

PARTICLE ENCAPSULATION

by

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Master of Engineering

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DECLARATION

I hereby certify that this material, which I now submit for assessment on the programme of study leading to the award of Master of Engineering, is entirely my own work and has not been taken from the work of others save and to the extent that such work has been cited and acknowledged within the text of my work.

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To my parents...

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Project Title: Particle Encapsulation

By Xiaobin Sun, B.Eng

ABSTRACT

Several engineering processes are powder based, ranging from food processing to engineering ceramic and composite production. In most of these processes, powders of different composition are mixed together in order to produce the final product, and this combining of powders of different density, shape, and surface properties is often very difficult. Mixtures may be quite inhomogeneous. This research focuses on a method of avoiding such problems, by coating individual particles of one material, with a relatively very thick layer of another powder material.

The study is limited to undertaking as a first step, the coating of large, nominally spherical particles with a more fine powder, and consists of two aspects. Experimental work investigates the principle of coating such particles using a 'snow-ball' process, and presents supporting mathematical models to describe the process.

The experimental study has identified particular media (particles, binders and powder), and processing conditions (speed of rolling) most suited to successful coating of large sized particles as a means of demonstration of the principles. It has been found that the process of a single pass through the coating equipment can be modelled using a simple exponential expression, in which constants represent the total increase in diameter, and the rate at which this is reached.

With further experimentation, the experience gained may be applied to encapsulation of smaller sized ceramic particles with metallic matrix materials using suitably modified experimental equipment and procedures.

CHAPTER 1 INTRODUCTION

1.1 INTRODUCTION

This project investigates the process of coating particles. Specifically it focuses on depositing high thickness coatings on nominally spherical particles. The project presents results of a mathematical model for particle coating and an investigation of various factors that affect the particle coating process.

1.2 PROJECT BACKGROUND

Powders are used in industry for a diversity of products, including food, ceramic, metal powder, etc. Traditional powder metallurgy is a process whereby a solid metal, alloy or ceramic in the form of a mass of dry particles, normally less than 150 microns in maximum diameter, is converted into an engineering component of predetermined shape and possessing properties which allow it to be used in most cases without further processing. The basic steps in the traditional process are those of powder production; compaction of the powder into a handleable preform; and sintering, which involves heating the preform to a temperature below the melting point of the major constituent when the powder particles lose their identities through inter-diffusion processes and the required properties are developed. During powder processing, particles are mixed with powders and binders in proportion, and then, they are pressed and sintered. The problems are that the distribution of binder materials and particles are not uniform and the automatic feeding is impossible. For example, in ceramic grinding wheels, it is difficult to make the distribution of the hard particles uniform.

Ceramic grinding wheels are currently manufactured using a conventional compaction and sintering route. The ceramic reinforcement particles are mixed with

powders (copper/brass) and appropriate binders in proportion so that the ceramic particles are distributed within the body of the metallic matrix, the binder material being burnt out during the sintering stage. However, the wheels manufactured using this route have many disadvantages.

Most importantly the distribution of the ceramic particles is seldom uniform within the wheel. For example, the grinding face of the wheel may have clusters of ceramic particles at some locations, and single particles at others with non-uniform gaps between the clusters and single particles. The effect of such distribution is inefficient grinding performance and shorter life of the wheel. In addition, during wheel production, die wear is severe because of the direct contact between the abrasive grains (ceramic powder) and the die wall. Also, the poor flowability of metal powder and the tendency of ceramic particles to segregate from the metal powder make automated feeding impossible, and therefore production is limited to inefficient batch processes.

The grinding performance of a wheel would be significantly increased if the ceramic particles could be distributed within the body of the wheel in a more regular pattern and without having cluster formations. The cutting efficiency would be much higher with single ceramic particles spread at notionally regular distances on the grinding face and at varying depths within the wheel. Obviously, the ideal distribution of ceramic particles may never be achievable. However, some improvement on the current situation is desirable and should be attainable.

The technical breakthrough which will be needed to achieve the above objective is the development of a process by which individual ceramic particles could be encapsulated within bronze/copper metallic shells of wall thickness of about ten times the size of the ceramic particles. Specifically a particle of 60 μm diameter would be contained within a shell of about 600 μm diameter. These encapsulated powders would have several advantages. When compacted and sintered in the normal manner, they would ensure improved distribution of ceramic particles within the metallic matrix of the resulting grinding wheel [1]. The encapsulated ceramic powder would allow

easy handling and automated feeding into a pressing machine. Die wear in the pressing machine would be minimised, because direct contact between the abrasive grains (ceramic particles) and the die wall would be eliminated.

These processing problems and the advantages of the suggested solution are not limited to the application of ceramic grinding wheels. Many other cutting tools and composites produced using powder technology share the requirement for good distribution of reinforcement.

There are a number of technical problems which will have to be overcome by any process in order to have reasonable potential for success. Many of these relate to the very small size of the ceramic particles. Handling of such particles is difficult. In addition, for particles with high surface to volume ratios, surface characteristics rather than bulk material properties tend to govern their behaviour.

Clustering of the particles will have to be avoided to gain advantage over current coating techniques. To achieve this, good separation of ceramic particles will have to be maintained. However, at the same time 'blank' agglomerates of the metal powder (in which no ceramic is present) must be avoided. This can be achieved by having conditions for agglomerations only at the ceramic/metal powder interface. It may not be possible to coat the ceramic to adequate thickness in one operation, and a secondary process, or a re-cycling through the coating process may be necessary. As a result, a separation stage will be necessary in which agglomerates with different properties can be separated and collected. As with other coating techniques, the equipment itself must not be coated to such an extent that its operation is interfered with, or that maintenance is difficult.

The present study investigates the principle of using a simple "snow-ball" approach to achieving the particle encapsulation necessary. This was done through experimentation using a scaled up version of the process. Results were subsequently modelled mathematically.

1.3 THESIS STRUCTURE

The objective of the current project is to examine processes for encapsulating nominally spherical particles within shells of up to ten times their diameter. As such this thesis is divided into a number of sections:

Following from the introduction, chapter 2 summaries the literature survey carried out for this research. This includes discussion of why particles are coated, and techniques used to do so, powder processing (particle size, shape, specific surface...), and a review of powder systems (powder handling, powder/air systems, powder/liquid systems...). Chapter 3 describes the experimental investigations carried out, including descriptions of the materials, processing equipment used and the test procedures carried out. The results obtained from the experimental work and the discussion based on it are presented in chapter 4, together with the composite modelling investigations carried out. Finally, chapter 5 draws conclusions from the present research and presents some proposals for the future development of the work.

CHAPTER 2 LITERATURE SURVEY

2.1 INTRODUCTION

Various types of particles are coated with different materials to impart useful properties. There are also many methods for coating particles. In order to develop the project, it is important to fully understand the motivation for coating particles, powder characteristics (particle size, shape, specific surface etc.), and powder systems in general (powder handling, powder/air systems, powder/liquid systems etc.). Aspects of each of these are discussed in this chapter even though the work presented here is more to do with coating of much larger particles for demonstration of the “Snow Ball” method using non-engineering materials; dried peas were used as the particles and powdered food materials were used as the thick coating layer.

2.2 WHY COAT PARTICLES?

2.2.1 Introduction

For many years traditional materials, such as metallic alloys, polymers and ceramics, have been successfully developed to meet the needs of industry. However, it has been realised that many specialised applications require combinations of materials to optimise properties, such as quality and safety in fuel [2], sintering properties [3] and thermal conductivity of ceramics [4]. etc. The solution is coating powder.

Fine, hard particles such as W, Mo, WC, diamond, and aluminium, are non-free flowing materials. Such powder has proved difficult to press [5]. Furthermore, the high interparticle friction associated with these fine powders makes handling difficult.

Consequently, coated particles are formed to aid flow and compressibility, while facilitating material combination, and improving homogeneity.

2.2.2 Sample Process: Cutting Wheel

Grinding wheels consisting of ceramic particles in a metal matrix are produced at present, using a number of techniques. However all of them have limitations, and that is the reason for the current study. In this section current practice is described, and associated problems and limitations are discussed.

In the first method, metal powders and ceramic particles are mixed in proportion and pressed by manual feeding method. During this process, skilled operator and intensive labour are needed. Due to poor flowability of metal powder and the tendency of ceramic particles to segregate from the metal powder, an automated feeding operation is impossible. In the second method, metal powders are granulated after mixing with binder solvent, then the granulated metal powders and ceramic particles are mixed in proportion and pressed. Automated feeding is possible for the metal powders during this process. During this process, flowability is improved for the metal powders. The disadvantage is: segregation of metal granules and ceramic particles during mixing; due to different size and density, automated feeding results in an uneven distribution, which affects the grade of the tool. In the third method, metal powders and ceramic particles are mixed in proportion, then they are granulated and pressed by an automated feeding method. The disadvantages of this process are that the granulator suffers heavy wear due to direct contact with ceramic particles, and only a small quantity of granules contain only one single ceramic particle while many more contain none or more ceramic particles, resulting in an uneven distribution of ceramic particles in the tool. In the fourth method, ceramic particles are coated with metal powders in proportion by adding some binder solvent, and metal powders are granulated after mixing with binder solvent. Then, the metal coated ceramic particles and granulated metal powders are mixed in proportion and pressed by an automated feeding method. The advantages of this process are: 1. Free flowing granules enable

increased automated feeding into the pressing machine. 2. Uniform distribution of ceramic particles in the tool, resulting in superior tool quality. 3. Die wear in the pressing machine is minimised as there is no direct contact with ceramic particles. 4. Stronger ceramic particles retaining forces resulting in longer tool life [1]. So, the fourth method is the best one. The problem is how to encapsulate ceramic particles singly.

2.3 TECHNIQUES FOR COATING PARTICLES

2.3.1 Introduction

Individual powder particles can be coated by spray drying, chemical processes (hydrochemical reduction), layering agglomeration, a combination process of spray drying and fluid bed, chemical vapour deposition (CVD), sol /gel processes etc. But most of these techniques are only suitable for thin coatings and they are very expensive.

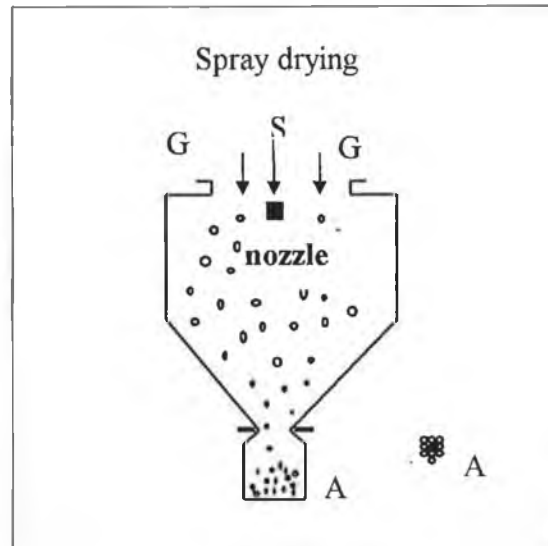
2.3.2 Spray Drying

Spray drying involves atomisation of feed into a spray, and contact between spray and drying medium resulting in moisture evaporation. The drying of the spray proceeds until the desired moisture content in the dried particles is obtained, and the product is then recovered from the air. In the spray drying process, powders are mixed with an organic and a volatile agent to form a slurry. The slurry is atomised and sprayed into a heated free-fall chamber where surface tension causes the formation of spherical agglomerates. Heating of the agglomerate during free-fall causes vaporisation of the volatile agent, giving a hard, dense-packed agglomerate. The resultant granulate is collected in the lower part of the tower. There are two terms used in association

with this process. One is “atomisation”, the other is “aggranulation”. Atomisation is the dispersion of slurry or molten metal into particles by a rapidly moving stream of gas or liquid, or by centrifugal force. Aggranulation is a tendency for fine particles to stick together and appear as larger particles. The bonding mechanisms for aggranulation can be: solid bridges formed by crystallised salts or sintering contacts, immobile liquid bridges formed by viscous binders, adhesives and adsorption layers, mobile liquid bridges (capillary forces), van der Waals and electrostatic forces and mechanical interlocking of powders. The adhesion forces decrease in magnitude in the sequence of this list, and increase with decreasing size of the primary particles [6]. During the spray drying process, the particle shape sequence with distance from the nozzle is cylinder-cone-sheet-ligament-sphere. Depending on the amount of superheat and other variables, any one of these shapes may be produced. Additionally, the turbulence and mixing near the nozzle will cause fine particles to reenter the gas expansion zone. This can result in aggranulation. Atomisation of the slurry is undertaken by centrifugal (rotating disc) atomisation or by single-fluid or two-fluid nozzles, similar to melt atomisation used in the production of metal powders. The granule size is determined by the size of the droplets and the solid content of the suspension. The largest agglomerate sizes achieved are 600 μm with a single-fluid nozzle, and 300 μm with rotating discs, which also yield the narrowest size distribution [6]. The spherical shape and large agglomerate diameter makes flow quite acceptable

Spray drying is the most important practical granulation method. It is used for most ceramic powders, for hard metal powder mixtures and other fine metal powders such as molybdenum. It is also widely used in food and chemical processing, for example, milk and silica white granulation [7]. Processing of these powders requires closed systems suitable for non-aqueous suspensions. Examples of this process are the production of composite metal coated ceramics [8] and cobalt coated WC powder [9], cobalt coated diamond particles which are discussed in detail later. All of the above methods are limited to very thin coatings. Disadvantages are :

The spray drying process is shown in Figure 2.1.



S: slurry; A: agglomerates; G: gas

Fig. 2.1. Coating Particles by Spray Drying

1. the organic binder which remains in the granule must be removed in the sintering cycle and may lead to significant porosity [5].
2. In the specific droplet drying process, spray dried agglomerates are often inhomogeneously packed, and sometimes even take the form of hollow spheres.

However, spray drying offers several advantages over other powder processing techniques, particularly in applications requiring agglomerates for subsequent pressing and sintering operations. Spray drying is a continuous rather than a batch process that allows close control of agglomerate size, bulk density, and moisture content. Agglomerate shape is spherical, facilitating excellent flowability. Particles have very short exposure time. Residence time within the dryer may range from two to twenty seconds, depending on dryer size. Therefore, heat-sensitive materials can be spray dried. Lubricating additives can be added easily for die pressing applications. Spray

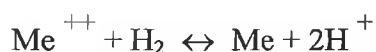
drying is one of the most economical ways of drying slurries. Although the cost of equipment is high, the overall cost of spray drying is relatively low compared to other processes. Oil and power consumption for various sizes of spray drying chambers is a function of evaporative capacity.

2.3.3 Chemical Processes

There are many examples of coating particles using chemical processes, such as $\text{Al}_2\text{O}_3/\text{Al}$ coated MgO , SiO_2 and Fe powder [10-13], Co coated Fe powder [14], carbon coated metal or SiC particle [15, 16], nickel coated alumina or Al_2O_3 particle [17-19], copper coated ceramic particle [20, 21]. etc. All of them are only suitable for thin coating layers.

Hydrochemical reduction (electrochemical process)

Several metals can be produced in powder directly from aqueous (or organic) solutions by reduction with gaseous hydrogen [6]. This process has been developed by Sheritt Gordon Mines Ltd, Canada, especially for the production of nickel and cobalt powder from sulphidic ores containing brass, nickel, cobalt, ferrous and sulphur. A convenient technique is the separation of a brass rich and a nickel + cobalt rich fraction by flotation of the fine ore with a subsequent leaching procedure in autoclaves with NH_4OH under air pressures of 7-9 bar. Nickel and cobalt form readily soluble ammines, ferrous forms insoluble $\text{Fe}(\text{OH})_3$ and the sulphur is transformed to sulphates. Most of the other constituents of the ore are insoluble in ammonia and form a solid phase together with $\text{Fe}(\text{OH})_3$. However, a few other metals, like tin (Sn) and cadmium (Cd) also enter the solution. The solution is then treated with gaseous hydrogen under 28-35 bar pressure at 180-220°C in an autoclave when the following reaction occurs:



The electrochemical conditions for the process are summarised in Figure 2.2. Gaseous hydrogen is able to precipitate metals from their ionic solution, when the H_2/H^+ potential is more negative than Me/Me^{n+} . There is a small dependence of the H_2/H^+ potential on the H_2 partial pressure, but a large dependence on pH value. Thus, most of the metals can be precipitated from alkaline solutions and only a few such as brass and silver from stronger acidic liquids. The precipitation potential of the complex ammine ion of cobalt and nickel is not very different from that of the simple hydrated ions shown in the figure. Furthermore, there is no large dependence of the potential on the ion concentration, which provides the possibility of nearly full deposition of cobalt and nickel from alkaline solutions. This also allows a selective precipitation of metals with a similar reduction potential, at least in a certain concentration range when the H_2 partial pressure and pH are kept constant.

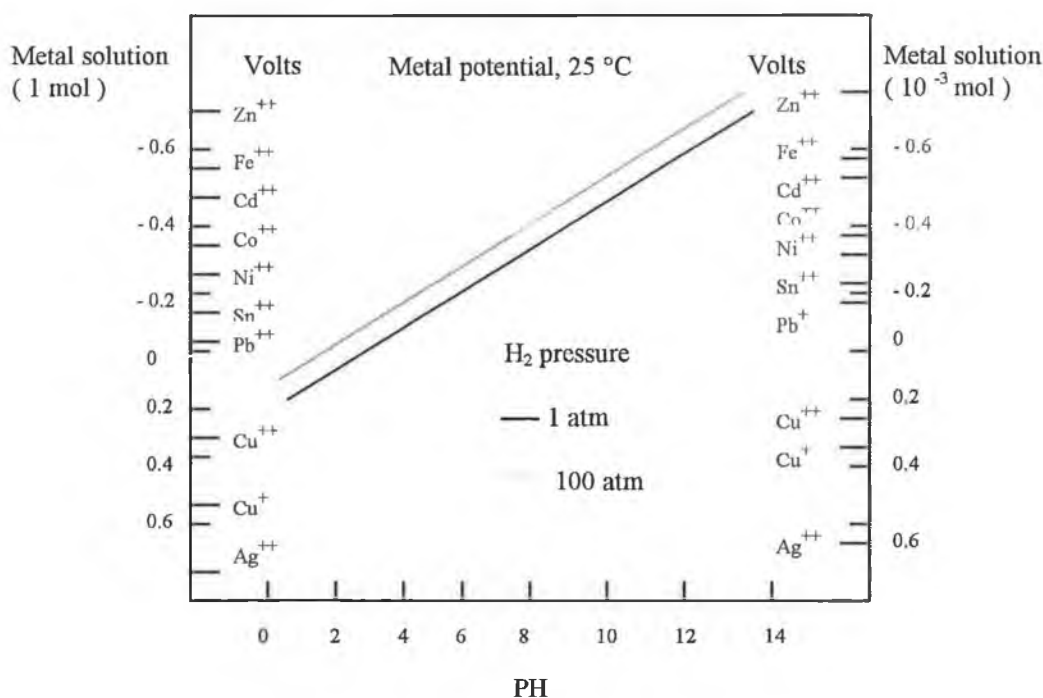


Figure 2.2 Metal and Hydrogen Potentials at Different pH and Different Metal-Ion Concentrations (Schaufelberger) [6]

The purity of the powders is nearly 99.8 % metal content; nickel contains some cobalt and vice versa. Depending upon the stage of the process, coarser or finer particle size distributions can be obtained. Because they are not continuous batch processes, the production rate is low.

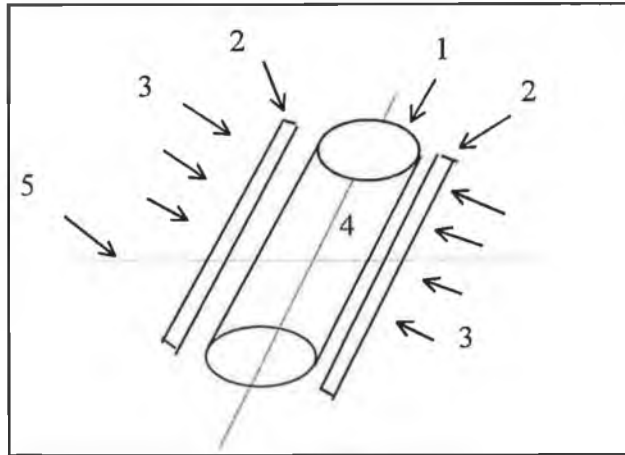
A special application of this process is the preparation of composite powders, when a 'seed powder' is dispersed in the liquid, which has to be kept in continuous motion in order to achieve a homogeneous dispersion. Each powder particle acts as a nucleus and is coated by the deposit. Examples are nickel-coated oxide or graphite and cobalt-coated diamond particles [6].

2.3.4 Layering agglomeration

An tumbling technique [22] of layering agglomeration/aggranulation has been developed to produce composite particles with a thick metal coating which consists of agglomeration fine metal powder on ceramic cores covered with binder. In layering agglomeration, fragmentation and agglomeration occur simultaneously, the latter with a higher probability resulting in increasing agglomerate size with processing time.

An example of this method is coating of ceramic particles with metal powder, which has been found very effective for various kinds of metal powders.

Binder-coated ceramic particles and fine metal powder are loaded in a brass cylinder internally coated with Teflon, and tilted at an angle to the horizontal axis of rotation as shown in Figure 2.3. The tumbling ensures good mixing and a uniform movement of the ceramic particles and of the metal powder. Upon heating the cylinder, the binder reaches a viscous state which allows the formation of a fine powder coating on the ceramic cores (Figure 2.4). In a subsequent step, heat treatment can be carried out to remove the binder and to sinter the powder layer which is then coherently bonded to the ceramic core.



1, tilted cylinder; 2, heating elements; 3, cooling medium; 4, load; 5, rotation axes

Figure 2.3. Representation of the Agglomeration Apparatus [22]

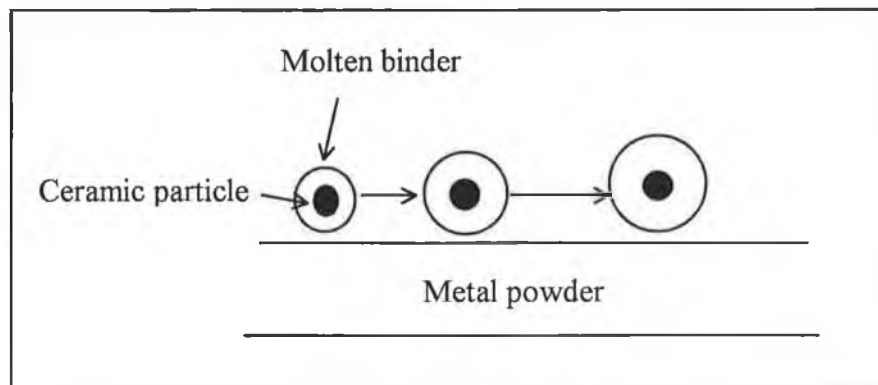


Figure 2.4. Mechanism of Coating Particles in Agglomeration Process [23]

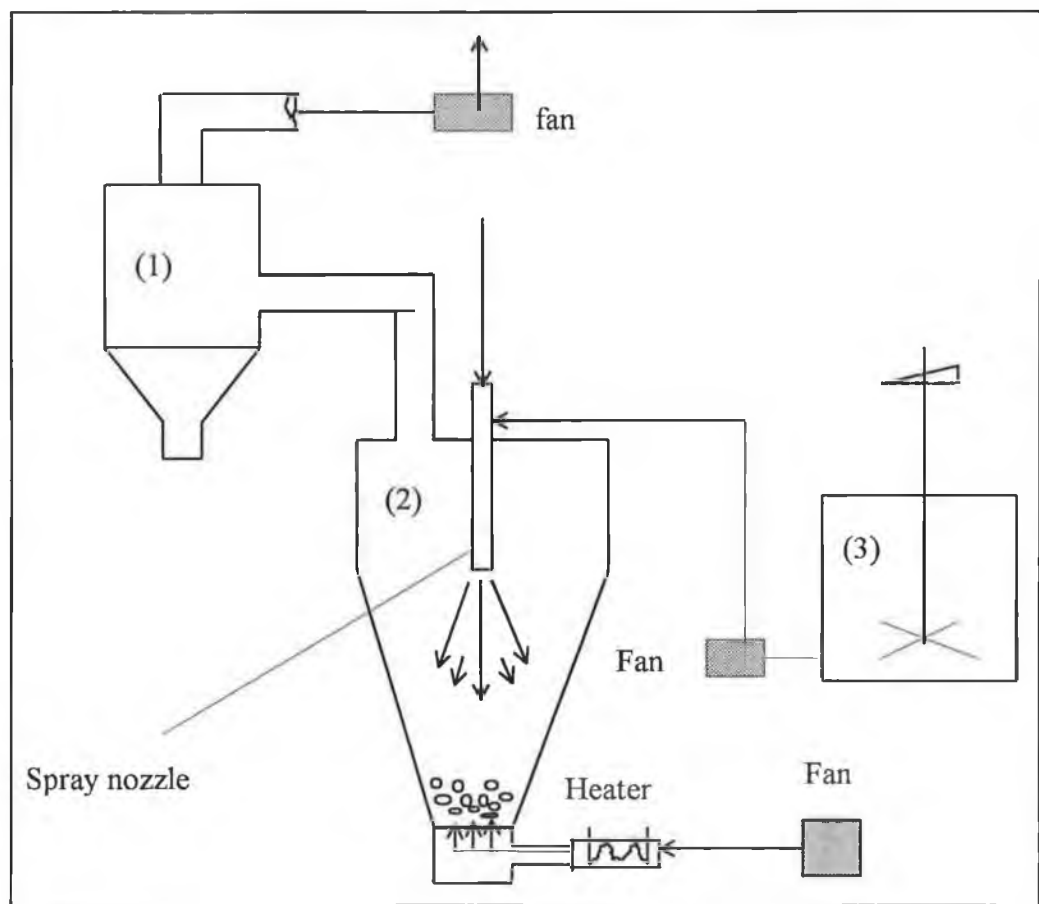
Another example of this method is Al, Ni, CO, Mo, W coated Al_2O_3 or SiC powder, for which the thickness of the coating layer can reach 60 vol% [23][24].

Layering agglomeration results in a relatively homogeneous arrangement of the primary particles and distribution of the binding agent added for granulation. Granules

of up to several millimetre in diameter can be produced by layer methods [6]. Because they are not continuous but separate batch processes, the production rate is low.

2.3.5 Combination Process

One typical approach to powder coating is to combine spray drying and fluidised bed principles. One example is the commercially available: DIAMOND COATER DC-1, used to coat diamond powders. Its basic principle is as follows:



(1) cyclone. (2) chamber. (3) mixer.

