber bend needs to be carried out to confirm its suitability. A small section of wear did occur at the entrance to the bend. This was in a region where high levels of impact were focused on an area that had a reduced ability to transfer the energy. A change in the design at the entrance to the bend may allow this problem to be solved and allow the wear to be prevented. Should this be the case, it would allow the rubber lining of bends to be successfully used in preventing the problem of lactose buildup.

Chapter 7 Conclusions and Recommendations

The attrition of lactose as a result of impact is controlled by the amount of kinetic energy the particle has before impact. The velocity and the size of the particle are the major factors controlling this energy. The physical properties of the material against which impact occurs have an influence over the way in which this energy is transformed and can control the size of the impulse force the crystal is subjected to.

Using these three factors it is possible to control the amount of attrition that lactose undergoes upon impact. Changing the impact surface from a hard rigid material such as stainless steel, to one which is soft and easily deformable, allows the average impact force to be reduced and as a consequence of this the attrition is also reduced. Lower velocities and smaller particle sizes also have the effect of reducing the amount of attrition.

The attrition that is observed when lactose undergoes impact was found to occur through two mechanisms, abrasion and fracture. Fracture occurred only when the impact force was high enough. This mechanism was shown to occur through both single particle impact tests and in a pneumatic conveying line.

The results from the single particle tests could be used, to some extent, to predict the pattern of breakage that will occur in a pneumatic conveying line. This needs further work to allow a full understanding of the breakage pattern of lactose crystals to be developed.

Experimental work showed that, lactose could be made to smear on surfaces through impact and also through the sliding of lactose particles across a surface. Both of these conditions involve the breakage of lactose. The sliding is breakage on a more microscopic scale than observed in the impact testing.

Amorphous lactose was found in higher concentrations in the lactose powder that had become smeared on the pipe walls than it was in the free flowing power. This is concluded to be a result of the amorphous layer transferring from the surface of the crystal to the surface of the pipe wall. Amorphous lactose was also shown to be present on the target plate after impact. However attempts to show that amorphous lactose was present in the lactose after breakage had occurred proved inconclusive. This was determined to be a result of the small amounts formed during the breakage of crystals being undetectable using the current methods available. The calculations used to determine this highlighted the strong relationship between impact energy and breakage that was identified by the impact testing results.

An attempt to use a rubber lined bend to reduce the buildup in the conveying line proved to be very successful. It was effective at preventing the adhesion of the lactose to the surface of the pipe. However, wear was observed in a section of the rubber lining. The location of the wear indicates that this was the result of a design defect. More testing needs to be done to allow a conclusion on the applicability of rubber for preventing the buildup of lactose in the conveying line.

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Appendix

Nomenclature

A	Radius of deformed region	M
C_F	Correction Factor for particle size	
C_L	Specific Heat α-lactose	kJ K^{-1} mol^{-1}
d_a	Depth of amorphous layer	M
d_{sv}	Diameter of particle	μm
E	Young's modulus	GPa
e	Coefficient of restitution	
E_1	Young Modulus of Impact Surface	GPa
E_2	Young Modulus of Impacting Particle	GPa
E_L	Lattice Energy	J
E_M	Energy to Melt α-lactose	J
F_{av}	Average Force on Impact	N
F_F	Friction Force	N
h	Half thickness of impact surface	m
I	Number of impacts	
J	Momentum	kg.ms^{-1}
KE_P	Kinetic Energy of Particle	J
m	Particle mass/crystal	kg
m_A	Mass flow rate of air	kg/s
M_{An}	Mass of Particles at sieve size	kg
M_{AS}	Mass of Impacted Crystals after sieving	kg
M_{BS}	Mass of Impacted Crystals	kg
m_c	Mass of crystal	kg
M_{Dn}	Mass of fines at size n with fracture included	kg
M_{In}	Mass of fines in sample before conveying	kg
M_L	Molar Mass of α-lactose	g
M_{na}	Mass of fines at size n from abrasion	kg
M_{nac}	Mass of fines in sample after conveying	Kg
M_{nf}	Mass of fines at size n from fracture	kg
M_T	Total Sample Mass	Kg
n_c	Moles on a crystal	
n_m	Number of moles	
P_u	Fraction of fines produced at velocity u	
r	Particle radius	m
S_{am}	Surface area of powder	m^2
SLR	Solids Loading ratio	Kg/kg
t	Time	s
T_g	Glass transition temperature	C°
t_I	Time of impact	s
u	Velocity of crystal/particle	m/s
u_L	Speed of Longitudinal wave	m/s
V	Volume of Crystal	m^3

V_A	Volume of amorphous lactose	m^3
V_L	Volume of Lactose	m^3
V_n	Normal Velocity of Impact	m/s
V_p	Parallel velocity of Impact	m/s
V_v	Vertical velocity of Impact	m/s
W	Normal Force	N
α	Alpha factor	M
ε	Strain	
ε_{lat}	Lateral strain	
ε_{lon}	Longitudinal stain	
π	Pi	
μ	Friction Coefficient	
ρ_1	Density of impact surface	kg/m^3
ρ_2	Density of Impacting Particle	kg/m^3
ρ_B	Bed density	kg/m^3
ρ_g	Gas density	kg/m^3
ρ_L	Density Alpha Lactose	kg/m^3
ρ_s	Solid density	kg/m^3
σ	Stress	N/m^2
v	Poisson's ratio	
v_1	Poisson Ratio of Impact Surface	
v_2	Poisson Ratio of Impacting Particle	
Ψ	Vickers hardness	GPa
ΔF	Heat of Fusion	kJ/mol
ΔT	Temperature Change	K