Figure 2.5. Basic Principle of DC-1

The metal powders are mixed with appropriate binders (polyethylene glycol (PEG)) in proportion in a mixer and to form a slurry, then the slurry is injected to the spray nozzle; Compressed air with diamond particles and solvent (alcohol) are injected to the spray nozzle at the same time. The slurry metal stream is disintegrated by rapid gas expansion out of the nozzle and impact with diamond particles. Because the slurry has very low surface energy and diamond has a very high surface energy (diamond has several thousand surface energies at room temperature), diamond particles are wetted (coated) by the slurry. When the coated diamond powders reach the lower part of the reaction chamber, where they meet the warm, dry air supplied by a fan and a heater at the bottom, the moisture content evaporates, and the coated diamond particles are fluidised by the hot air, then, the products are conveyed out the chamber. The vapour and air enter into a cyclone, where possibly entrained fine particles are separated from the air. The air and vapour finally are drawn out by an exhaust fan.

It seems that this process is not very effective in encapsulating diamond particles singly, because most encapsulated products may contain multiple diamond particles. The result is clusters of the diamond particles.

Composite powders can also be produced by chemical vapour deposition (CVD). Vapours of chlorine or other compounds react with reducing gas (e.g. H_2) on the hot particle surface. Successful examples include cobalt coated Al_2O_3 , WC and W powder [25-27] and SiO_2 coated TiO_2 powder [28]. and others [29-32]. There are many other kind of methods used to coat particles, such as sol /gel processes [33-37]; plasma process [38-41], others [42][43]. In general, they refer to thin film only, and would not be economical for the present purpose.

2.4 POWDER CHARACTERISTICS

2.4.1 Introduction

Powders are usually defined as particles in the size range 0.1 to 1000 μm . Most of the powders produced are in the size range 10 to 500 μm . A powder has a relatively large specific surface area, or surface area per given weight of material, usually measured in square meters per gram, or m^2/g . The main advantage of a high surface area is that, when a consolidated collection of powders is heated, it will easily sinter and become a single, large solid body. The main disadvantage of the large specific surface area is the tendency to agglomerate, or form uncontrolled lumps and clusters. Usually the higher the surface area, the more the tendency to become agglomerated, other factors being equal. All powder processing starts with a powder, the understanding of the process relies upon good understanding of the powder. The flow characteristics of the powder aggregate are important in powder metallurgy (PM), and these are determined in part by particle size, size range and shape.

2.4.2 Particle Size

Particle size is a determination of the dimensions of a particle. Shape is an important particle property and the particle size is based on it. A spherical particle is easily defined by its diameter. Irregular particles may have a diameter defined terms of a three-dimensional equivalence, such as: 1) the diameter of a sphere having the same surface area, 2) the diameter of a sphere having the same volume or mass, 3) the size of hole (circular or square) through which the particle will just pass. Alternatively the equivalent diameter could be defined in terms of a two dimensional equivalence, such as: 1) the diameter of an inscribed circle, 2) the diameter of a circumscribed circle, 3) the diameter of a circle with the same perimeter. There are also statistical diameters such as Feret's diameter (the distance between the tangents to extremities of the particle, measured in a fixed direction) and Martin's diameter (the length of the line, in

a fixed direction, which divides the particle seen in three-dimensions into two equal areas) [6].

2.4.3 Measurement Techniques

There are many methods of determining the particle size. The four most commonly used methods are sieving, microscopy, sedimentation/elutriation, and the electrical sensing zone technique. The approximate useful range of each method is dry sieving from 100 000 to 50 μm , wet sieving from 100 000 to 35 μm , microscopy (light) from 200 to 10 μm , microscopy (electron) from 1 to 0.01 μm , sedimentation and elutriation from 75 to 2 μm and electrical sensing zone from 800 to 1 μm [5].

Sieving

The most common technique for rapidly analysing particle size is based on sieving. Sieves are manufactured in a range of aperture sizes, often described by mesh number. The most common convention for specifying mesh size relies on the number of wires per inch. The mesh size is determined by the number of wires per unit length. The opening size varies inversely with the mesh size; large mesh sizes imply small opening sizes and vice versa. Sieve analysis begins with a stack of sieves of increasing mesh sizes, with the smallest opening size at the bottom. The powder is loaded onto the top sieve and the sieve stack is vibrated for a period of 20 to 30 minutes. For particle size analysis, a sample size of 200 g is usually sufficient when using 20 cm diameter screens. After vibration, the amount of powder in each size interval is weighed and the interval percent calculated for each size fraction.

Sieving may be carried out either wet or dry. In wet sieving, water is sprayed on to the bulk solid. This has the effect of washing off small particles adhering to larger particles and assisting the passage of fine particles through the sieve. Wet sieving is more efficient than dry sieving at small aperture sizes.

Sieving is the most widely used size analysis technique; however, it is not without its problems [5]. Screens will have from 3% to 7% permissible variation in opening size as a manufacturing tolerance. This creates a soft cut point rather than a sharp division. A common problem is overloading, especially with the finer screen openings. Overloading inhibits powder from reaching the limiting screen size. Hence, the size data is skewed towards the coarser particle sizes. This problem increases with the amount of powder per unit screen area, content of fine particles, and smaller screen openings. Another difficulty results from the differences in screens and techniques. In general, an 8% error can be expected in a screen analysis if neither the screen nor the technique are specified. The difference between screening results can be reduced to approximately 4% if the screen types are the same. Differences as small as 1% can be achieved if full control is exercised over the analysis. Defects in screens can allow oversized particles to pass. Too long a screening duration can result in the particles breaking into finer pieces. Too short a screening duration will give insufficient time for fines to pass all the way through the screen stack. Because of these various recognised difficulties, it is important to use standardised test methodologies.

Microscopy

Microscopy is a widely applied technique for particle sizes measurement. Typically, a scanning electron or optical micrograph will provide sufficient shape detail for comparison to some standards. In the optical microscope method, a sample of material is dispersed on a glass slide and the individual particles are observed and measured.

Particles finer than one micron can be sized by similar means using a transmission electron microscope. The principal disadvantage of sizing by microscopy is that it can be both tedious and time consuming. With the use of sophisticated electronic image analysing and counting techniques the time element can be reduced considerably. Another problem with the microscopy method is that it is difficult to obtain a dispersed sample for image analysis. Frequently, agglomeration or coincidence occurs making it difficult to distinguish the actual particle size and shape. There are

also problems with measurements dependent on particle orientation. Generally it is preferable to rely on parameters which are orientation independent [5].

A particular advantage of viewing the material through a microscope is that, unlike all other sizing techniques, it also gives the opportunity to learn something of the shape and structure of the particles.

Sedimentation

The sedimentation method is based on the rate of settling of particles. It is carried out by dispersing the sample in a liquid. If the bulk solid dissolves in water a suitable non-reactive liquid must be used. It is the 'Stokes' diameter which is determined, which is the diameter of a sphere which has the same density and free-fall velocity as the irregular particles under test [5].

Elutriation

The elutriation method is based on the vertical lift of particles from a porous surface by an upward flow of air at a known velocity. The Stokes diameter is determined. The proportion of the sample which is removed at a given air velocity is measured. The air velocity is then increased and the process is repeated. Elutriation is a slow process as only one size fraction can be measured at a time but it is ideal for materials which are very friable and susceptible to degradation on sieves [44].

Electrical Sensing Zone Technique

The electrical sensing zone method enables both the size and number of particles to be determined. The material sample is mixed in an electrically conductive liquid and the suspension is passed through a small orifice. On either side of the orifice is an electrode and any particle which passes through the orifice momentarily increases the resistance between the electrodes. This generates a voltage pulse, the magnitude of which is a function of the volume particle.

The results are given in terms of the diameter of a sphere of equivalent volume. These pulses are electronically scaled and counted. From the resulting data the size distribution of the sample can be determined, either in gravimetric or particle number terms.

2.4.4 Particle Size Distribution

Two methods of presenting particle size data are commonly used. One is a cumulative plot and the other is a fractional size plot. Both linear and logarithmic scales are used for the particle size axis. Cumulative representation is generally presented on a percentage mass basis and can either be in terms of the percentage greater than a given particle size, or the percentage below the size. The particle size corresponding to the 50 percent value is generally referred to as the median value or mean particle size. Fractional representation may be represented in the form of a histogram. This is particularly useful for comparative purposes as it has the effect of magnifying the results for individual particle size bands [5].

2.4.5 Particle Shape

The shape of a particle is a distributed parameter which can influence packing, flow, and compressibility of a powder. It is characteristic of the powder fabrication route and helps explain many processing characteristics. The established approach is to describe shape in qualitative terms which give an indication of the shape of the particles as observed with the naked eye or through a microscope. In some instances it might be necessary to ascribe a numerical value to particle shape; a sphere is generally taken as the reference shape. Sometimes, qualitative descriptors are used. For example, diamond particles are cubic, however, after air atomisation, coated diamond particles are spherical.

2.4.6 Specific surface

The specific surface of a material is expressed in terms of the total surface area per unit mass of the material. Specific surface is often used as a measure of the 'fineness' of a material. As the surface area increases, the amount of friction in a powder mass increases. Consequently, the friction between particles increases, giving less efficient flow and packing. Gas adsorption methods and permeametry are used for surface analysis [6]. Several different methods for determining a value of specific surface have been developed.

Surface area calculations

For the simple case of spherical particles that are all of the same size, the specific surface area can be calculated from the particle size by the geometric relationship

$$S = \frac{A}{w} = \frac{A}{V\rho} = \frac{4\pi r^2}{\frac{4\pi r^3 \rho}{3}} = \frac{3}{r\rho} = \frac{6}{d\rho}$$

Where, S = specific surface area, [m²/kg]

A = surface area, [m²]

w = mass in gram, [kg]

V = volume, [m³]

ρ = density, [kg/m³]

r = radius, [m]

d = diameter, [m].

2.4.7 Powder Chemistry and Purity

Chemical impurities are a common problem in all industrial materials. Because of the high specific surface of a powder, a large amount of the material is directly exposed to the surroundings during different stages of powder production and processing. Thus impurities can easily be picked up through adsorption and chemisorption from the surrounding medium. The impurity content of powders, therefore, is often greater than that of solid materials. In many cases these impurities remain concentrated at the surface, giving rise to a second specifically powder metallurgy problem: the effect of the impurities on the final material properties. Even very small impurity concentrations can sometimes completely change these properties [5]. Oxygen and carbon are the most important impurity in metal and non-oxide ceramic powders.

Atomic absorption spectrometry is a standard method for the determination of the content of metallic impurities in hard metals, but other common methods of wet chemical analysis can be applied.

Because of the problems resulting from impurity segregation, microanalytical methods are widely used in impurity control, but cannot yet be regarded as standard methods. Especially surface sensitive methods such as Auger electron spectroscopy, electron energy loss spectroscopy are suitable tools [6].

2.5 COMPONENT PRODUCTION BY POWDER TECHNIQUES

There are several stages to the manufacture of powder based engineering components which influence the characteristics and properties of the final product. The stages involved are mixing, compression and sintering. The parameters governing the outcome of each of these processes are discussed below. Figure 2.6 shows the main processing routes employed for the powder metallurgical processing.

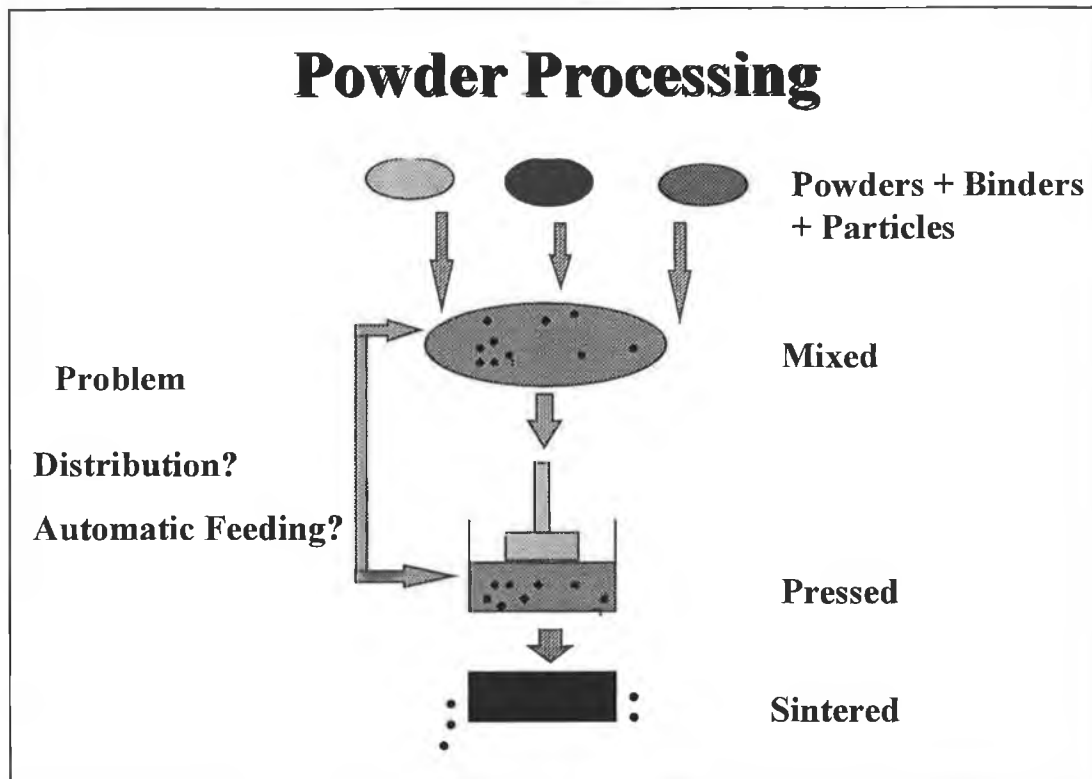


Figure 2.6 . Powder Processing Route.

2.5.1 Mixing

Before mixing, the powder should be classified. Large screens or air classifiers are used to remove selective size fractions. For high quality products, the classification process may involve the removal of contaminants such as inclusions or crucible materials. In certain powders, the main impurities are concentrated in a narrow particle size range. Consequently, removal of that particle size provides an approach to enhanced product purity.

The aim of the powder mixing step is to create a homogenous mixture of the primary powders required for the production of the composite material. Mixing implies intermingling powders of different chemical composition. The optimum dispersion which can be expected from a mixing process is a random distribution of the different types of particles. Mixing is primarily carried out by the mechanical rotation of a

suitably shaped sealed vessel containing a charge of the composite powder. Very varied equipment is available for mixing. Examples include tumble mixers, shear/centrifugal mixers, Spartan granulator, ball mills, attritors etc. The mechanisms involved in the mixing process are complex and can be influenced by many factors [45-47]. Agglomerated powders only attain homogeneity if the agglomerates are broken down during mixing. Wet mixing is often applied for fine ceramic or hard metal powders. For a metal based material with ceramic particle reinforcement, the final homogeneity of the mix depends on matrix powder to reinforcement size ratio, absolute powder size, powder density, charge volume to container volume ratio, speed, direction and duration of mixing and container shape [6]. In the case of mixing fine cobalt powder and PEG, because cobalt is toxic material, skin, eye and respiratory protection must be used during handling [48].

The distribution analysis of powder blends is carried out by statistical means. Samples of powder can be extracted from a batch, chemically or physically analysed, and compared to a theoretical standard distribution. The distribution of reinforcement particles in the final composite material is directly dependent on the effectiveness of the mixing process. Image analysis of micrographs have been used to quantify and compare the homogeneity of fully processed composites [49][50]. However this form of analysis could be misleading since the micrographs are only two dimensional images of a three dimensional material. No standard method of distribution investigation has been devised for these materials.

Interparticle Friction

Under the heading of interparticle friction come two main concerns: powder flow and packing. These concerns are important in automatic die filling during powder compaction, as well as packaging, transportation, and mixing of powders. The main feature of friction is a resistance to flow. In the investigation of the flow of diamond powder by Mikhnovskaya, et al [51], similarity criteria are found for the fluidity process, and the main relations between them are determined. Methods are proposed for improving the flow of the diamond powders. The flowability of diamond powder

about 60 μm is very poor. But after diamond coating, their flowability is improved greatly.

Interparticle friction effect on mixing and blending

There are two sources of interparticle forces, intrinsic and extrinsic. The intrinsic forces are inherent to a material. The extrinsic forces are dependent on properties such as the particle size and shape. The observed friction between particles is a combination of both factors. The extrinsic effects on powder friction can be controlled. Smoothing the surface, using a coarse particle size, oxidising powders generally increases flow abilities. A common problem with metal powders is agglomeration due to the adsorption of humidity. Above approximately 0.5% water added to the powder, flow ceases. Dry powder is preferred to minimise interparticle attractive forces [52].

2.5.2 Compression

Compaction converts a loose powder into a shaped mass with sufficient strength for subsequent handling. Powder compaction is the first step in shaping and imparting specific properties to metal powders. The compaction step has a major influence on the shape, dimensions and density of the part. However, compaction is insufficient in providing acceptable properties; for this reason sintering is more of a concern in determining properties. Compaction is most typically performed using uniaxial pressurisation of the powder in a die. The powder does not respond uniformly to the applied pressure, consequently there are density gradients in the part. The compaction stage of powder metallurgy (PM) production can take many forms. Hot Isostatic Pressing (HIP) and hot extrusion have been found to produce composite material which exhibit excellent material characteristics. However, these processes involve canning, degassing, specialised consolidation equipment and can involve long processing times. These costly production routes are used extensively and a number of

companies have developed mass production facilities based on HIP and hot extrusion [53][54]. The HIP procedure involves the positioning of a degassed container of composite powder in the centre of a cavity within the HIP unit. The heating element within the cavity surrounds the container. Heat and pressure are applied to the container. The pressure is produced by pumping either argon or nitrogen gas into the cavity. The temperature, pressure and duration of the cycle is dependent on the materials being processed. After the hot pressing cycle is complete the pressure is released and the container is removed from the cavity. The composite billet is then extracted from the original container by stripping the container walls. HIP can produce near net shape components which only require minimal machining.

Coating axial pressing and cold isostatic pressing (CIP) are normally used to provide manageable billets of partially consolidated powder before degassing, sintering and/or hot working is applied [55]. The CIP process utilises a fluid medium to apply pressure in all directions and therefore produces a uniformly compacted green billet. Cold axial pressing does not provide the same uniformity of pressure distribution throughout the green compact [56]. The density distribution of the green material will vary with distance from the planes of pressure application. In the case of double acting and floating die uni-axial pressing the axially central volume of the powder compact will exhibit a low density compared to the areas closer to the punch contact areas. The effect can cause inconsistency of the composite material after hot processing [50].

2.5.3 Sintering

Sintering is the process whereby particles bond together at temperatures typically below the melting point of the material by atomic transport events. When metal powder coated ceramic particles are heated, the surface area associated with unsintered metal powders implies an energy excess. At a sufficiently high temperature, mass transport by processes such as lattice diffusion become significant. Reinforcement and metal will bond together after removal of any organic additives that are present [5]. On a microstructure scale, cohesion takes place as necks (welds) grow at the

points of particle contact. The motion of an atom into the neck region is favourable since it reduces the net surface energy by decreasing the total surface area. The events leading to neck growth depend on several possible transport mechanisms, with the main focus on diffusion processes. Diffusion is thermally activated, meaning there is an activation energy necessary for atomic movement.

The sintering properties of powder compounds produced by coating differs from that of elemental powder blends [57] and the main advantage of coated powders are high uniformity [58]. The advantages of coated powders over mixed powders are evident particularly when a small amount of a second component is to be introduced; for example the uniform distribution of sintering additives in high-melting point powders.

When producing diamond grinding tools with certain processes, care needs to be taken to avoid excessively high temperatures. This is because above 900°C, diamond starts changing into graphite. Bronze binders, for instance, require temperatures between 650 and 800°C, ferrous alloys, nickel and cobalt up to 900°C. In the case of hard metal binders, time spent at the necessary high sintering temperatures (above 1000°C) must be kept as short as possible, or the operation needs to be carried out under high pressure. Sintering of diamond powder takes place at about 3000°C under high pressure (to 12 GPa). The aim is to achieve the highest possible degree of direct bonding between the diamond particles, with hardness values of 8000 HVN. This type corresponds to black diamond, which occurs naturally and is less brittle than naturally occurring monocrystals [58].

Stages of Sintering

- *First stage* The particle contacts are transformed to sintered bridges, called 'necks'. Even before sintering, the contacts exist in form of microplanes instead of point contacts, their extension dependent upon compacting pressure. During this stage the powder particles remain discrete. Grain boundaries are often formed between two

adjacent particles in the plane of contact. The centre of the particles approach only slightly.

- *Intermediate stage.* When the size ratio (neck dimension divided by the particle size) exceeds a certain value after strong neck growth, the single particles begin to lose their identity. A coherent network of pores is formed and grain growth occurs, which results in a new microstructure. The grain boundaries usually run from pore to pore. Most of the shrinkage takes place at this stage.
- *Final stage.* Between 90 and 95% of the theoretical density, the relative proportion of the closed pore space increases rapidly. The isolated pores become increasingly spheroidised. In cases where gases cannot diffuse out, they are enclosed in the porosity and further densification becomes impossible as soon as the gas pressure reaches equilibrium with the pressure due to surface tension. When the pores are essentially empty or the entrapped gases are easily diffusible in the solid matrix, further slow densification may occur, especially in fine grained microstructures.

2.6 POWDERS IN FLUIDS (COLLOID SCIENCE)

2.6.1 Introduction

During the particle coating process, ceramic particles are introduced to the coating chamber singly by using solvents, coating metal powders are wetted by binders, and finally, single ceramic particles are wetted by the slurry of metal powders. All of this falls under the category of colloid science. Also, ceramists, for example, use the scientific principles of colloid science to disperse their coarse material, which if not effectively dispersed, can cause trouble in mass production processing [48]. So, it is necessary to fully understand the aggranulation principle through understanding the colloid science. Surface characteristics (chemistry, charge) are central to the stability of

a given fluid/powder mixture. Based on these, powders may remain separate or agglomerate.

An understanding of chemical bonds is central to discussion of colloid science. There are three kind of chemical bonds in the materials: ionic bonds, covalent bonds and co-ordinate bond (Hydrogen bonds). Physical bonds are the van der Waals bonds.

Ionic Bonds

According to quantum mechanics, certain pairings of electrons are far more likely to occur than others, and these specific pairings result in the bonds between the atoms themselves. Example HF, NaCl, CoO. The bond is very strong.

Covalent Bonds

The covalent bond is similar except that its two-electron orbital extends around both atoms. Examples include diamond, H₂. Covalent bonds are particularly common in organic compounds. The bond is strong.

Coordinate Bonds (Hydrogen bonds)

The hydrogen bond is the most important bond to ceramists, it is due to a hydrogen ion forming a rather strong bond between two anions. The hydrogen bond is largely ionic and is formed only with highly electronegative anions. This bond of type has been suggested as one of the main causes of powder agglomeration, which can be a critical factor in ceramic processing. In many cases it is also involved in the adherence of organic additive molecules to powder surfaces, and in the solubility of binders.

Van der Waals bonds

An additional bonding force is the weak electrostatic forces between atoms or molecules known as the van der Waals, or dispersion, forces. For any atom or molecule there is a fluctuating dipole moment which varies with the instantaneous positions of electrons. The field associated with this moment induces a moment in neighbouring atoms, and the interaction of induced and original moments leads to an attractive force. The bonding energies in this case are weak but of major importance for rare gases and between molecules for which other forces are absent. Dry agglomeration is caused mainly by two things, van der Waals forces [59] and H-bond [60].

Polarity

This can be defined as the degree to which the electrons spend more time on one side of a chemical compound than they do on the other side. Pure diamond is a completely nonpolar material because of the strong covalent bond [48].

2.6.2 Adsorption

On the scale of atomic sizes, most powder surfaces are somewhat rough, with micro-size pits that might be classified as open pores. Many examples of adsorption onto a surface also involve some absorption into these pores. So, absorption is one material penetrating into the open pores of another one, while adsorption is one material on the surface of another.

Bonding of the Adsorbate to the Powder.

Because van der Waals forces can act on all materials, they are often one of the driving forces for physical-bonding adsorption (physisorption). Another cause for adsorption is that some powders tend to have electrically charged or at least slightly polarised surfaces, and these will attract any adsorbate ions which have the opposite charge. For example, when cobalt mixes with PEG, cobalt tends to give up electrons (two 4s electrons) and takes on a positive charge. In another example, water may be adsorbed on the surface of ceramic particles by van der Waals forces. Lewis acid/base coordinate bonds can also be the driving force for chemisorption. Hydrogen bonds can link the adsorbate liquid or gas to the powder, if both it and the surface contain atoms that can readily be H-bonded together [48]. On diamond surfaces, stronger covalent chemical bonds can be formed. It was noted that carbon-carbon double bonds involve π electrons which stick out from the molecule in a manner that allows them to have a Lewis base effect [61]. For this reason, unsaturated organic compounds such as oleic acid become adsorbed strongly to Lewis acid compounds such as cobalt. When diamond powders are dispersed in alcohol, the surface of the diamond powder will be covered by thin layer of alcohol (alcohol particle is adsorbed on the diamond surface).

Hydration of the Surface.

Almost all ceramic powders are either partially or entirely covered with adsorbed water vapour [62]. Because water is highly polar, and polarity is a major aspect of the van der Waals force, water can stick to most materials. Even relatively inert graphite adsorbs some water in a partial layer [63], because of an “image force,” in which the electron-rich oxygen of the water molecule repels some electrons in the carbon surface, thus “inducing” a slight positive charge, and it is then attracted to the same charge that it induced. Materials more polar than carbon, such as almost all oxide ceramics, strongly attract water by van der Waals forces, usually more than a monolayer. Another cause of water becoming adsorbed onto most oxides is H-

bonding. Also, many oxide surfaces will chemically react with water in an ionic manner, making a monomolecular layer of a new hydroxide compound at the surface. Because of these almost-universal adsorptions of water in ordinary atmospheres, the surface of nearly all powders should be considered to be somewhat hydrated surfaces.

2.6.3 Surfaces, Interfaces and Grain Boundaries

The surfaces and interfaces between different grains and phase are important in determining many properties and processes. They have a strong influence on many mechanical properties, chemical phenomena, and electrical properties.

Surface Tension and Surface Energy

There is a strong tendency for the distribution of material to be such that the minimum surface energy results. If a small amount of a low-surface-tension component is added, it tends to concentrate in the surface layer so that the surface energy is sharply decreased with but small additions. If a high-surface-tension component is added to one of lower surface energy, it tends to be less concentrated in the surface layer than in the bulk and has only a slight influence on the surface tension. Particularly for high-surface-energy materials, such as diamond and metals, (diamond has several thousand surface energies at room temperature), the effects of surface-active materials are very great [64].

2.6.4 Wetting

For liquid to spread uniformly over solid particles it must wet the solid surface. The wetting of a solid by a liquid can be explained in terms of surface energies. The equilibrium shape of liquid placed on a solid surface conforms to the minimum total interfacial energy for all the phase boundaries present. If the solid-liquid interfacial

energy (γ_{sl}) is high, the liquid tends to form a ball to give a small solid-liquid interfacial energy since the system desires the lowest energy. This is non-wetting behaviour. If the solid-vapour interface energy (γ_{sv}) is high, the liquid tends to spread out to eliminate the solid-vapour interface. This is wetting behaviour. Between these two extremes the situation is intermediate, as is illustrated in Fig. 2.7. The angle between the solid surface and the tangent to the liquid surface at the contact point may vary from 0 to 180° . This angle specifies the conditions for minimum energy according to the relation $\gamma_{sl} + \gamma_{lv} \cos\theta = \gamma_{sv}$. $\theta = 90^\circ$ is defined as the boundary between non-wetting and wetting behaviour. If $\theta > 90^\circ$ the system shows non-wetting behaviour and if $\theta < 90^\circ$ wetting occurs. A liquid will wet a solid when $\gamma_{sv} \geq \gamma_{sl} + \gamma_{lv} \cos\theta$.

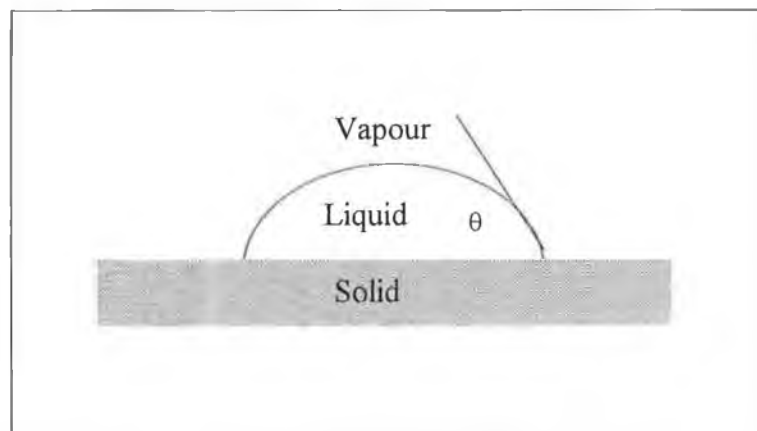


Figure 2.7. The Morphology with Intermediate Energies for Liquid wetting the solid Surface

Wetting in ceramic systems is more complex than this simple analysis since the solid is often partly dissolved in the liquid, changing its composition during the process [65]. Transient wetting is known where the liquid initially spreads and assists densification but its composition is altered so much that it later dewets and, for example, moves to the triple points or is incorporated into the grains of the microstructure. This transient liquid sintering can be achieved by control of the liquid composition to give a wetting intermediate liquid but non-wetting final liquid, using starting powders that go through a series of chemical reactions prior to the formation

of the final stable phase with one of the intermediate compounds being a wetting liquid and using starting powders that form a solid solution at equilibrium but pass through a liquid stage before equilibrium is reached [65].

2.6.5 Charged Particles in Suspension

Sources of Charge

A powder in acid solution, if the surface is neutral or acidic (such as Titania, Silica), will become positively charged ($-\text{MOH} \rightarrow -\text{MOH}_2^+$). If the surface is basic (such as Calcia), it will also become positively charged ($-\text{MOH} \rightarrow -\text{M}^+ + \text{H}_2\text{O}$). Powder in base solution, if the surface is neutral or basic (such as Titania, Calcia), will become negatively charged ($-\text{MOH} \rightarrow -\text{MOH}(\text{OH})^-$). If the surface is acidic (such as Silica), it will also become negatively charged ($-\text{MOH} \rightarrow -\text{MO}^- + \text{H}_2\text{O}$). So, powder surface will have a positive charge when it is in acid suspension. Powder surface will have negative charge when it is in base suspension. Almost all ceramic powders (including graphite) when placed in an acidic aqueous solution have some tendency to become positively charged, and in bases they are negative [66].

The double layer

When phases come into contact, some redistribution of positive and negative charge invariably occurs, leading to the formation of an electric double layer, as shown in Figure 2.8. Double layers can be formed by one or more of the following mechanisms, selective adsorption of ions onto the solid particle from the liquid phase, dissociation of ions from the solid phase into the liquid, adsorption or orientation of

dipolar molecules at the particle surface and electron transfer between solid and liquid phased due to differences in work function.

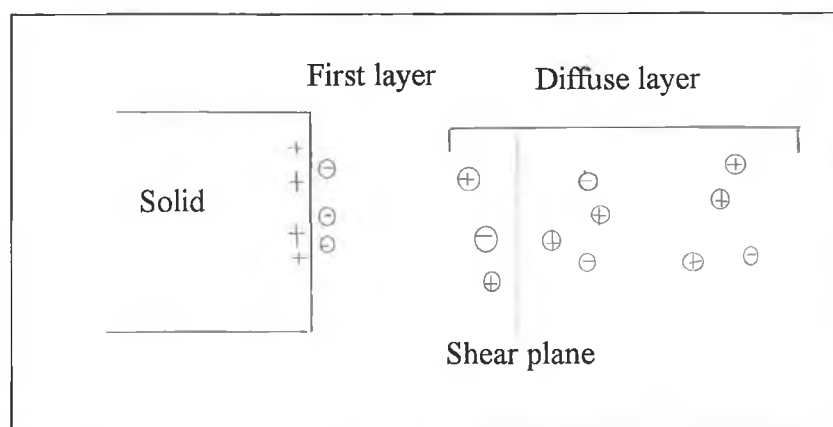


Figure 2.8. The Forming of Double Layer When Solid in Liquid.

The outermost is the diffuse layer. It contains positive and negative charge, which tend to further neutralise the charges. This will become somewhat decreased by the formation of a double layer.

Two particles having the same charge will repel each other electrostatically, with a repulsion force proportional to the inverse of the distance squared. On the other hand, there will be a van der Waals force of attraction, which is proportional to the inverse of distance cubed. As the particles approach to nanometer distances, the van der Waals forces take over, and there will be a net attraction until contact is made.

The potential energy decreases at very small distances. If the particles are farther away, the van der Waals attraction decreases sharply because of the large exponent of inverse distance, and the electrostatic repulsion takes over. The net effects of attraction and repulsion at various distances were studied by B, Derjaguin, et al

[66]. The point of particle to particle contact is called the primary minimum in energy. As the particles separate, another point of low potential energy is reached in some cases, this is called the secondary minimum. A pair of particles at this distance will tend to stay there. Ceramic particles which have been given an electrostatic charge by any of the above mentioned means are often found to behave in this manner. That is, there is a stable distance of separation. This distance varies, depending on both the electric charge on the particles and also on the concentration of other ions in the diffuse layer. It should be obvious that this distance can vary considerably, depending on the pH and the nature of the adsorbates. One of the largest effects on the dimensions of the double layer is the concentration of charges from other ions which are dissolved in the solution. Some additions [48] of ions such as chloride in large amounts, are added in order to not fully collapse the double layer, but broaden it somewhat. This tends to keep the ceramic powder particles slightly farther away from each other, decreasing the tendency toward agglomeration.

2.6.6 Stabilised Suspensions

In most nonaqueous solvent systems, there is little or no ionisation possible, and ionic repulsion is usually a minor factor. Instead, another type of stabilisation is observed. This involves the mechanical prevention of two particles approaching each other closely enough to fall into agglomerate. If a uniform coating of a very non-polar organic compound is adsorbed around each of two powder particles, and the particles then move toward each other until the coatings make contact, the particles will not stick but will bounce away from each other. The reason is : 1) the non-polar materials have much less van der Waals attraction than the highly polar oxide powder surfaces do. 2) the coatings are weak and will break easily even if they do adhere. This phenomenon is called steric hindrance. In order to be adsorbed onto the powder, these coated molecules often have one polar end for van der Waals or other bonding to the particle, and one non-polar end to prevent sticking to the other particle's coating [48]

2.6.7 Binder

The binder is a temporary vehicle for translating the powders into the desired shape and provide green strength until the beginning of sintering. The binders are needed if the interparticle adhesion in the formed compact is insufficient. Interparticle adhesion depends on the size and quality of the contact planes between neighbour particles, and is usually strong in parts produced by compacting from plastically deformable metal powders. The adhesion decreases with decreasing plastic deformation of the particles. Binders are therefore needed for hard powders and forming operations with small interactions between particles [67]. Desirable characteristics of binders are: 1) easy burn out. 2) strong green body. 3) solubility in fluidizing liquid. 4) low cost.

Adhesion

There are three kind of adhesion methods between powder and binder: van der Waals, chemical and mechanical. Van der Waals needs clean surfaces and the adhesion is relatively weak. It can be strong enough if the surface has just been scraped clean by the powerful wiping action of sliding ball mill media. Chemical bond is strong even if part of the area is not clean or flat. Mechanical bond is for the rough surface interlocking.

2.6.8 Solvents

Solvent is used to temporarily make the system more fluid. Binder, which is usually an organic chemical in solid form, is ordinarily dissolved in this solvent.

Water is the most commonly used solvent in ceramic technology. The main advantages of water are that it is cheap and safe. Water also has disadvantages, such as a tendency to chemically react with certain ceramic powders like barium titanate,

degrading their properties. Water sometimes causes the slip to be too viscous. In addition, water does not evaporate as quickly as some organic solvents, and therefore drying is much slower and requires more heating. For these reasons, organic solvents are often used instead of water [48].

2.6.9 Dispersants

These materials, sometimes called surfactants, are put into the particles and liquid composition for two main reasons: keeping the powder-solvent slip from becoming too viscous and preventing agglomeration (lumpiness). The addition of surface active agents in conventional wet granulation is not uncommon [68]. It has been found that the wettability and adhesion probability will greatly improved if some Texapon K12-96 surfactant added to 16 wt.% lactose solution [69]. It was found that Cetyltrimethylammonium bromide (CTAB) was a powerful dispersants for disaggregation diamond powders [70].

2.7 POWDERS IN AIR FLOW

The most commonly used conveying systems for powders are pneumatic conveying systems. For coating ceramic parcels and gaining a homogenous distribution in component production, it is important to effectively disperse powder particles during the conveyance before they are coated. It is therefore necessary to fully understand the behaviour of the fine particles dispersed in air flow.

2.7.1 Phase Density ϕ

In general, there are two modes of flow of material in a pipeline: Dilute Phase and Dense Phase.

The phase density of a conveyed material is the ratio of the mass flow rate of the material being conveyed, m_p , to the mass flow rate of the air used for conveying, m_a , i.e. $\phi = m_p / m_a$.

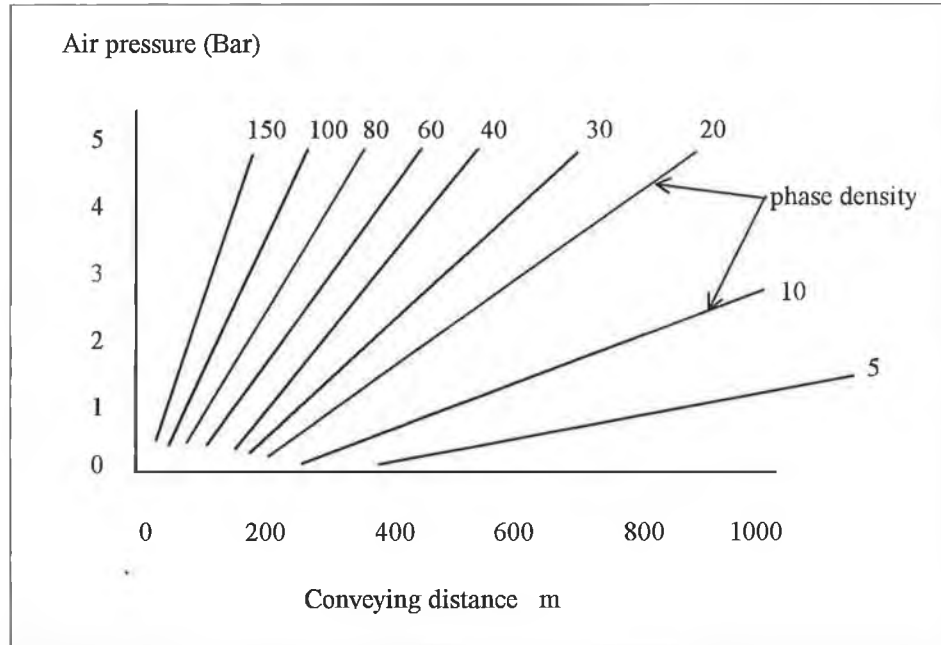


Figure 2.9. Influence of Air Supply Pressure and Conveying Distance on Phase Density for High Pressure Systems [44].

Since air is a compressible fluid its density changes with pressure, as shown in the figure above. If very long conveying distances are required, the phase density will be relatively low, even with a high pressure system [44].

The value of phase density is often used to define the mode of flow of material in the pipeline. Classification can be based on two broad categories.

In dilute phase (suspension flow), material is conveyed essentially in suspension in air through the pipeline. In terms of phase densities the appropriate range is below a value of ten. In the dilute phase range the mean spacing of the particles is fairly wide [44].

At a phase density of ten a minimum conveying air velocity of about 14 m/s will normally be required. It is obviously that the higher the velocity of the air the smaller the phase densities, and the bigger the distance between two particles. Because velocity is the most important variable affecting degradation of the particles, the air velocity cannot be too large. As particle velocity increases, the degree of fragmentation of the particles also increases. So the velocity of conveying air is very important. The range of velocity, therefore, is relatively narrow, particularly in dilute phase systems. Varying from a minimum of about 15 m/s to a maximum of around 30 m/s. It requires a minimum air velocity of about 13 to 16 m/s for conveying in dilute phase.

CHAPTER 3 EXPERIMENTAL WORK

3.1 INTRODUCTION

In this study, the experimental work was concentrated on the investigation of the principle of coating of particles by using a ‘snow ball’ process, and supporting mathematical models have been developed to describe the process.

The study is limited to the coating of large, nominally spherical food particles (dried Peas) with more fine, food powders (Flour and Almond Powder). This is a simplified process to demonstrate the validity of the snow ball process. Ceramic particles were replaced by dried peas (they have similar rolling behaviours), metal powders were replaced by food powders (all of them have similar powder properties), PEG (polyethylene glycol) binder were replaced by water, olive oil etc. (they have lower surface energy and when they come in contact with solid, the solid surface will be wetted by them just like PEG) [5][48][64]. In the experimental work, although the materials used are different from the real material envisaged for engineering application, the basic coating principle is the same, and the materials just influence the coating particles properties (coating thickness etc.). To facilitate these demonstration tests, a simple experimental rig was designed and constructed. In addition to carrying out this demonstration experimental programme, three different conceptual designs were also produced which may utilise the snow ball principle of coating engineering particles. As part of further investigation, any new researcher may decide to construct one of these systems for coating ceramic particles.

3.2 EQUIPMENT AND SYSTEM LAYOUT FOR COATING

The equipment used for the experimental work consists of a plate, tilted at an angle (variable), onto which a powder bed is placed and a row of wet particles are released from the upper edge of the plate. The surface of the plate is roughened such that the powder bed will not slip downward easily. The particles dry while rolling down the plate, and are then collected, sprayed with mist and released again from the top edge of the sloped plate. Figure 3.1 shows a photograph of the apparatus.

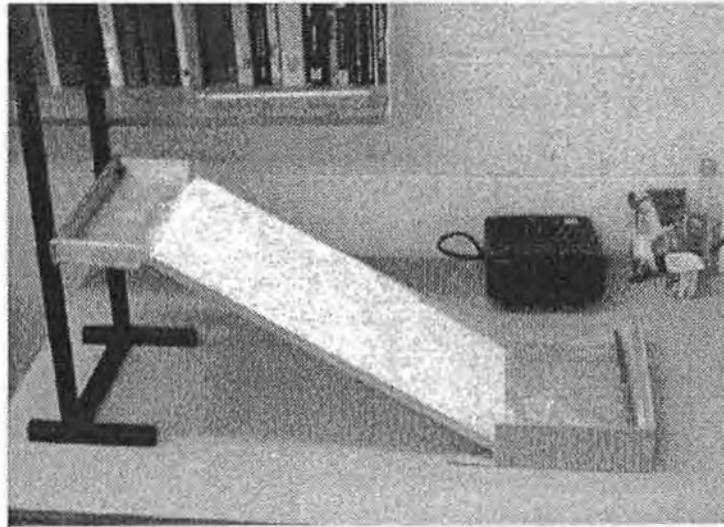


Figure 3.1. The Equipment for the Particle Coating Process

The detailed dimensions are shown in the plan and top view of the apparatus given in Figure 3.2.

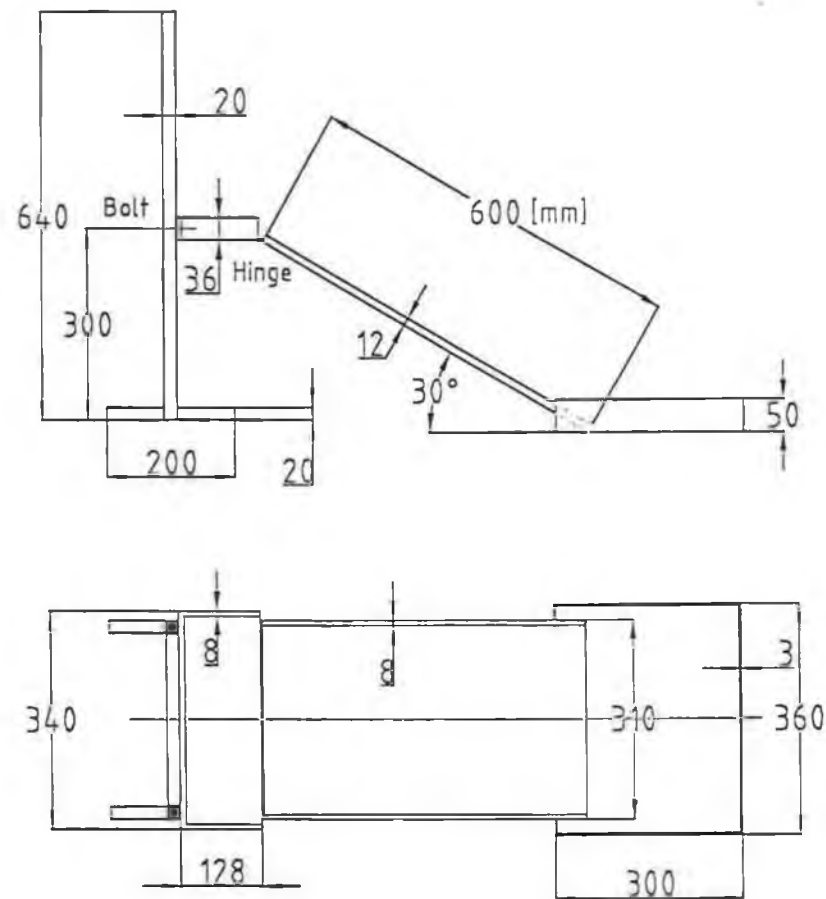


Figure 3.2. Equipment for Powder Coating

3.3 MATERIALS USED IN THE EXPERIMENT WORK

During the experimental work, Marrowfat Peas with average mass of 0.39g and Moong Beans with average mass of 0.07g were selected as particles for coating (they have similar rolling behaviours as ceramic particles). Water, Olive Oil and a mixed solution of pre-prepared 16%wt Lactose and 0.085g/l Texapon were used as binder (all of them have the properties of binder and have lower surface energy). Flour, Ground Almond and Icing Sugar were used as coating powder (all of them are very fine and have similar powder properties).

3.4 RANGE OF EXPERIMENTS

The experimental work included the investigation of some system factors, i.e. drying temperature, drying time and the tilt of the powder bed. Additionally the suitability of various media were investigated: particles, powder, and binder type.

A series of tests were carried out, each of which concentrated on a number of individual processing parameters. Table 3.1 lists each test by indicating the materials used, and the parameters varied within each test.

Table 3.1. Test Matrix for Particle Coating Process

No.	Particles	Powder	Binder	Drying Conditions		Powder Bed Height (mm)	Max. Cycles
				Time (min)	Temperature (°C)		
test 1	Marrowfat Peas	Flour	Water	1	89	a: 160. b: 300	1
test 2	Marrowfat Peas	a: Flour b: Ground Almond c: Icing Sugar	Water	10	21	300	5
test 3	a: Same as Test 2a b: Moong Beans	Flour	Water	10	21	300	1
test 4	a: Same as Test 2b b: Moong Beans	Ground Almond	Water	10	21	300	1
test 5	a: Same as Test 2c b: Moong Beans	Icing Sugar	Water	10	21	300	1
test 6	Marrowfat Peas	Flour	a: Water b: Olive Oil c: Lactose+Salt	10	21	300	1
test 7	Marrowfat Peas	Ground Almond	a: Water b: Olive Oil c: Lactose+Salt	10	21	300	1
test 8	Marrowfat Peas	Icing Sugar	a: Water b: Olive Oil c: Lactose+Salt	10	21	300	1

In the table, Lactose + Salt is: 16 wt % Lactose + 0.085 g/l Texapon

3.5 GENERAL PROCEDURE FOR COATING

The general procedure for coating particles is described in this section. Stages of the procedure include: Adjusting the height of the tilt plate, putting the coating powder on the coating bed, dividing the powder bed by using paper sheets, picking up the particles and measuring the weight and diameter of the particles, spraying binder thoroughly onto the particles, rolling the particles, drying the coated particles, measuring the diameter and weighing of the coated particles. The step by step procedure is as follows:

- 1 Adjust the height of the equipment. A spanner and a meter ruler were used to adjust the height of one end of the powder bed. The height of the bed could be adjusted from zero to 300 mm.
- 2 The coating powder was put on the surface of the coating bed. Powder was scattered on the plate and a comb was used to make the powder bed surface smooth. The powder bed length was 400 mm, width was 310 mm, depth was 10 mm.
- 3 The powder bed surface was divided using six paper sheets in this step. A paper sheet was cut using a knife into strips of 287 mm by 25 mm. Each strip was slotted into the powder on the powder bed at the desired position and was held in a standing position by the powder. The locations of paper strips were at distances 25 mm, 50 mm, 75 mm, 100 mm, 150 mm and 200 mm from the top edge of the plate, as shown in the photograph in figure 3.3.

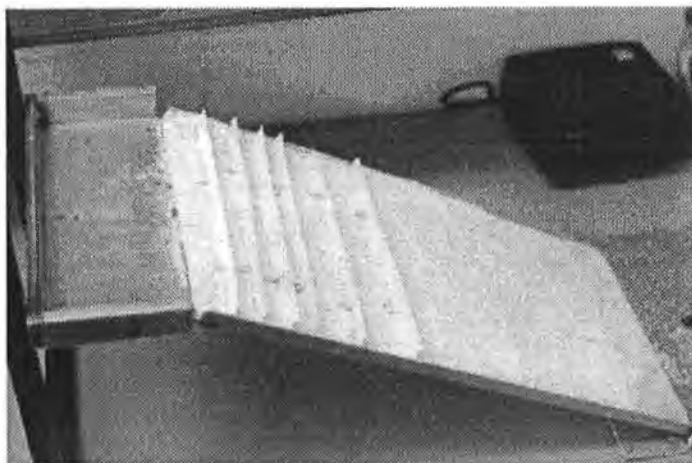


Figure 3.3. The Powder Bed Surface Is Divided by Paper Strips.

4 Particles to be coated were sorted according to their weight using a scale and then stored in plastic trays according to different mass (shown in the photograph in figure 3.4). A tweezer was used to pick up the particles.

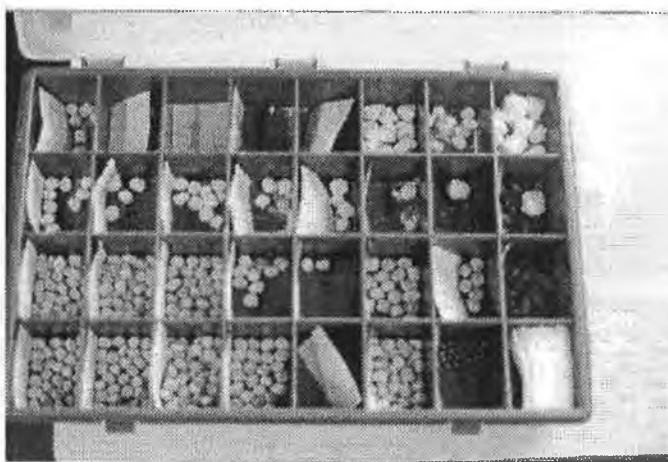


Figure 3.4. Particles Are Sorted and Stored According to Mass

The method of weighing the particles is as follows:

A The scale was put on the surface of a flat, horizontal table, far from vibration influence.

B The balance of the scale was adjusted. It had a level control and adjustable levelling feet to compensate for slight irregularities in the weighing bench surface. A spirit level was provided to indicate when the balance was exactly horizontal.

C The power supply was connected after checking that the imprinted voltage value on the AC adapter matched the local supply voltage.

D To obtain accurate weighing results, the balance must be matched to the acceleration due to gravity at its location.

- Any load was removed from weighing pan and the ON key pressed. When zero was displayed, the balance was ready for operation.
- The particles were placed on the weighing pan. When the stability detector “0” disappeared, read the result.
- The OFF key was pressed until “OFF” appeared in the display, and the key was released.

The photograph of the scale is shown in Figure 3.5.

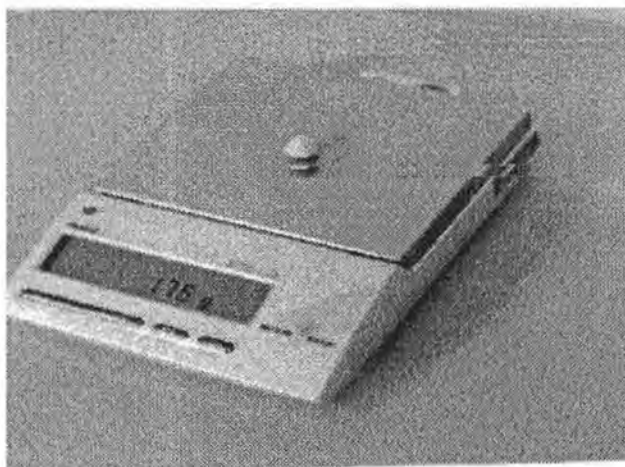


Figure 3.5. The Scale for Weighing the Particles

5 The initial diameter of the particles were measured using a micrometer. Only the almost spherical-shaped particles were accepted. An example of a set of data is given in Appendix B. Because the particles were not uniform spheres, the diameter of the particles was taken to be the average diameter in three directions. For example: d_{01a} , d_{01b} , d_{01c} (see Figure 3.6). The average diameter d_{01} is:

$$d_{01} = \frac{d_{01a} + d_{01b} + d_{01c}}{3}$$

During the measurement, in order to avoid crushing the coating surface, the particle was gently put on a piece of plain paper, and then the micrometer was placed close to the particle, and the knob carefully turned until the two ends touched the particle surface.

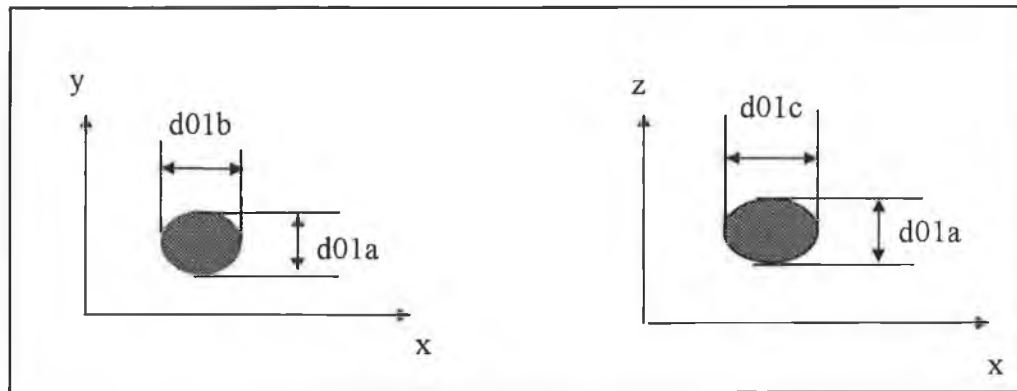


Figure 3.6. Particle Diameter Measurements

The photograph of the micrometer is shown in Figure 3.7.



Figure 3.7. The Micrometer for Measuring Diameter

Cycle 1 Coating

6 A tweezers was used to pick up the peas and a spraying bottle with a nozzle which generates a fine mist was used to spray the binder onto the peas. Particles were placed in a line at the top of the powder bed.

7 Particles were released from the top of the plate to roll on the powder bed surface. Depending on the position of the paper stops, they rolled a given distance.

8 The coated particles were picked up carefully and put onto a clean paper and left to dry for a specific time. An electric fan may be used during this step.

9 The final diameter and weight of the particles were measured following the same procedure as given above.

10 A thermometer was used to measure the temperature of the coating environment. The range of the thermometer was from zero to 100 °C. The coating environment temperature could be changed by an electric fan.

11 The increase in diameters were recorded. During one coating cycle, usually 12 particles (with nominally the same weight) were used for coating, although in some cases two particles were used.

A For the case of 12 particles, the coating system indicated in figure 5.8 was used in calculations. “d = diameter” followed by a number indicating the distance rolled (1 = 25 mm, 2 = 50 mm ...) followed by a number indicating the number of the particles which rolled a given distance (1.2). The Changed diameter were described by d1, d2, d3....d10, d11i, d12i. For example: $d1 = d11 - d01$, $d2 = d12 - d02$, $d11i = d61 - d011$, $d12i = d62 - d012$. Average changed diameter $((d1 + d2) / 2, (d3 + d4) / 2....(d11i + d12i) / 2)$ was used for plotting the curve in chapter 4.

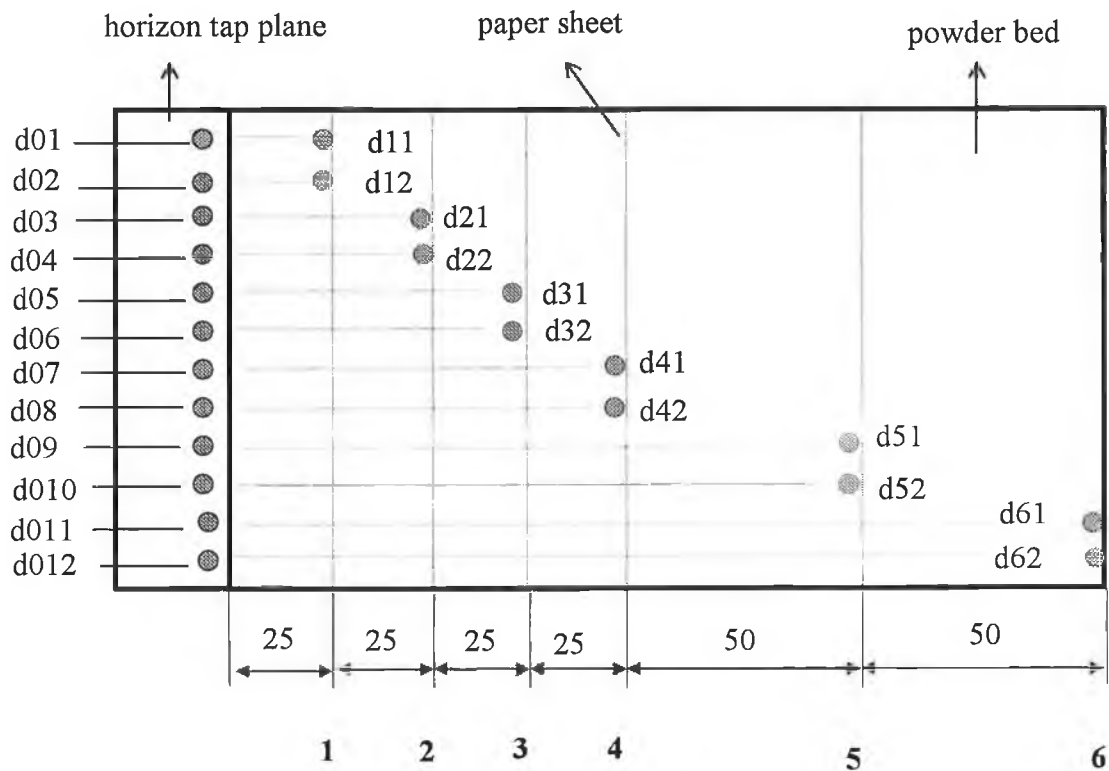


Figure 3.8. Coating Process for 12 Particles

B In the case where only two particles were used, they were coated over a given distance, cleaned and subsequently coating by rolling over a longer distance desired, and the process was repeated. The same labelling convention was used to record the diameter and the weight of the particle coated over a given distance.

Cycle 2 Coating

12 Particles for a second coating cycle had been first coated over a full 200 mm distance of powder bed, and then dried. The particles with the same weight after coating one cycle were picked for use in the second cycle and the procedure described above was repeated. The particles which had been coated on the first two complete cycles (two times 200 mm) and with the same weight are picked for use in the third cycle.

CHAPTER 4 RESULTS AND DISCUSSION

4.1 INTRODUCTION

The results of the powder coating process from the present work are described in this chapter. It includes: the effect of the release height (test 1), the effect of the type of coating powder (test 2), the effect of the particle size (test 3 - test 5) and the effect of the binder (test 6 - test 8).

4.2 EFFECT OF RELEASE HEIGHT

Test 1

Particles:	Two Marrowfat Peas (the average initial mass is: 0.39 g. the average initial diameter is: 8.13 mm)
Coating powder:	Flour
Binder:	Water
Drying time:	1 min
Drying temperature:	89 °C
Release height:	a: 160 mm b: 300 mm

a: Diameter Increase vs. Rolling Distance

Data fit for release height of 160 mm is: diameter increase = $805 * (1 - e^{-0.05 * x})$, and for 300 mm is: $d = 688 * (1 - e^{-0.03 * x})$, where e is the base of natural logarithm and x is the rolling distance, as shown in figure 4.1.

The curve fit was achieved using the following approach. The first step was to pick the best type of model (shape of curve). For example, the model could be linear, exponential or parabolic. Having compared data to each of these, and other type curves, it was found that the exponential model of $Y = A (1 - e^{-K * x})$ was the best. It reflected best the process of coating. The second step was to select the curve parameters to fit the data. It can be seen that A equals the maximum increase in

diameter or mass. This was taken from the data, and then K varied until the model curve fit the data best.

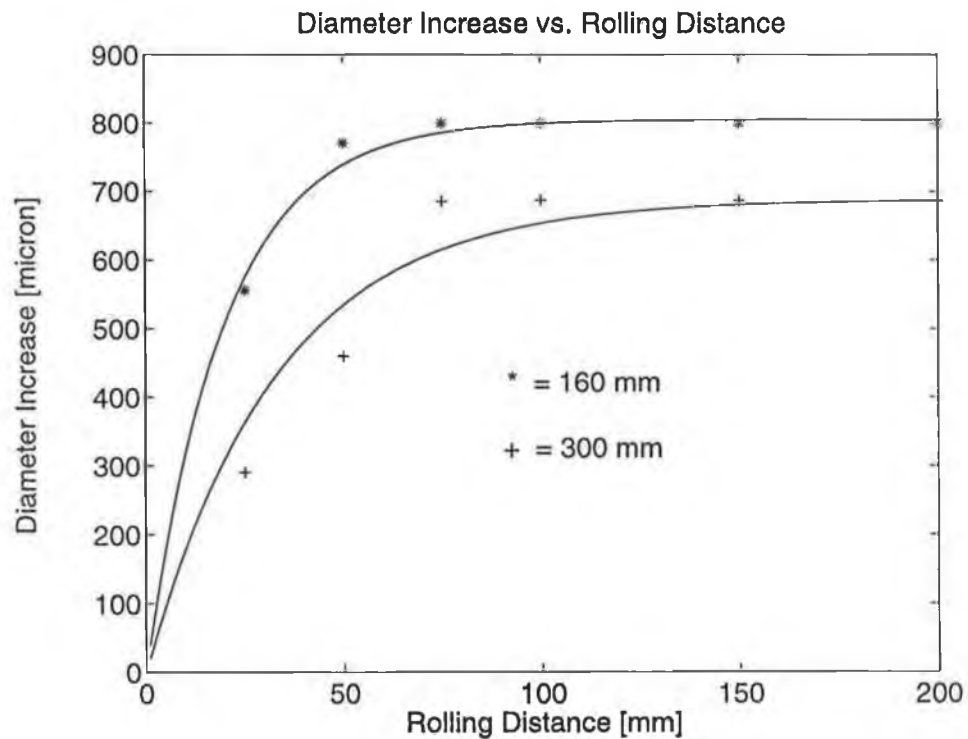


Figure 4.1. Test 1: Effect of the release height.

b: Mass Increase vs. Rolling Distance

Data fit for release height of 160 mm is: mass increase = $10 * (1 - e^{-0.05 * x})$, and for 300 mm is: $m = 7 * (1 - e^{-0.04 * x})$, where x = rolling distance, as shown in figure 4.2.

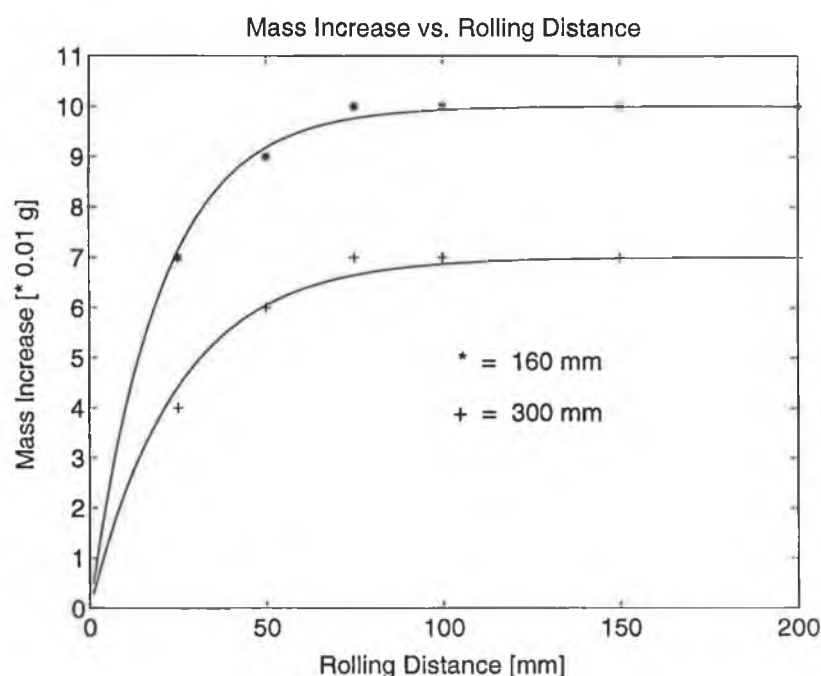


Figure 4.2. Test 1: Effect of the release height.

It can be seen from figures 4.1 and 4.2 that most of the coating occurs over the initial short distance, and when the release height is 160 mm, the diameter increase is approximately 17 % greater and the mass increase is approximately 42.9 % greater than those when the release height is 300 mm. This is because the adhesion probability (h) is greatly influenced by the relative velocity (v) between the particle and coating powder ($h \propto 1/v$). The height is the best when the particles can just start to roll down the slope and hence the velocity is lowest. Preliminary experiments show that the best height is different for different particles. Different types of particles have different surface properties and the amount of coating can be large or small. The more coating on the surface, the bigger the resistance is to the rolling movement. The height of 300 mm was selected afterwards so that all kinds of particles could be rolled down the slope.

4.3 MULTI - COATING

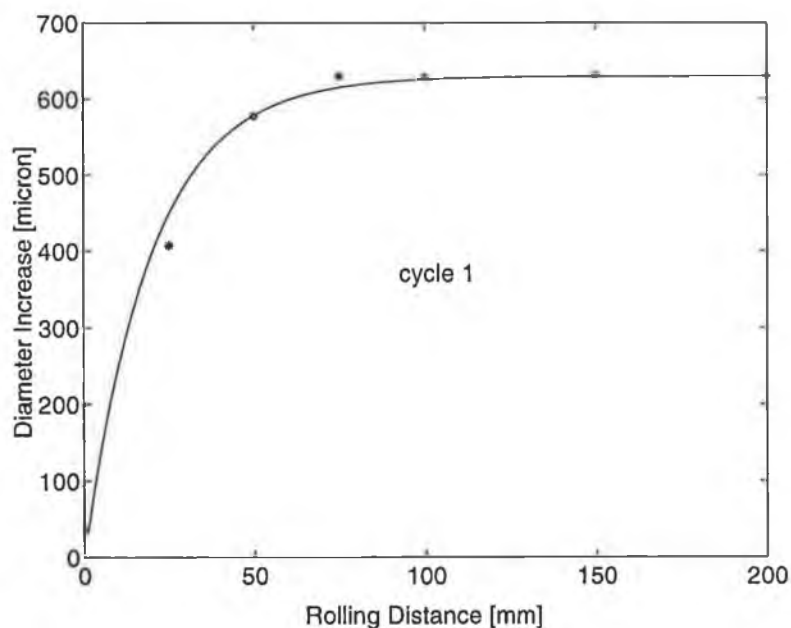
In these tests, the coated particles were returned through the coating process several times, in order to build up a thicker coating.

4.3.1 Flour

Test 2a

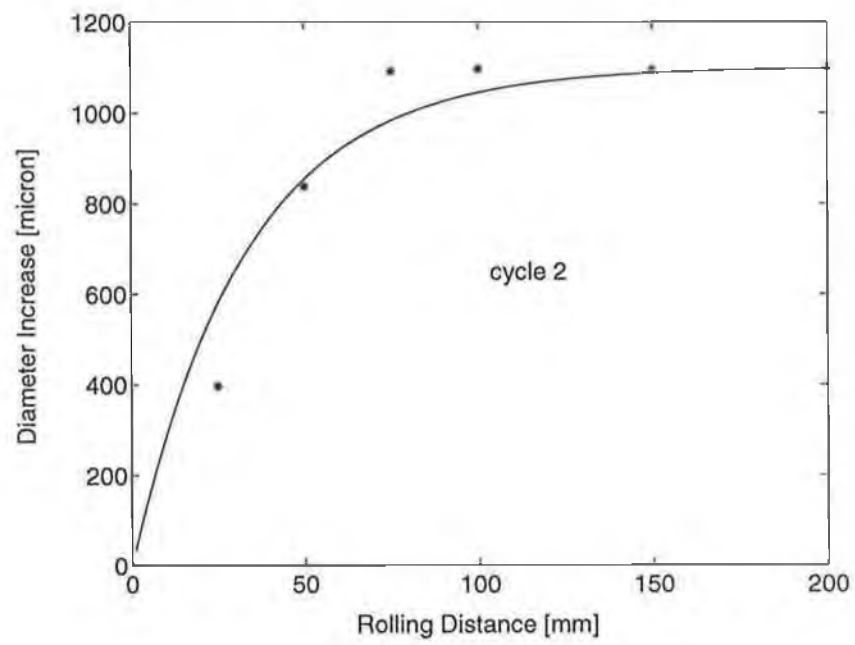
Particle:	Two Marrowfat Peas (the average initial mass is: 0.39 g. the average initial diameter is: 8.13 mm)
Coating Powder:	Flour
Binder:	Water
Drying time:	10 min
Drying temperature:	21 °C
Release height:	300 mm

The drying time and temperature are arbitrarily chosen for the purpose of convenience of measurement. It is found that after 10 minutes at 21 °C, the particles are dry enough for handling. In the curve, “*” stands for the average diameter increase from the experiment, each based on micrometer measurement. Measurement error is less than 0.01 mm. Figure 4.3 shows increase in diameter vs. rolling distance for 5 coating cycles.

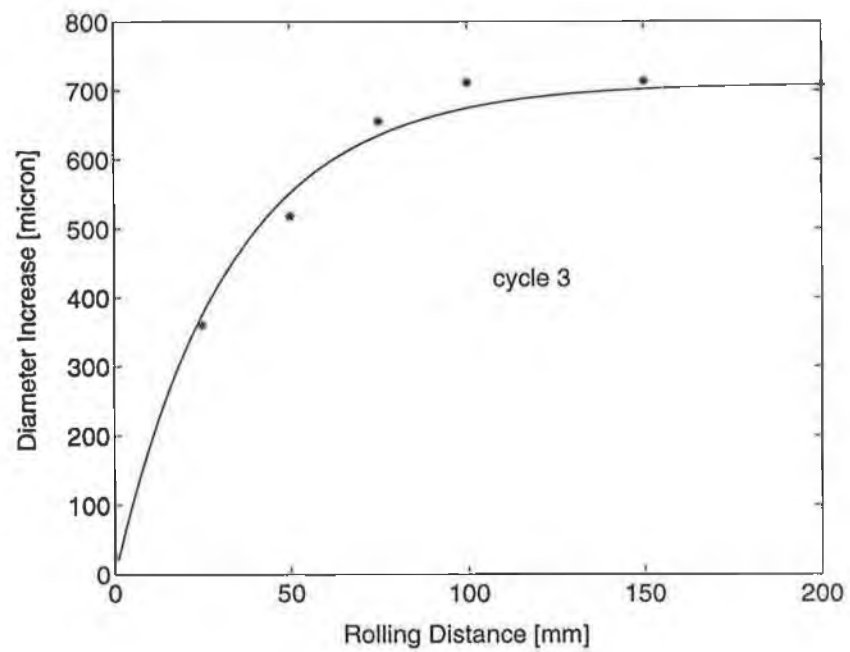


$$(a): d = 630 * (1 - e^{-0.05 * x})$$

Figure 4.3 Increase in diameter vs. rolling distance

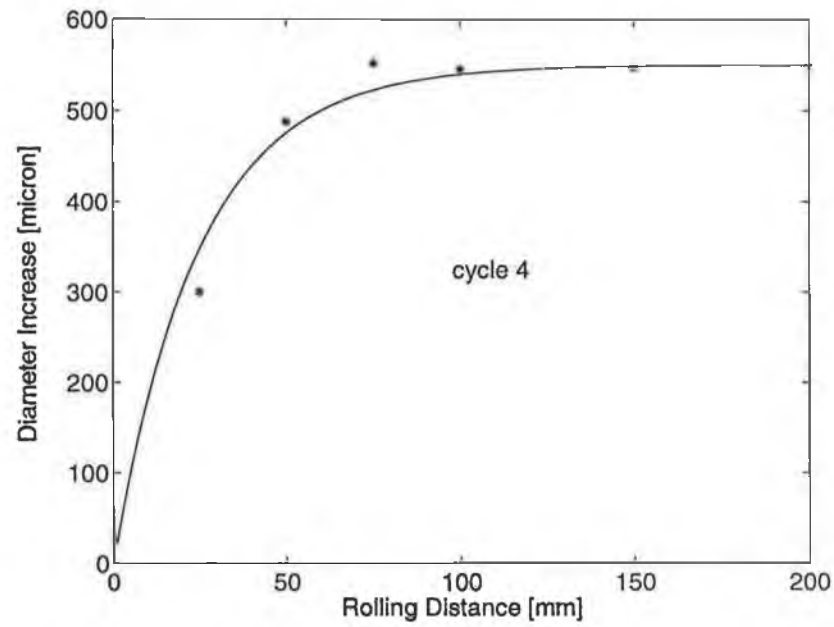


(b): $d = 1100 * (1 - e^{-0.003 * X})$

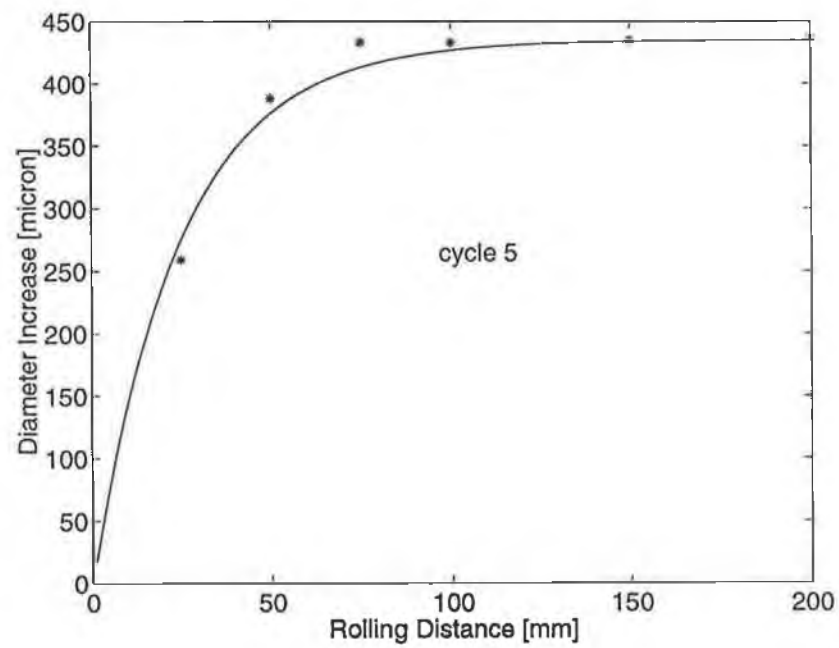


(c): $d = 710 * (1 - e^{-0.003 * X})$

Figure 4.3 Increase in diameter vs. rolling distance



$$(d): d = 550 * (1 - e^{-0.04 * x})$$



$$(e): d = 435 * (1 - e^{-0.04 * x})$$

Figure 4.3 Increase in diameter vs. rolling distance

The cumulative increase in diameter has been plotted against the cumulative rolling distance as shown in figure 4.4.

In figure 4.4, the distances 0 - 200 mm represents cycle 1, 200 - 400 mm represents cycle 2, 400 - 600 mm represents cycle 3, 600 - 800 mm represents cycle 4, and 800 - 1000 mm represents cycle 5. It is clear from this distance that the same level of total increase in diameter may be obtained within a rolling distance of half that shown in figure 4.4, provided in process wetting and drying can be achieved.

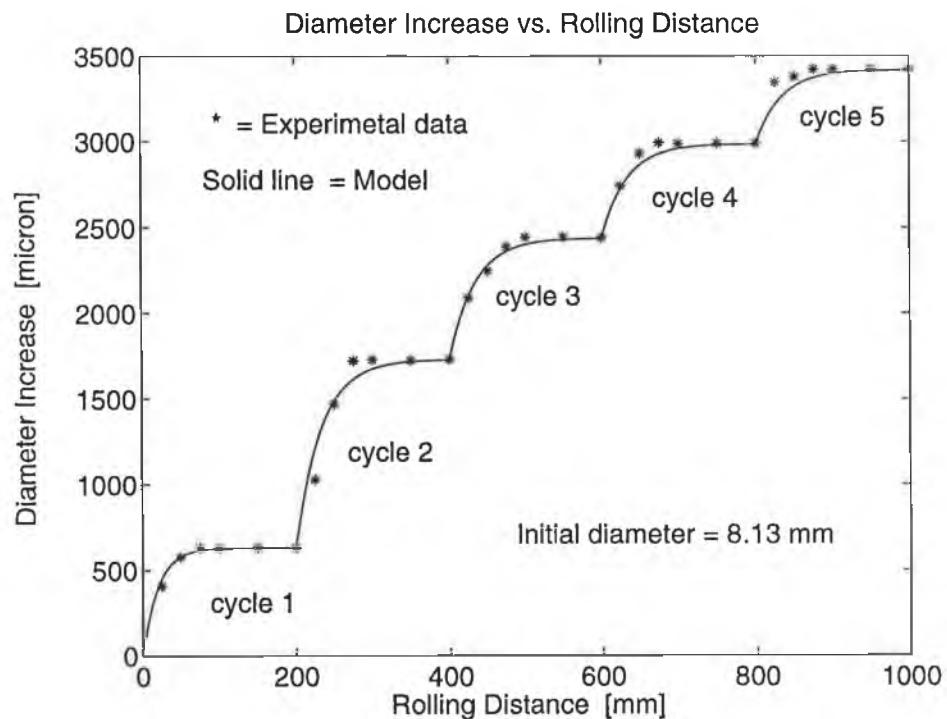
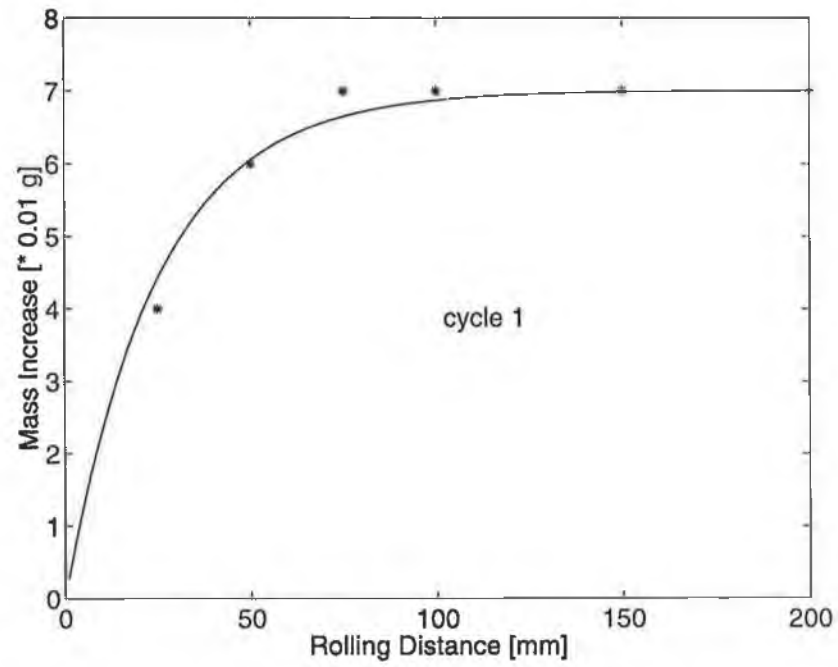
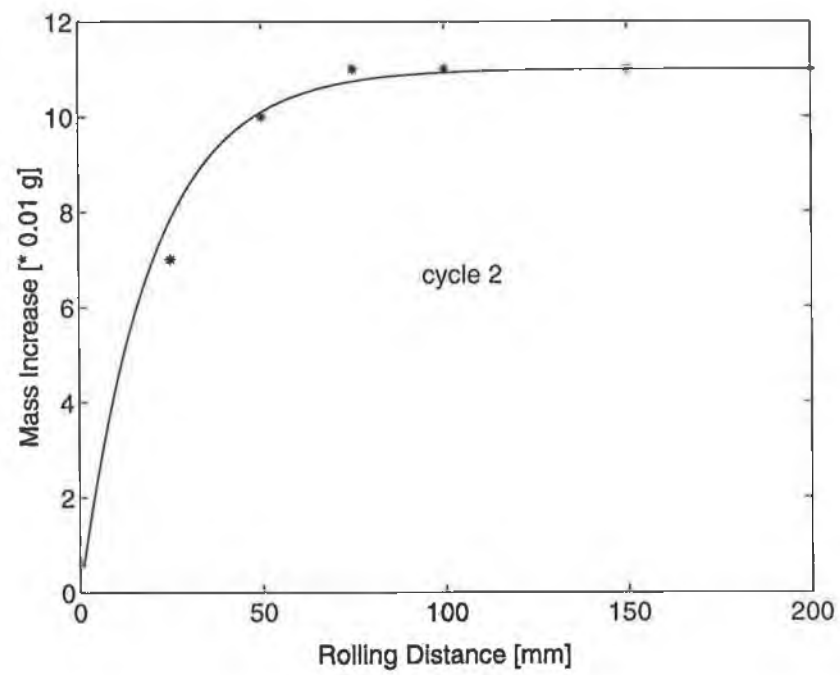


Figure 4.4. Increase in Diameter over 5 Coating Cycles for Flour

Figure 4.5 shows increase in mass vs. rolling distance for 5 coating cycles.

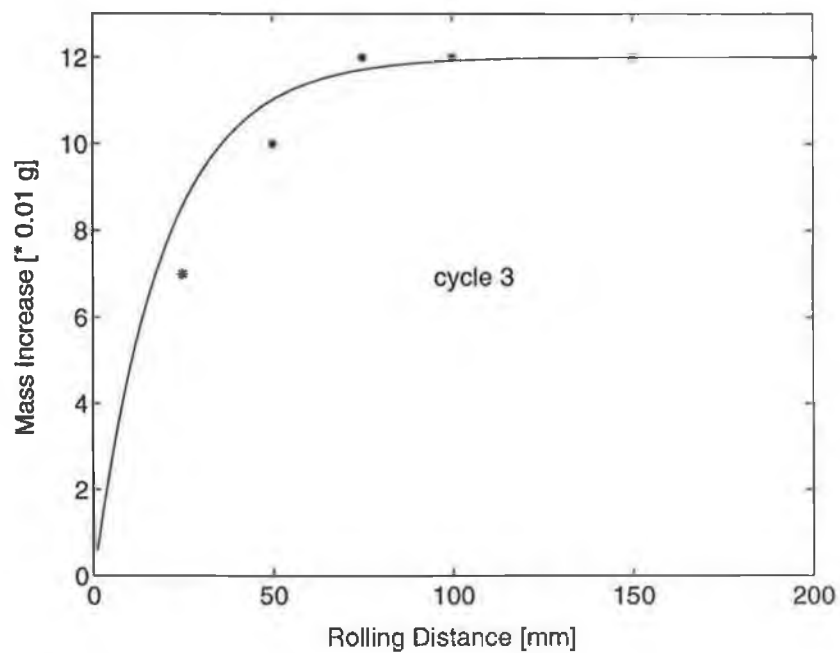


(a): $m = 7 * (1 - e^{-0.04 * X})$

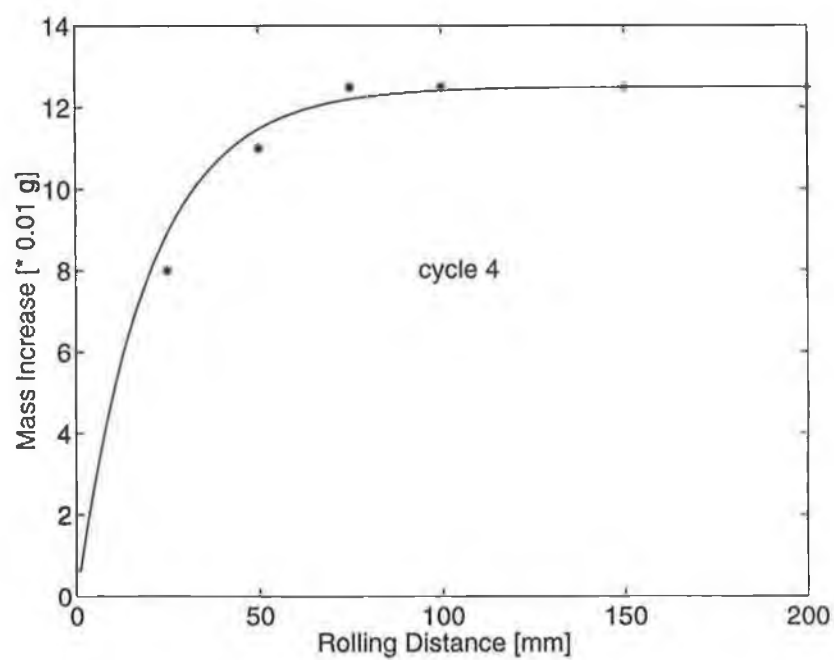


(b): $m = 11 * (1 - e^{-0.05 * X})$

Figure 4.5 Increase in mass vs. rolling distance

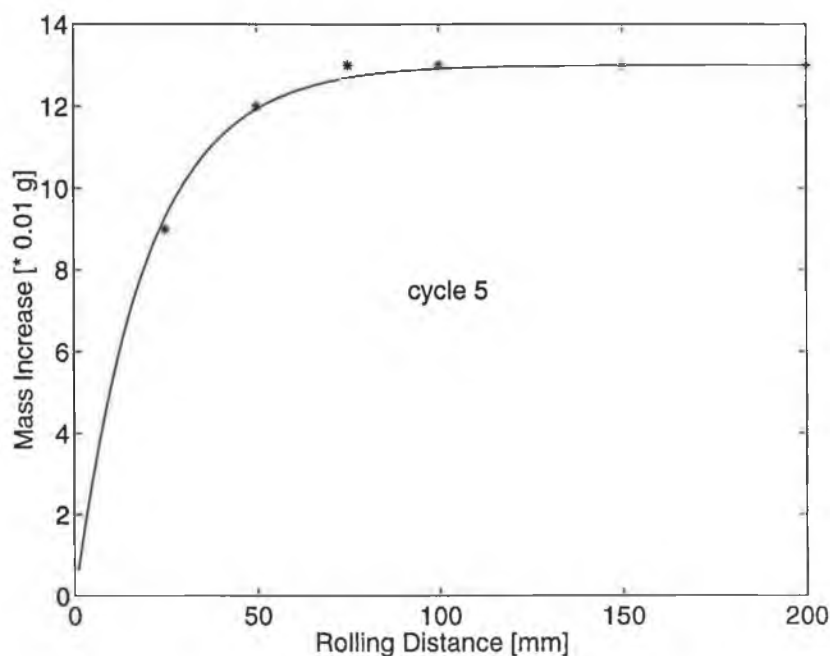


(c): $m = 12 * (1 - e^{-0.05 * X})$



(d): $m = 12.5 * (1 - e^{-0.05 * X})$

Figure 4.5 Increase in mass vs. rolling distance



$$(e): m = 13 * (1 - e^{-0.05 * X})$$

Figure 4.5 Increase in mass vs. rolling distance

The cumulative increase in mass has been plotted against the cumulative rolling distance as shown in figure 4.6.

In figure 4.6, the distances 0 - 200 mm represents cycle 1, 200 - 400 mm cycle 2, 400 - 600 mm cycle 3, 600 - 800 cycle 4, and 800 - 1000 cycle 5.

It can be seen from figures 4.3 and 4.4 that coating occurs over a short distance at the start of each cycle, the maximum increase in diameter occurs in cycle 2. This is because after the first coating cycle, the particle surface is almost all covered with flour which is more viscous, hence easy to coat more; on the other hand, compared with cycles 3, 4 and 5, during cycle 2, the coated particle is still small in diameter, which is almost half-buried in the powder bed, thus almost all the particle surface will be covered with the powder, when the particle becomes bigger (cycles 3, 4, and 5), only a small part of the particle is 'buried' in the powder bed, and not all its surface will be covered with the powder. As a result, for each single cycle, the diameter increase for a smaller particle is greater than that for a larger particle. Figures 4.5 and

4.6 show that mass increase for each cycle is greater than that for previous cycles. This is because with the coating process going on, the coating surface becomes greater and therefore more flour powder is coated on the particle.

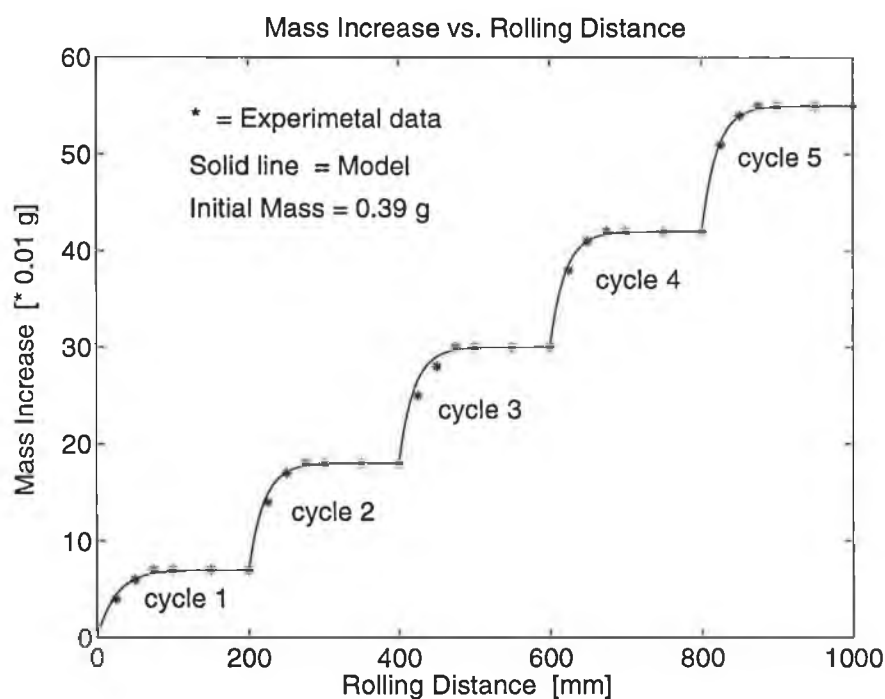


Figure 4.6. Increase in Mass over 5 Coating Cycles for Flour

4.3.2 Ground Almond

Test 2b

Particles: Two Marrowfat Peas (0.39 g each) were used in cycle 1
 12 Marrowfat Peas (0.39 g each) were used in cycle 2 - cycle 5
 the average initial diameter is 8.06 mm.

Coating Powder: Ground Almond

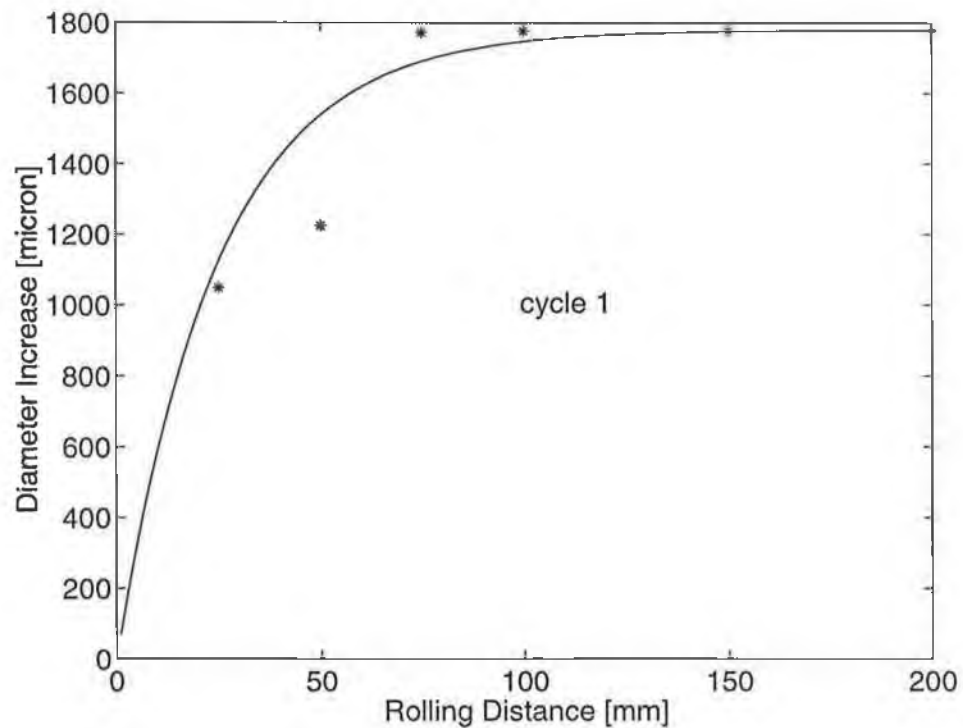
Binder: Water

Drying time: 10 min

Drying temperature: 21 °C

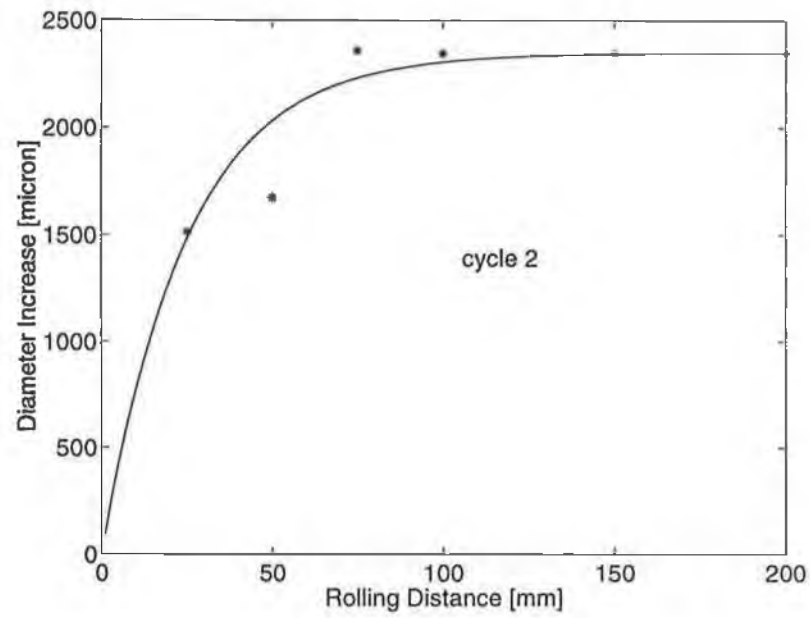
Release height: 300 mm

In the following figure, the star mark “*” stands for the average diameter from the experiment, each based on micrometer measurement. Measurement error is less than 0.01 mm. Figure 4.7 shows the increase in diameter against the rolling distance for 5 cycles of coating.

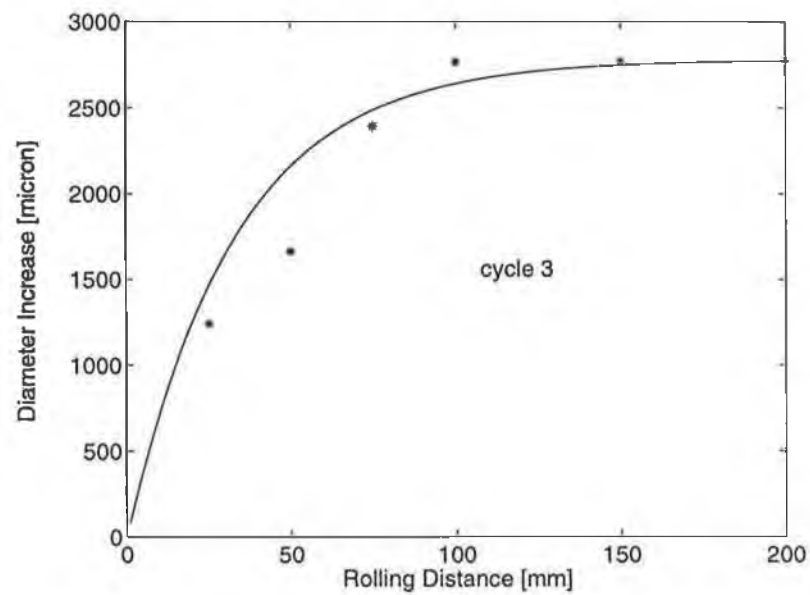


$$(a): d = 1780 * (1 - e^{-0.04 * X})$$

Figure 4.7 Increase in diameter against the rolling distance



$$(b): d = 2350 * (1 - e^{-0.04 * X})$$



$$(c): d = 2780 * (1 - e^{-0.03 * X})$$

Figure 4.7 Increase in diameter against the rolling distance

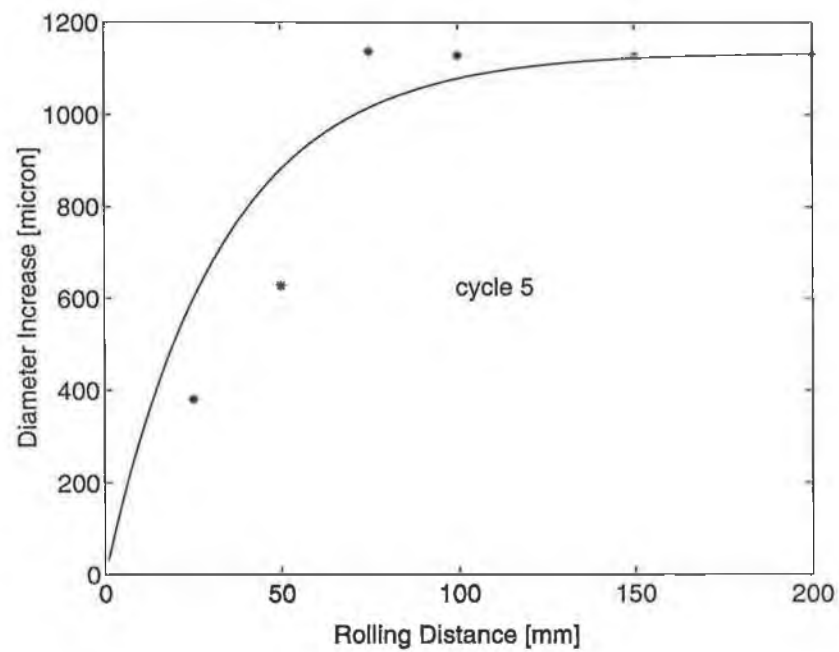
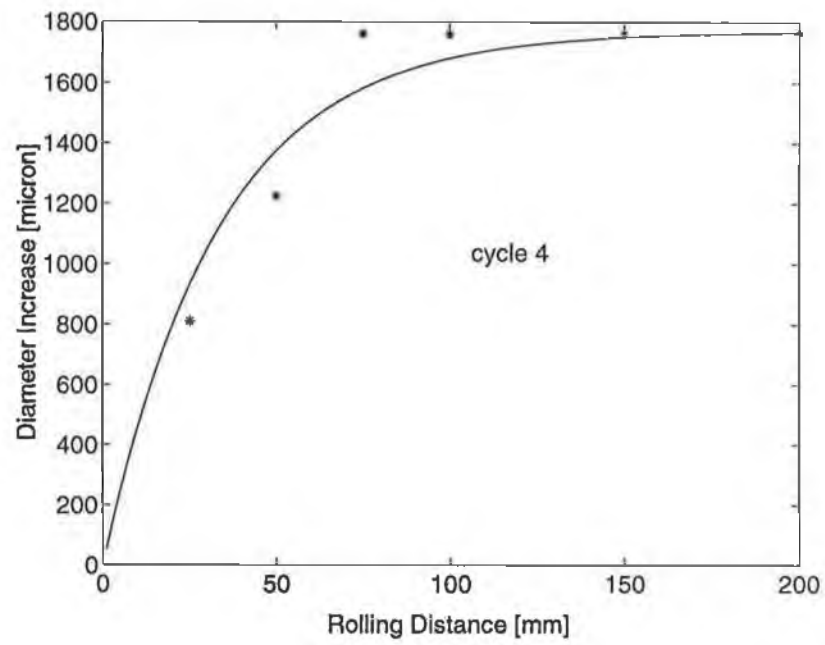


Figure 4.7 Increase in diameter against the rolling distance

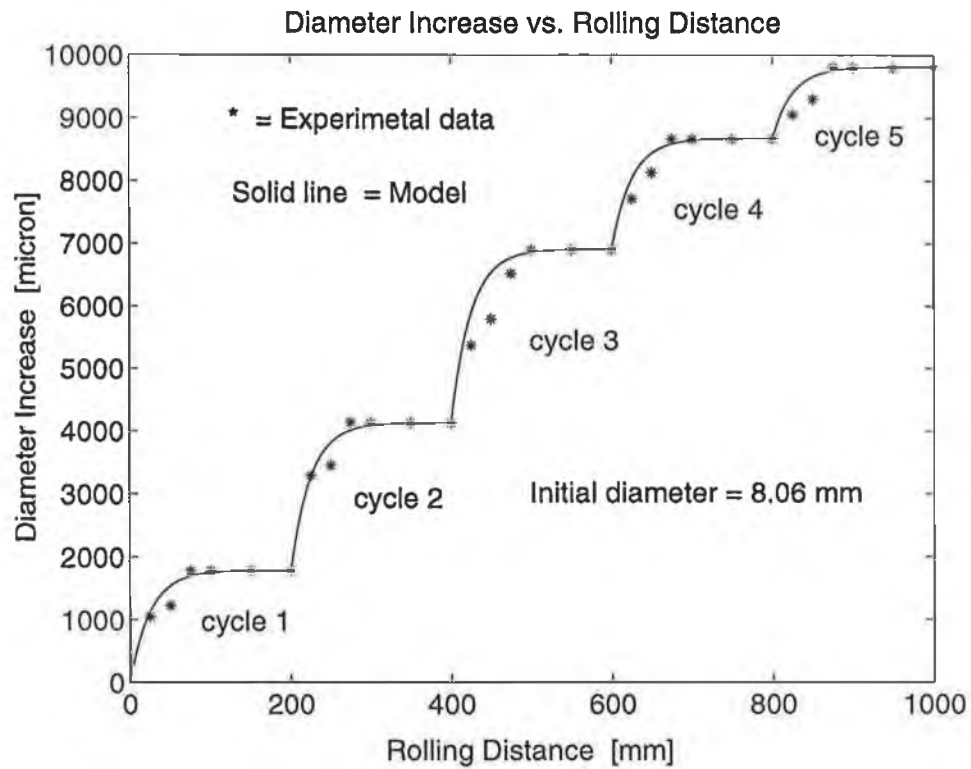
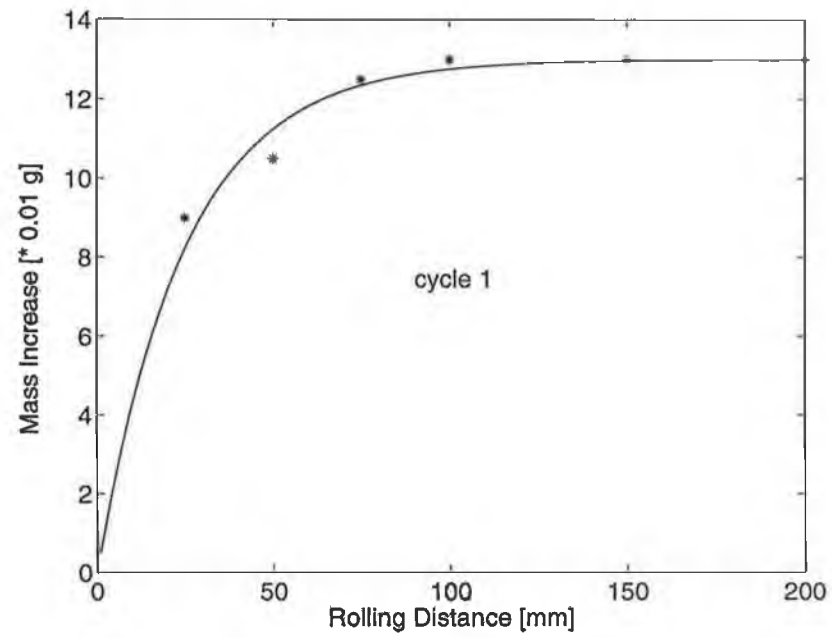


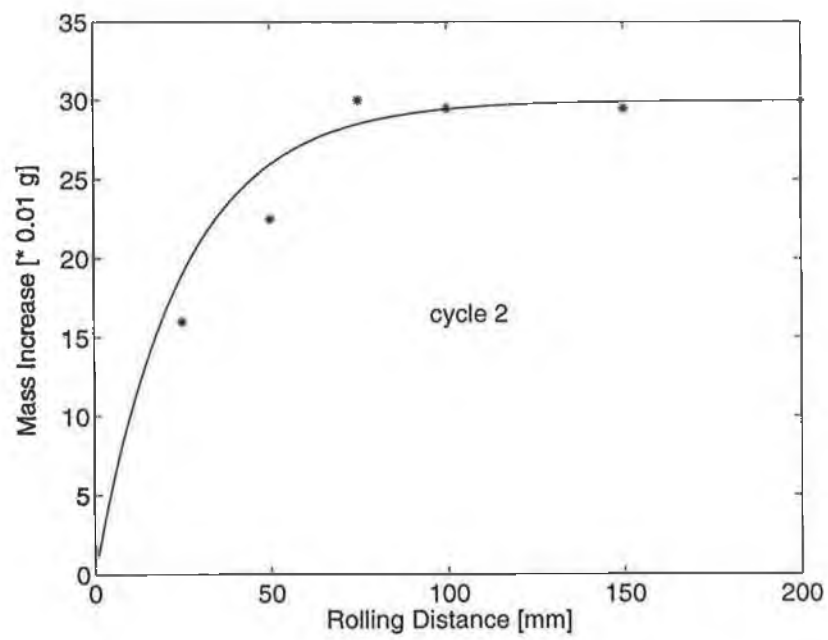
Figure 4.8. Increase in Diameter over 5 Coating Cycles for Almond

The cumulative increase in the diameter has been plotted in Figure 4.8. In this figure, the distances 0 - 200 mm represents cycle 1, 200 - 400 mm cycle 2, 400 - 600 mm cycle 3, 600 - 800 cycle 4, and 800 - 1000 cycle 5.

Figure 4.9 shows increase in mass versus rolling distance for 5 coating cycles.

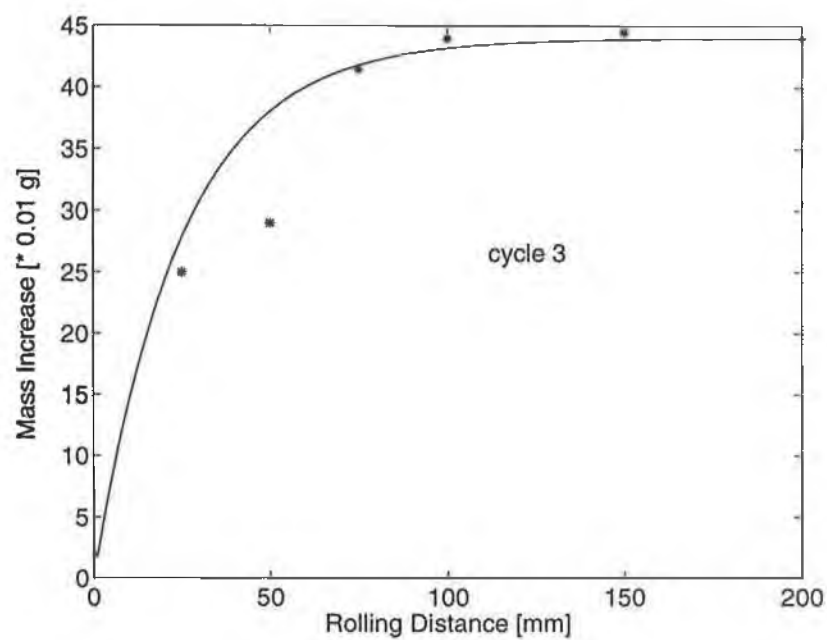


(a): $m = 13 * (1 - e^{-0.04 * X})$

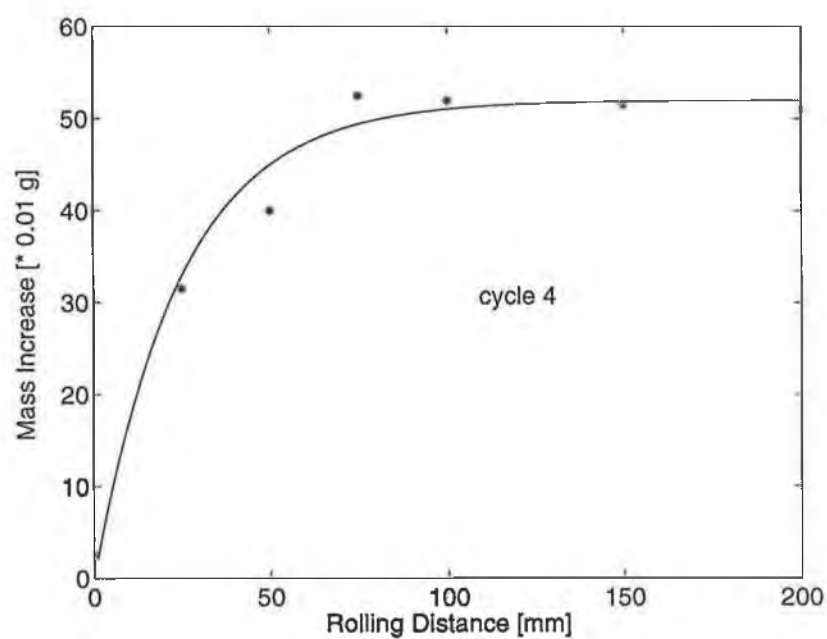


(b): $m = 30 * (1 - e^{-0.04 * X})$

Figure 4.9 Increase in mass vs. rolling distance

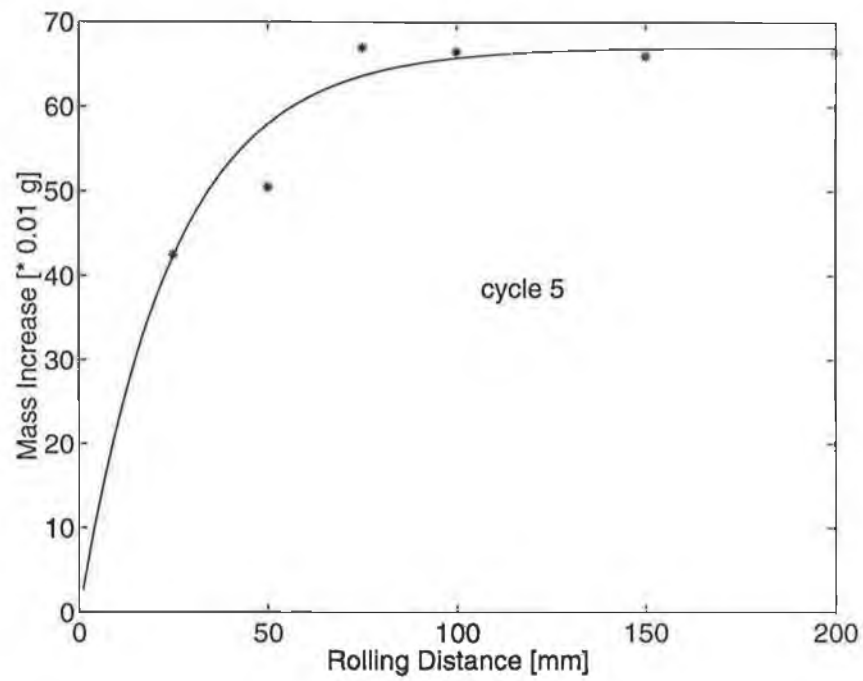


$$(c): m = 44 * (1 - e^{-0.04 * X})$$



$$(d): m = 52 * (1 - e^{-0.04 * X})$$

Figure 4.9 Increase in mass vs. rolling distance



$$(e): m = 67 * (1 - e^{-0.04 * x})$$

Figure 4.9 Increase in mass vs. rolling distance

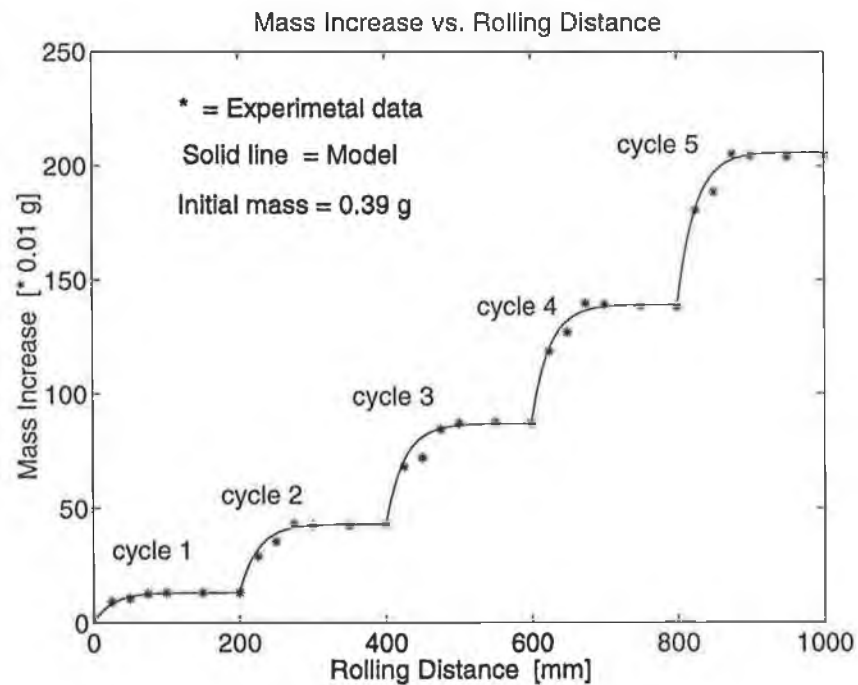


Figure 4.10. Increase in Mass over 5 Coating Cycles for Almond

In figure 4.10, the distances 0 - 200 mm represents cycle 1, 200 - 400 mm cycle 2, 400 - 600 mm cycle 3, 600 - 800 cycle 4, and 800 - 1000 cycle 5.

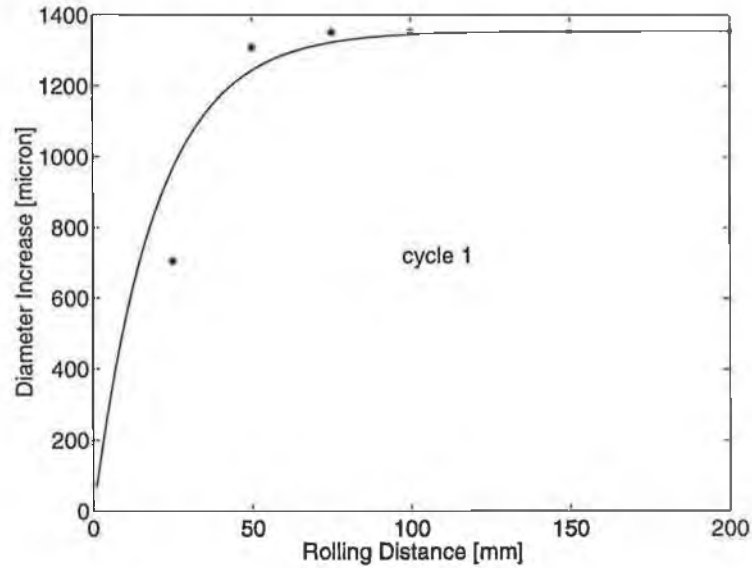
It can be seen from figures 4.7 and 4.8 that the maximum increase in diameter occurs in cycle 3. Similar to section 4.3.1, when the particle surface is covered with the coating powder (almond), its adhesiveness is increased, thus it can be coated with more powder (almond). However, unlike flour which is very fine, the almond powder is much greater and coating in the first cycle is not sufficient and even. After the second cycle, the particle surface is sufficiently covered by the almond powder. This is why the largest increase in diameter occurs in cycle 3, instead of in cycle 2, as is the case for flour coating in section 4.3.1. Figures 4.9 and 4.10 show that mass increase for each cycle is greater than that for previous cycles. This is because with the coating process going on, the coating surface becomes greater and therefore more almond powder is attached on the particle. Compared with flour coating, the tendency of mass increase for almond coating is more significant. The reason may be that the almond powder particles is bigger in size than flour powder, and initially (the first cycle) it is more difficult to attach itself onto the particle surface than it is in the case of flour coating.

4.3.3 Icing Sugar

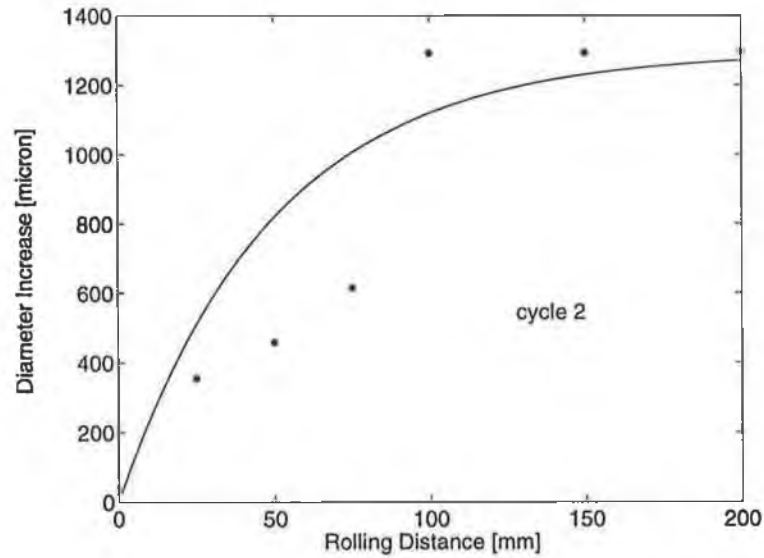
Test 2c

Particles:	Two Marrowfat Peas (0.39 g each) were used in cycle 1 12 Marrowfat Peas (0.39 g each) were used in cycle 2 - cycle 5 the average initial diameter is 8.17 mm.
Coating Powder:	Icing Sugar
Binder:	Water
Drying time:	10 min
Drying temperature:	21 °C
Release height:	300 mm

The increase in diameter for rolling distance is shown in figure 4.11 for five different cycles. In this figure, the points represented by “*” stands for the average diameter increase from the experiment, each based on micrometer measurement. Measurement error is less than 0.01 mm.

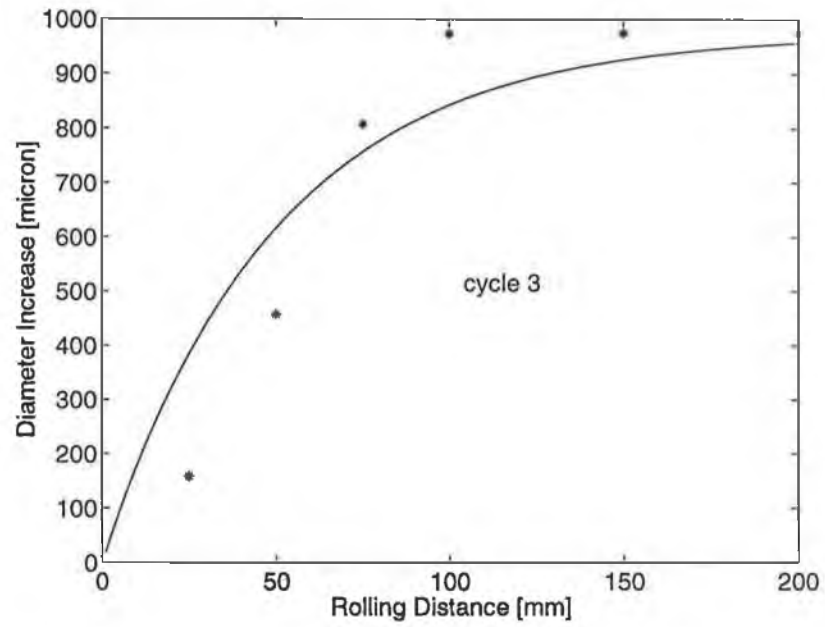


$$(a): d = 1355 * (1 - e^{-0.05 * X})$$

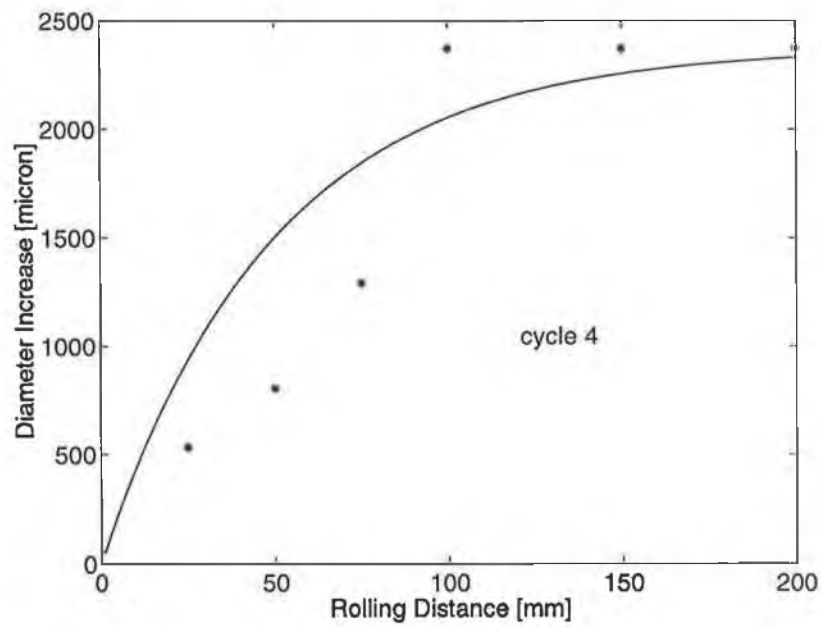


$$(b): d = 1295 * (1 - e^{-0.02 * X})$$

Figure 4.11 Increase in diameter vs. rolling distance

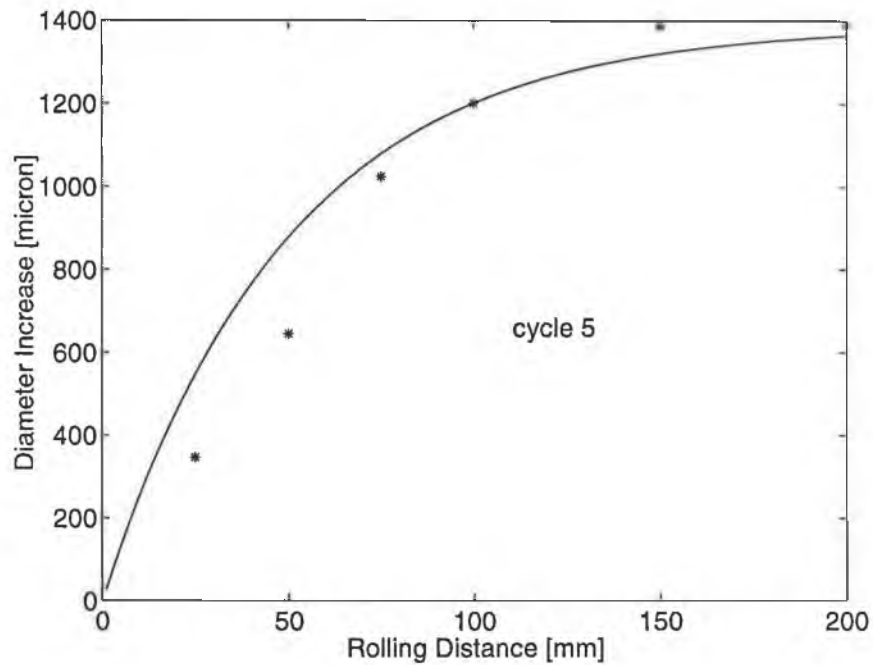


$$(c): d = 975 * (1 - e^{-0.02 * X})$$



$$(d): d = 2375 * (1 - e^{-0.02 * X})$$

Figure 4.11 Increase in diameter vs. rolling distance



$$(e): d = 1390 * (1 - e^{-0.02 * X})$$

Figure 4.11 Increase in diameter vs. rolling distance

The cumulative increase in diameter is shown in figure 4.12 over a total rolling distance of 1000 mm. In this figure, the rolling distances 0 - 200 mm represents cycle 1, 200 - 400 mm cycle 2, 400 - 600 mm cycle 3, 600 - 800 cycle 4, and 800 - 1000 cycle 5.

It is clear that if the particle can be kept continuously wet as it keeps rolling on the powder bed, the total increase in diameter may be achieved over a rolling distance of less than 500 mm. This is because in each cycle, most of the increase in diameter occurs over the first 100 mm of the 200 mm rolling distance.

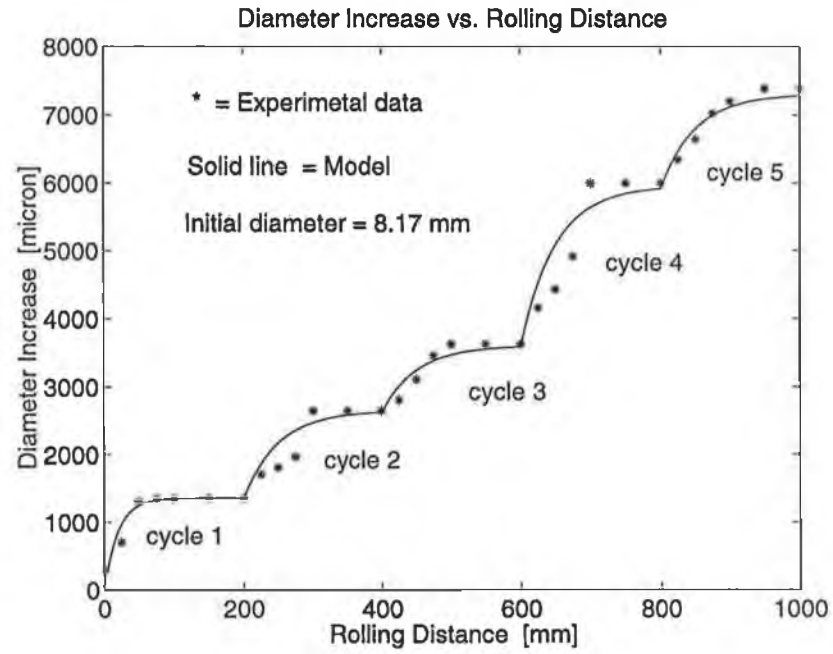
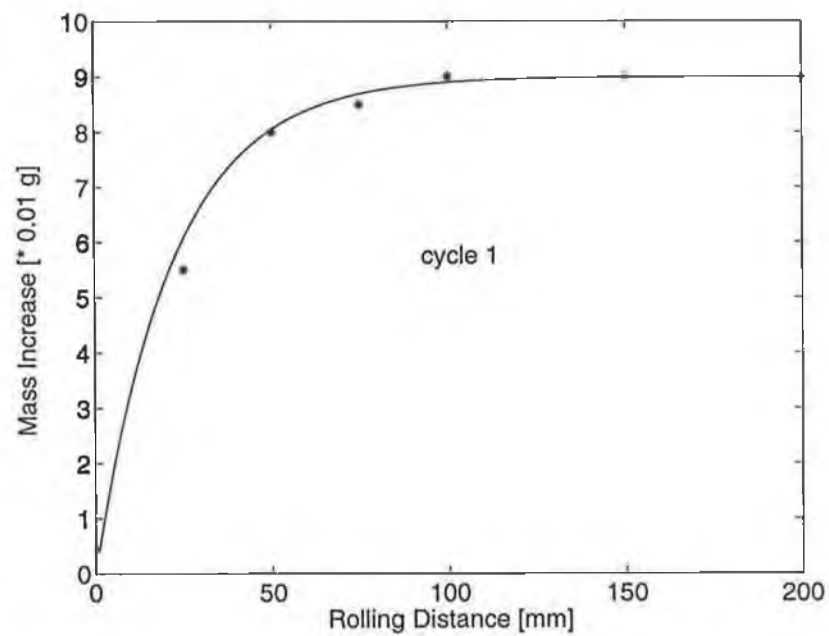


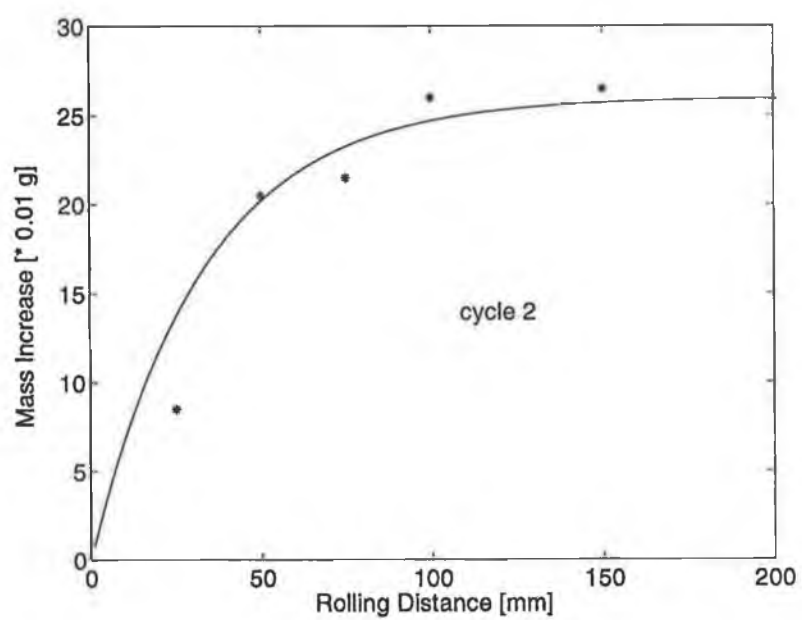
Figure 4.12. Increase in Diameter over 5 Coating Cycles for Icing Sugar

The increase in mass over five successive cycles of rolling is shown in figure 4.13.

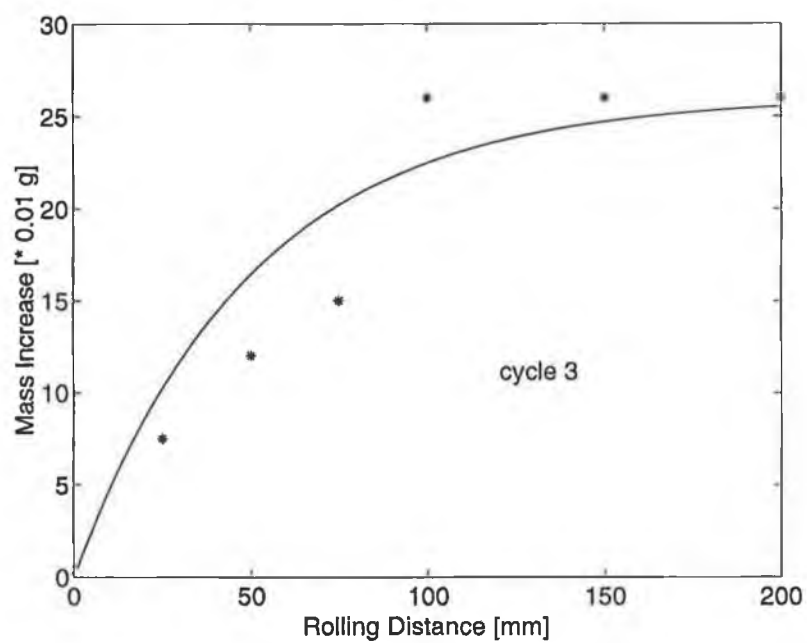


$$(a): m = 9 * (1 - e^{-0.045 * X})$$

Figure 4.13 Increase in mass vs. rolling distance

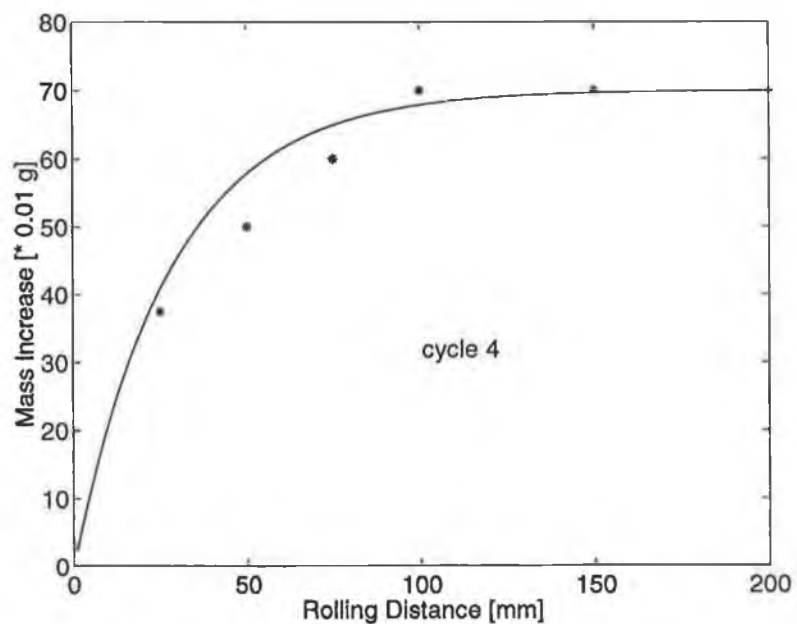


(b): $m = 26 * (1 - e^{-0.03 * X})$

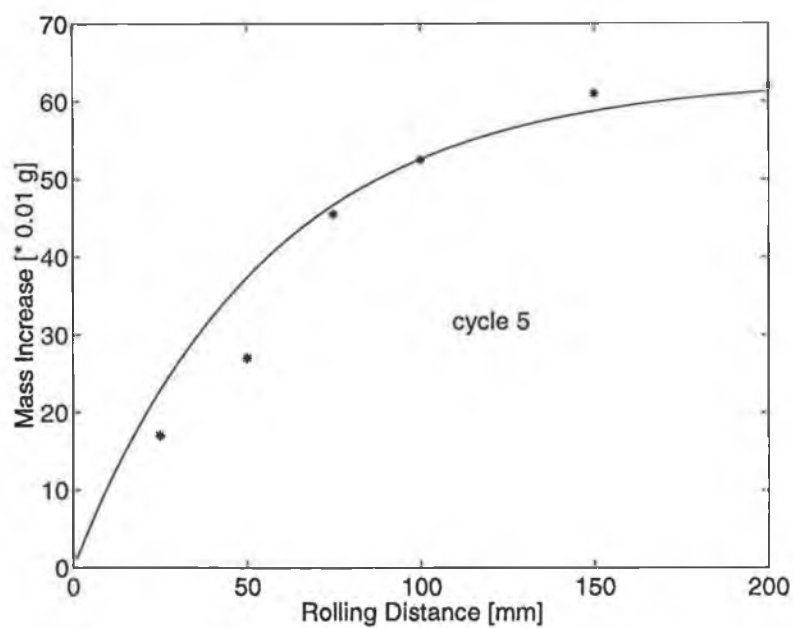


(c): $m = 26 * (1 - e^{-0.02 * X})$

Figure 4.13 Increase in mass vs. rolling distance



(d): $m = 70 * (1 - e^{-0.035 * X})$



(e): $m = 63 * (1 - e^{-0.018 * X})$

Figure 4.13 Increase in mass vs. rolling distance

The cumulative increase in mass is plotted in figure 4.14 over a total rolling

distance of 1000 mm. In figure 4.14, the distances 0 - 200 mm is for cycle 1, 200 - 400 mm is for cycle 2, 400 - 600 mm is for cycle 3, 600 - 800 mm is for cycle 4, 800 - 1000 mm is for cycle 5.

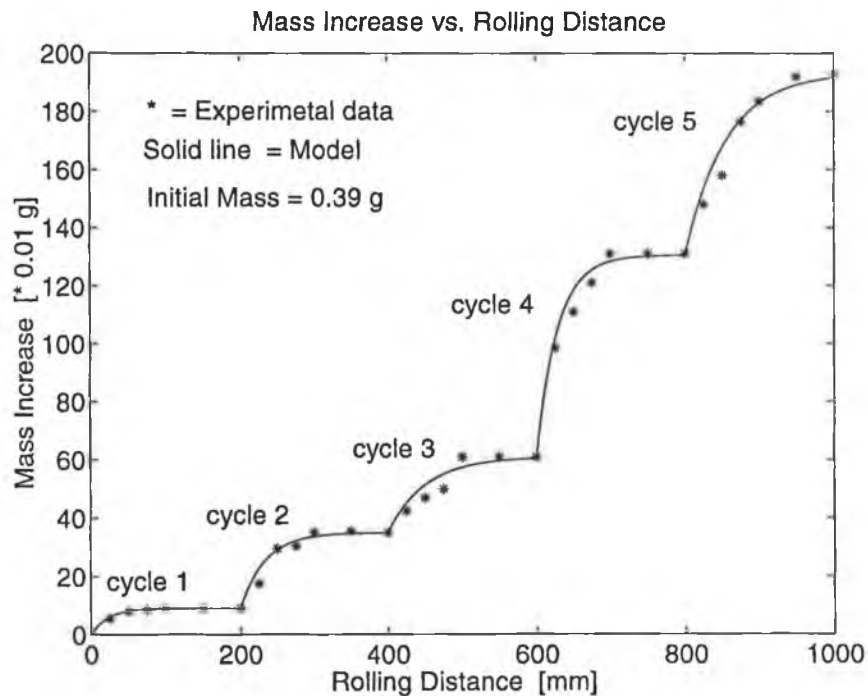


Figure 4.14. Increase in Mass over 5 Coating Cycles for Icing Sugar

Figures 4.11 to 4.14 show that icing sugar coating has the similar pattern of diameter and mass increase to the patterns of flour coating and ground almond coating. However due to its greater dissolvability, icing sugar dissolves very quickly and is capable of collecting more sugar powders in a more irregular pattern, the shape of the coated particles is somewhat like cubic instead of spherical as is the case for flour and ground almond. Because of the irregular shape of the coated particles, the diameter and mass increase of icing sugar coating are not as regular as in the case for flour and ground almond coating.

This multi-cycle coating process as indicated by the above experiments is used primarily to demonstrate the principle of thick coating, that is, thick coating can be

achieved by the multi-cycle coating process. Because of this, all the experiments were carried out over only five cycles.

4.4 EFFECT OF COATING POWDER

In order to compare the increase in diameter and mass when different powders were used, the results are shown in figure 4.15 and 4.16.

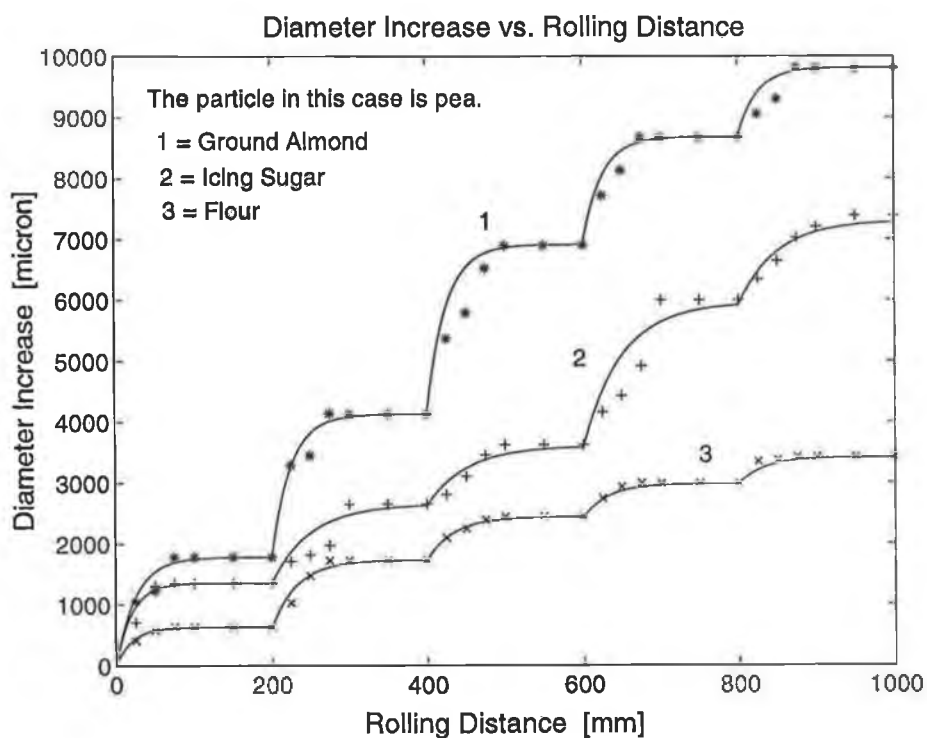


Figure 4.15. Effect of Coating Powder on Diameter Increase

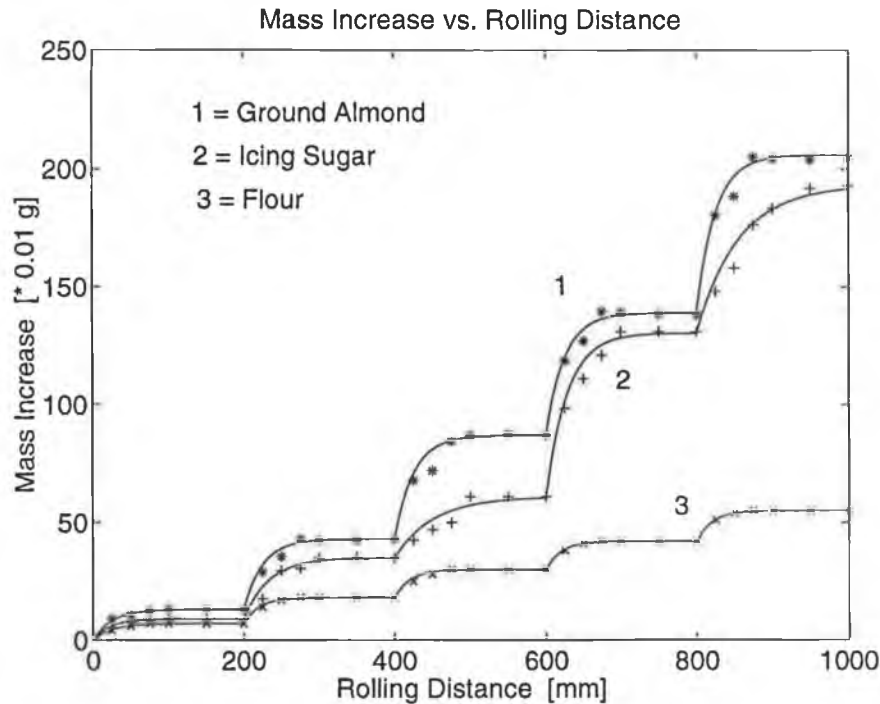


Figure 4.16. Effect of Coating Powder on Mass Increase

It can be seen from figures 4.15 and 4.16 for all these powder types, the coating thickness and mass increase when the number of rolling cycle increases, and within each cycle, the increases of coating thickness and mass generally occur over the first half of the coating distance in each cycle, and the increases of the coating thickness and mass over the second half coating distance are neglectable. Diameter (thickness) and mass changes are different using different coating powders. It is found that for the same coating distance of 1000 mm, the diameter increase is 1.9 times greater, mass increase is 2.7 times greater using ground almond than those using flour; diameter increase is 1.1 times greater, mass increase is 2.5 times greater using icing sugar than those using flour. This is because of the different characteristics of the coating powder. The experiments show that in terms of coating thickness and mass, ground almond is better than icing sugar and icing sugar is better than flour as coating powders.

4.5 SURFACE CHARACTERISTICS OF COATED PARTICLES

The surface of flour coated marrowfat peas is generally smooth. The coated flour is difficult to split off (two upper particles in figure 4.17) and the shape of coated particles is almost spherical. This is probably due to the higher adhesiveness of flour when sprayed with water. The surface of icing sugar coated marrowfat peas is also smooth. The coated icing sugar is difficult to peel off (two lower particles in the photograph) and the shape of coated particles is not spherical. The surface of ground almonds coated marrowfat peas is not smooth. The coated ground almonds are easy to peel off (two middle particles in the photograph). The shape of coated particles are nominally spherical. This is probably due to the lower adhesive bonding property of ground almond mixed with water.



Figure 4.17. Photograph for Coated Particles

4.6 EFFECT OF PARTICLE SIZE

Tests were carried out to determine the effect of the diameter of the articles on the coating efficiency. Figure 4.18 shows the increase in diameter for rolling distance

of 200 mm for Peas of 8.35 mm and Beans of 4.46 mm diameter.

Particles: a: Two Marrowfat Peas (average diameter: 8.35 mm)
 b: Two Moong Beans (average diameter: 4.46 mm)
 Note: greater Marrowfat pea particles were not available in the
 market, and smaller Moong beans were beyond the reliable
 measurement range.

Coating powder: Flour
 Binder: Water
 Drying time: 10 min
 Drying temperature: 21 °C
 Release height: 300 mm

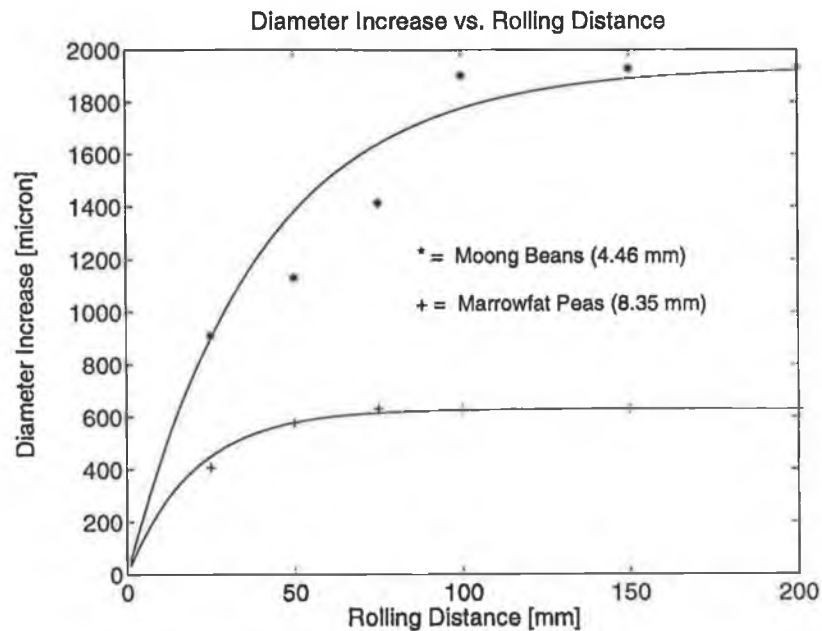


Figure 4.18. Effect of the Particle Size on Diameter Increase for Flour

Data fit for Moong Bean (4.46 mm) is: diameter increase = $1935 * (1 - e^{-0.025 * x})$, for Marrowfat Peas (8.35 mm) is: $\Delta d = 630 * (1 - e^{-0.05 * x})$, where $x =$

rolling distance.

Figure 4.19 shows the increase in the mass during rolling over a distance of 200 mm for two different sized particles as those in figure 4.18.

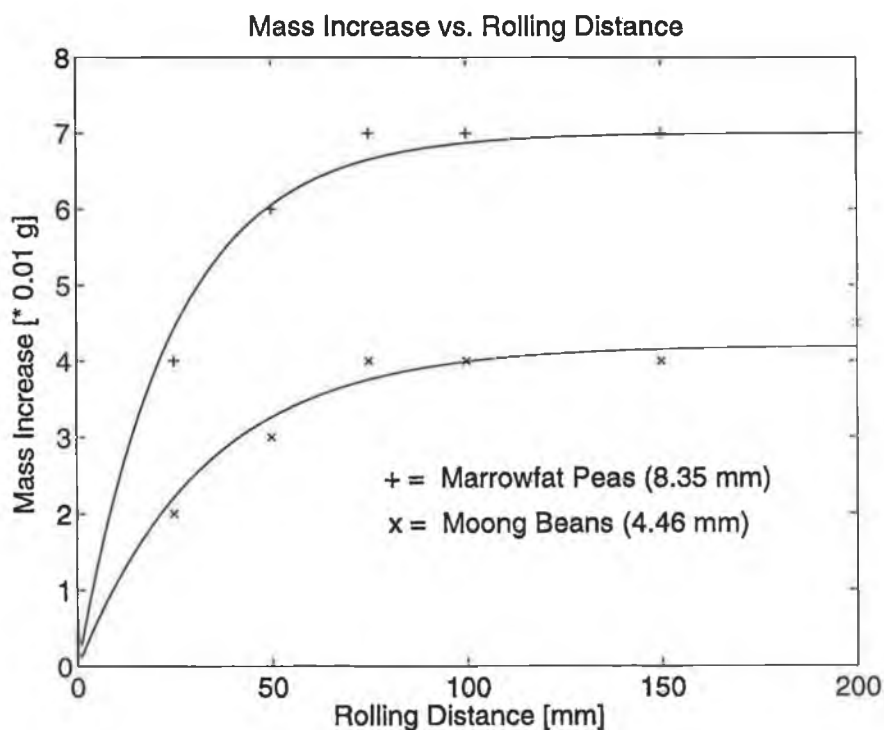


Figure 4.19. Effect of the Particle Size on Mass Increase for Flour

Data fit for Moong Bean (4.46 mm) is: mass increase = $4.2 * (1 - e^{-0.03 * x})$, for Marrowfat Peas (8.35 mm) is: $\Delta m = 7 * (1 - e^{-0.04 * x})$, where x = rolling distance.

These tests were also carried out with almond powder and icing sugar and the results are presented as follows (test 4 and test 5).

Particles: a: Two Marrowfat Peas (average diameter: 8.35 mm)
 b: Two Moong Beans (average diameter: 4.46 mm)
 Coating powder: Ground Almond
 Binder: Water
 Drying time: 10 min
 Drying temperature: 21 °C
 Release height: 300 mm

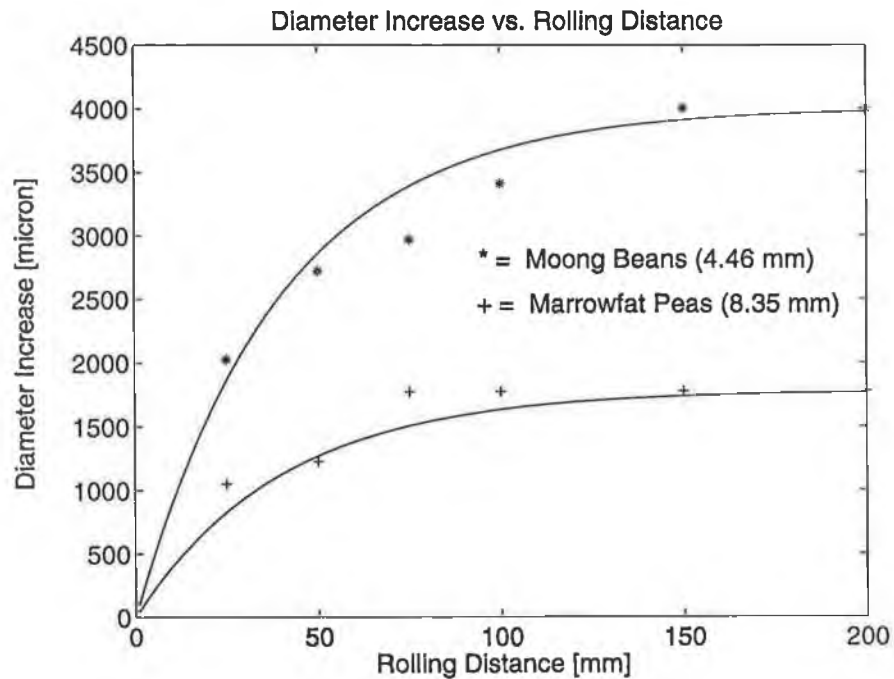


Figure 4.20. Effect of the Particle Size on Diameter Increase for Almond

Data fit for Moong Bean (4.46 mm) is: diameter increase = $4005 * (1 - e^{-0.025 * x})$, for Marrowfat Peas (8.35 mm) is: $\Delta d = 1780 * (1 - e^{-0.04 * x})$, where x = rolling distance.

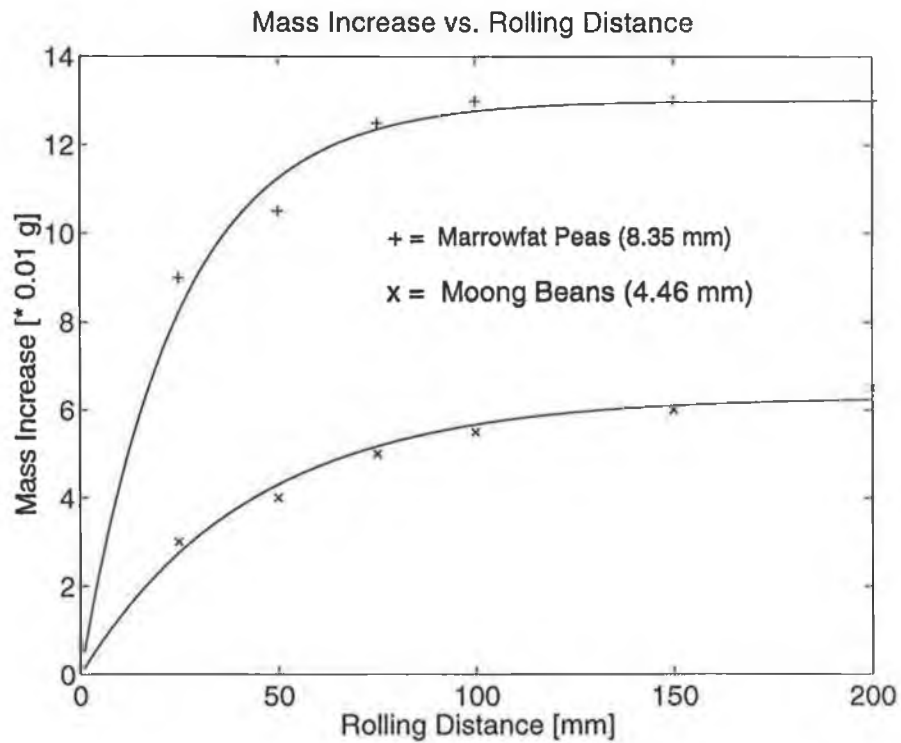


Figure 4.21. Effect of the Particle Size on Mass Increase for Almond

Data fit for Moong Bean (4.46 mm) is: mass increase = $6.3 * (1 - e^{-0.023 * x})$,
 for Marrowfat Peas (8.35 mm) is: $\Delta m = 13 * (1 - e^{-0.04 * x})$.

Particles:	a: Two Marrowfat Peas (average diameter: 8.35 mm)
	b: Two Moong Beans (average diameter: 4.46 mm)
Coating powder:	Icing Sugar
Binder:	Water
Drying time:	10 min
Drying temperature:	21 °C
Release height:	300 mm

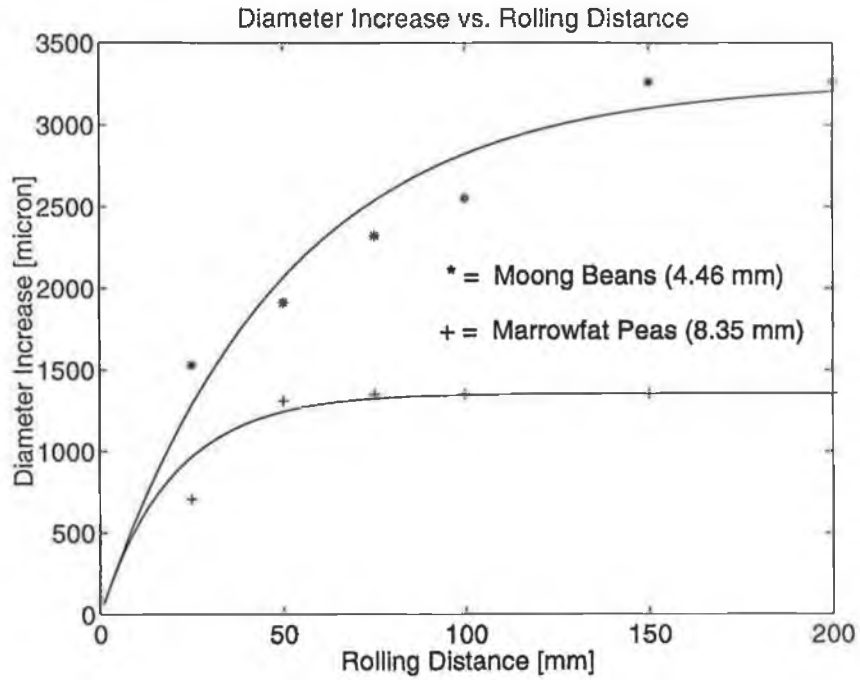


Figure 4.22. Effect of the Particle Size on Diameter Increase for Sugar

Data fit for Moong Bean (4.46 mm) is: diameter increase = $3265 * (1 - e^{-0.02 * x})$, for Marrowfat Peas (8.35 mm) is: $\Delta d = 1355 * (1 - e^{-0.05 * x})$, where x = rolling distance.

Data fit for Moong Bean (4.46 mm) is: mass increase = $6.6 * (1 - e^{-0.015 * x})$, for Marrowfat Peas (8.35 mm) is: $\Delta m = 9 * (1 - e^{-0.045 * x})$, where x = rolling distance.

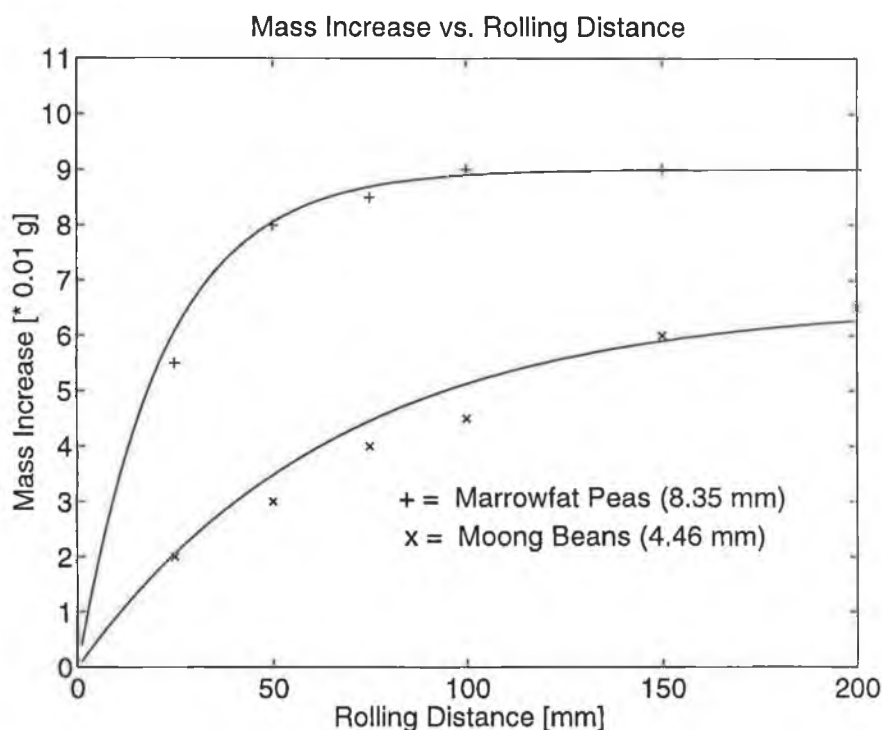


Figure 4.23. Effect of the Particle Size on Mass Increase for Sugar

It can be seen from figures 4.18 to 4.23 that coating occurs over a short distance and is greatly influenced by the size of particles. Figures 4.18 and 4.19 show that when the particle size is 4.5 mm, the diameter increase is approximately 112 % greater and the mass increase is approximately 40 % less than those when the particle size is 8.3 mm by using flour as the coating powder. Figures 4.20 and 4.21 show that when the particle size is 4.5 mm, the diameter increase is approximately 115 % greater and the mass increase is approximately 52 % less than those when the particle size is 8.3 mm by using ground almond as the coating powder. Figures 4.22 and 4.23 show that when the particle size is 4.5 mm, the diameter increase is approximately 141 % greater and the mass increase is approximately 27 % less than those when the particle size is 8.3 mm by using icing sugar as the coating powder. That is, coating on smaller particles will result in greater percentage diameter increase and less percentage mass increase for all three different coating powders. This may be explained as follows. The smaller particles are almost 'half-buried' in the coating powder bed while rolling down, and in each single cycle almost all the surface of the small particles is covered with coating powder. Since the measured diameter is the

average of the measurements in three different directions, it follows that for the small particles the measurements in three directions are very close and therefore the final diameter increase is also very close to the diameter increase measured along the coating direction. For bigger particles only a small part of their surface is in contact with the powder bed and can be covered with powder afterwards. This means that of the three measurements in three directions, one diameter increase measurement is significantly less than the two measured along the coating direction and the final diameter increase is therefore less than the diameter increase measured along the coating direction. This is the reason why diameter increase is greater for smaller particles than for bigger particles.

However, since the bigger particles have greater peripheries they obviously have greater area available for coating for each single cycle, and therefore, mass increase for the bigger particles is greater than for the smaller particles.

4.7 EFFECT OF BINDER

Tests were carried out to investigate the influence of the binder material on the diameter increase during coating by rolling the particles over 200 mm (test 6).

Particles:	Two Marrowfat Peas (0.39g each)
Coating powder:	Flour
Binder:	a: Water
	b: Olive Oil
	c: 16 wt % Lactose + 0.085 g/l Texapon.
Drying time:	10 min
Drying temperature:	21 °C
Release height:	300 mm

Figures 4.24 and 4.25 show the results of diameter increase of marrowfat peas for 3 different binder materials; the coating material being flour.

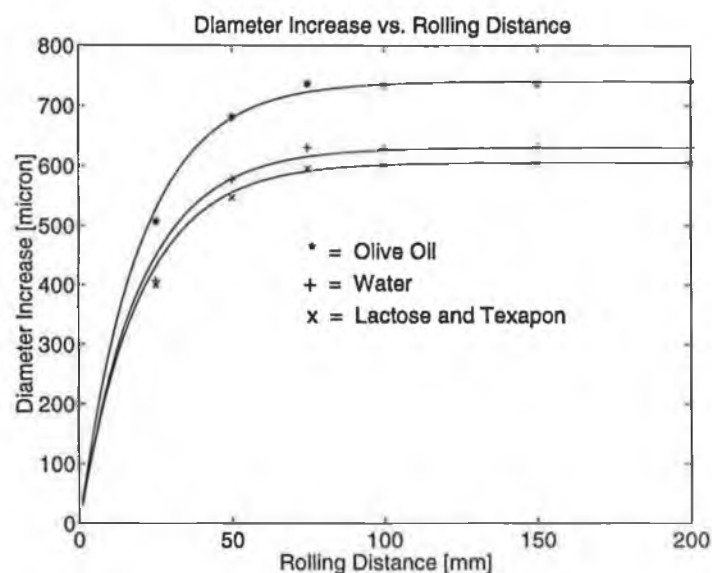


Figure 4.24. Effect of Binder on Diameter Increase for Flour

Data fit for Olive Oil is: diameter increase = $740 * (1 - e^{-0.05 * x})$, for Water is: $\Delta d = 630 * (1 - e^{-0.05 * x})$, for Lactose and Texapon is: $\Delta d = 605 * (1 - e^{-0.05 * x})$, where x = rolling distance.

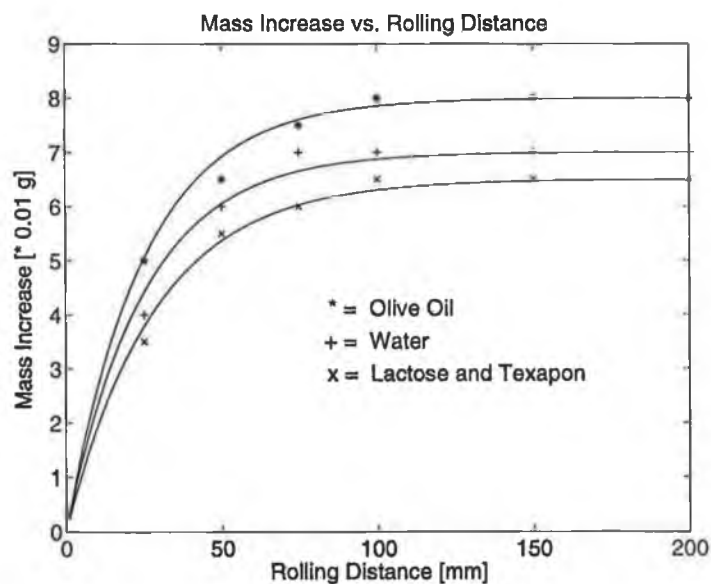


Figure 4.25. Effect of Binder on Mass Increase for Flour

Data fit for Olive Oil is: $\text{mass increase} = 8 * (1 - e^{-0.04 * x})$, for Water is: $\Delta m = 7 * (1 - e^{-0.04 * x})$, for Lactose and Texapon is: $\Delta m = 6.5 * (1 - e^{-0.035 * x})$, where x = rolling distance.

Figure 4.26 and 4.27 show these results for the same three binder materials when the coating material is ground almond (test 7).

Particles: Two Marrowfat Peas (0.39g each)
 Coating powder: Ground Almond
 Binder:
 a: Water
 b: Olive Oil
 c: 16 wt % Lactose + 0.085 g/l Texapon.
 Drying time: 10 min
 Drying temperature: 21 °C
 Release height: 300 mm

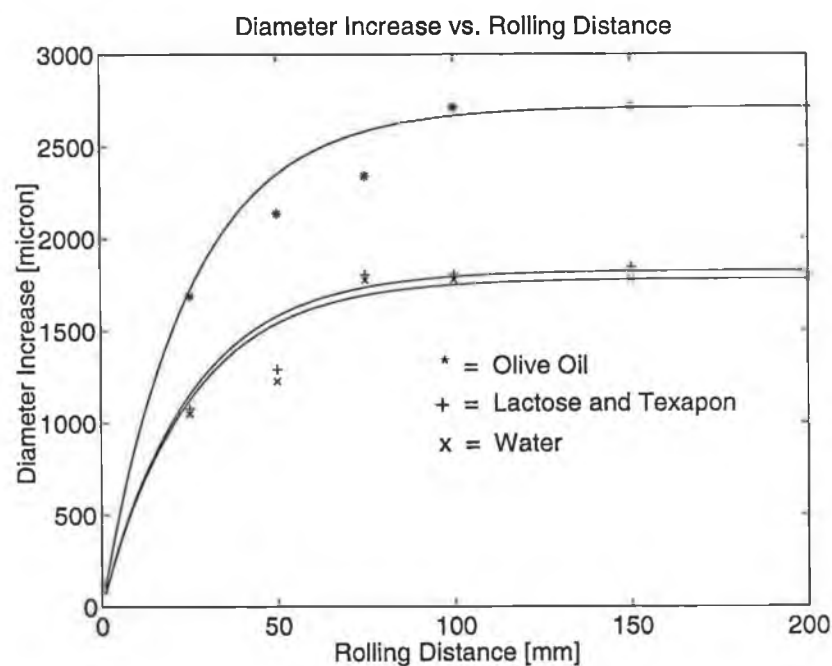


Figure 4.26. Effect of Binder on Diameter Increase for Ground Almond

Data fit for Olive Oil is: diameter increase = $2716 * (1 - e^{-0.04 * x})$, for Lactose and Texapon is: $\Delta d = 1825 * (1 - e^{-0.04 * x})$, and for Water is: $\Delta d = 1780 * (1 - e^{-0.04 * x})$, where x = rolling distance.

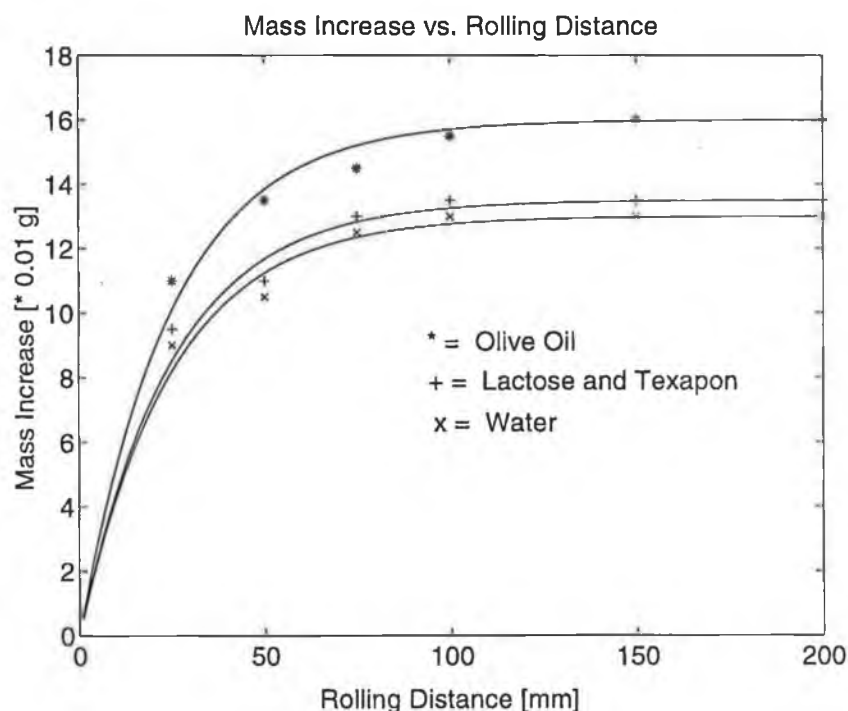


Figure 4.27. Effect of Binder on Mass Increase for Ground Almond

Data fit for Olive Oil is: mass increase = $16 * (1 - e^{-0.04 * x})$, for Lactose and Texapon is: $\Delta m = 13.5 * (1 - e^{-0.04 * x})$, and for Water is: $\Delta m = 13 * (1 - e^{-0.04 * x})$, where x = rolling distance.

It can be seen from figures 4.24 to 4.27 that coating occurs over a short distance and is influenced by the binder. Figures 4.24 and 4.25 show that when the binder is olive oil, the diameter increase is approximately 22% greater and the mass increase is approximately 23% greater than those when the binder is lactose and texapon solution by using flour as the coating powder; when the binder is water, the

diameter increase and mass increase are 17% and 8% greater than those when the binder is lactose and texapon solution, respectively.

Figures 4.26 and 4.27 show that when the binder is olive oil, the diameter increase is approximately 53% greater and the mass increase is approximately 23% greater than those when the binder is water by using ground almond as the coating powder; when the binder is lactose and texapon, the diameter increase and mass increase are very close to those when the binder is water (within a margin of 2% and 4%, respectively).

Figures 4.24 to 4.27 also show that both the maximum diameter and the mass increase considerably when olive oil is used as binder and flour is used as the coating powder. This is probably due to the fact that the viscosity of olive oil is higher than those of water and lactose + texapon. The viscosity of water and lactose + texapon is almost the same, and similar effectiveness is seen in figure 4.26 for ground almond as the coating powder, where water was marginally better for flour.

Figure 4.28 and 4.29 shows diameter and mass increase for the same three binder materials when icing sugar is used as the coating materials.

Particles:	Two Marrowfat Peas (0.39g each)
Coating powder:	Icing Sugar
Binder:	a: Water
	b: Olive Oil
	c: 16 wt % Lactose + 0.085 g/l Texapon.
Drying time:	10 min
Drying temperature:	21 °C
Release height:	300 mm

Data fit for lactose and texapon is: diameter increase = $1376 * (1 - e^{-0.05 * x})$,

for water is: $\Delta d = 1355 * (1 - e^{-0.05 * x})$, and for olive oil is: $\Delta d = 923 * (1 - e^{-0.028 * x})$, where x = rolling distance.

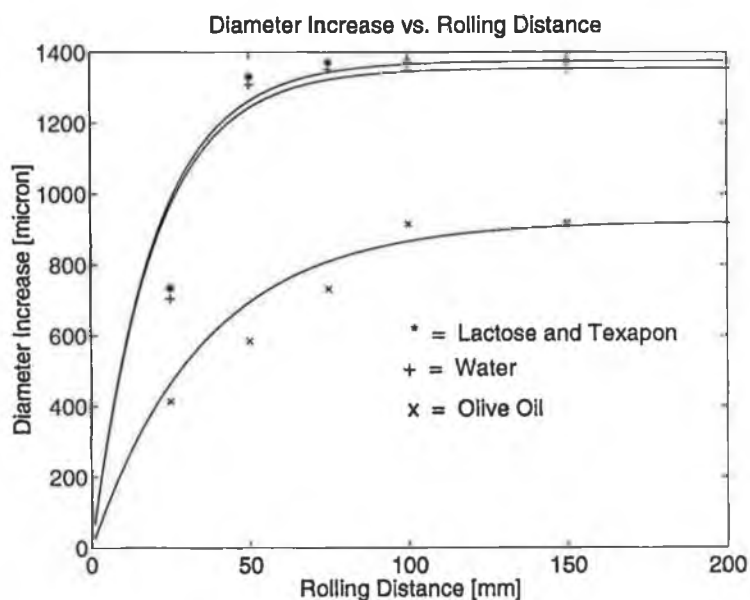


Figure 4.28. Effect of Binder on Diameter Increase for Icing Sugar

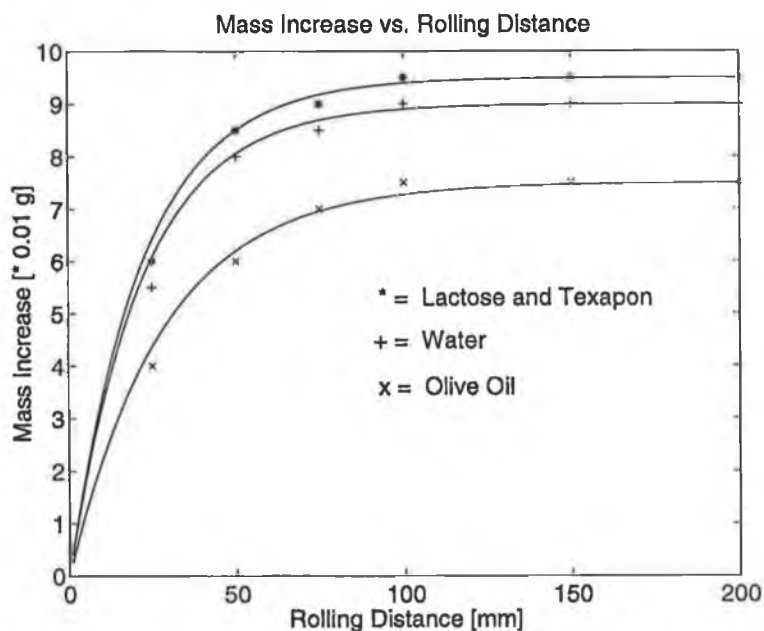


Figure 4.29. Effect of Binder on Mass Increase for Icing Sugar

Data fit for lactose and texapon is: mass increase = $9.5 * (1 - e^{-0.045 * x})$, for

water is: $\Delta m = 9 * (1 - e^{-0.045 * x})$, and for olive oil is: $\Delta m = 7.5 * (1 - e^{-0.035 * x})$, where x = rolling distance.

Figures 4.28 and 4.29 show that when the binder is lactose and texapon solution, the diameter increase is approximately 49% greater and the mass increase is approximately 27% greater than those when the binder is olive oil by using icing sugar as the coating powder; when the binder is water, the diameter and mass increases are very close to the diameter and mass increases, when binder is lactose and texapon solution. This is probably due to the higher dissolvability of the icing sugar in water and lactose + texapon solution and relatively lower dissolvability of the icing sugar in olive oil. The dissolvability of icing sugar in water and in lactose + texapon solution is almost the same.

4.8 MODELLING FOR POWDER COATING

4.8.1 Introduction

The experimental work is necessary to test the principle of coating by rolling, and to gain experience of the degree of success achieved using various materials. However, the project requires that an understanding of the 'why' behind phenomena is achieved, and ideally that this understanding is formulated into a model, which can be used to predict the behaviour when some aspect of the system is changed.

A model of a system can be something very simple. Essentially, a complex physical component is approximated by one for which the behaviour is less difficult to describe (for example an irregular shaped particle can be approximated by a sphere, an agglomerate of small powder particles can be approximated by a solid particle). In making such approximations, it is obviously no longer describing the system *exactly*, but it can be used to carry out calculations of how the system behaves (for example:

based on mechanics, chemistry etc.) giving results which can be very useful.

Essentially modelling involves two stages:

- Approximate the system by 'ideal' components
- Describe this idealised system using mathematics

4.8.2 The Mathematical Model

The task in this project is to model the coating of a particle as it rolls down a slope, over a bed of powder. The particle starts off wet, which facilitates collection of powder as it rolls. However, as it gathers more and more powder, its ability to collect powder diminishes, and so it collects at a slower rate, until eventually it does not collect any more powder. The situation is complicated by the fact that the sphere may not roll in a straight line down the slope (along one circumference), but will roll sideways in response to the changing distribution of its weight as it gathers powder.

This is a very complex 3 dimensional process, with many parameters affecting the output. In this real case, output can be taken as thickness of the coating, and some number describing how evenly the coating is distributed. Inputs will include (and are probably not limited to) the characteristics of the particles and powder used (surface properties, size, density, shape, adhesion), the thickness of the powder bed, the friction between powder and particles, and between the powder itself, and the angle of slope of the bed, the initial velocity of the particles, the type of wetting agent etc.

The second stage of the model development is to describe mathematically what is happening in this approximated model.

To describe it mathematically, the output (i.e. what describes the behaviour), and what inputs are relevant should be decided. The changed diameter of the particle is chosen as the primary output, as this increases as the powder attaches, and therefore

reflects the effectiveness of the coating process. When there is no further increase in diameter, coating has ceased. The distance along the slope is chosen as the primary input, while some constants are included in the equation to account for material and other properties.

Based on the experimental data, the proposed mathematical model is:

$$Y = A (1 - e^{-K \cdot X})$$

in which Y is the increase in diameter/mass, X is the rolling distance, in this equation, A is the maximum increase in diameter/mass and K indicates the rate of increase.

It is a difficult task to describe such a system using mathematics, which is why the system must be approximated. A very simple mathematical model which describes a phenomenon similar to, but more simple than that which occurs in practice should be built.

It was proposed that:

- the particle be presumed to be a sphere
- the 3 dimensional nature of behaviour (i.e. rolling sideways down the plate) be ignored, and the problem be considered only as 2 dimensional (i.e. a sphere rolling in a straight line along the slope of the plate)
- that the powder picked up by the rolling sphere be represented not as discrete (individual) particles, but as a continuum.
- that the diminishing pick up of powder particles be represented as being a distance based continuous function (for example: it could be diminishing linearly or exponentially). This means that the continuum attaching to the rolling particle becomes thinner and thinner in some continuous way. From these basic approximations came the representation shown below:

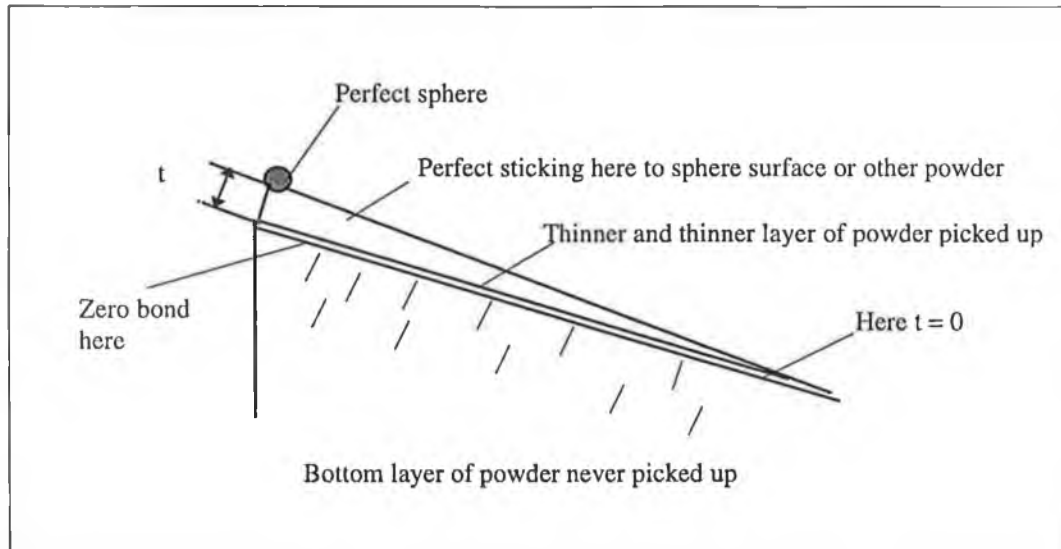


Figure 4.30. Model of Powder Coating

Obviously this is not the same as the actual problem, but it is a valid, useful first approximation to it.

The results as shown in the figures in this chapter demonstrate that the mathematical model for the powder coating process, i.e., $Y = A (1 - e^{-K \cdot X})$, agrees well with the data from the experiment work. Various media - particle, powder, and binder, etc., greatly influence the particle coating. Generally, the coating effectiveness is a function of many factors as expressed below:

$$\text{coating effectiveness} = \text{fn}(\text{powder, particle size, binder, height, ...})$$

of which the type of coating powder, particle size and binder are the most significant factors. For example, figures 4.15 and 4.16 show that using ground almond and icing sugar as the coating powder, the diameter and mass increases are much greater (up to 2.7 times) than those using flour as the coating powder; figures 4.18 to 4.23 show that using smaller particles, the mass increase is much less (up to 52%) than those using bigger particles; figures 4.26 and 4.27 show that using olive oil as the binder, the diameter increase is much greater (up to 53%) than those using water as the binder.

The values of the constants A and K are shown in Table 4.1 and Table 4.2 for different test conditions outlined.

Table 4.1 Constant A and K for diameter increase value

Test No.	Particle type	Coating material	Initial particle size (mm)	Binder material	Cycle No.	A (mm)	K
1a	Marrowfat peas	Flour	8.132	Water	1	805	0.05
1b	Marrowfat peas	Flour	8.132	Water	1	688	0.03
2a	Marrowfat peas	Flour	8.132	Water	1	630	0.05
					2	1100	0.03
					3	710	0.03
					4	550	0.04
					5	435	0.04
2b	Marrowfat peas	Ground almond	8.063	Water	1	1780	0.04
					2	2350	0.04
					3	2780	0.03
					4	1770	0.03
					5	1135	0.03
2c	Marrowfat peas	Icing sugar	8.168	Water	1	1355	0.05
					2	1295	0.02
					3	975	0.02
					4	2375	0.02
					5	1390	0.02
3a	Marrowfat peas	Flour	8.354	Water	1	630	0.05
3b	Moong beans	Flour	4.464	Water	1	1935	0.025
4a	Marrowfat peas	Almond	8.354	Water	1	1780	0.04
4b	Moong beans	Almond	4.464	Water	1	4005	0.025
5a	Marrowfat peas	Sugar	8.354	Water	1	1355	0.05
5b	Moong beans	Sugar	4.464	Water	1	3265	0.02
6	Marrowfat peas	Flour	8.354	Water	1	630	0.05
				Olive oil	1	740	0.05
				Lactose	1	605	0.05
7	Marrowfat peas	Almond	8.063	Water	1	1780	0.04
				Olive oil	1	2716	0.04
				Lactose	1	1825	0.04
8	Marrowfat peas	Sugar	7.950	Water	1	1355	0.05
				Olive oil	1	923	0.028
				Lactose	1	1376	0.05

Table 4.1 Constant A and K for Mass increase value

Test No.	Particle type	Coating material	Initial particle weight (g)	Binder material	Cycle No.	A (*0.01g)	K
1a	Marrowfat peas	Flour	0.39	Water	1	10	0.05
1b	Marrowfat peas	Flour	0.39	Water	1	7	0.04
2a	Marrowfat peas	Flour	0.39	Water	1	7	0.04
					2	11	0.05
					3	12	0.05
					4	12.5	0.05
					5	13	0.05
2b	Marrowfat peas	Ground almond	0.39	Water	1	13	0.04
					2	30	0.04
					3	44	0.04
					4	52	0.04
					5	67	0.04
2c	Marrowfat peas	Icing sugar	0.39	Water	1	9	0.045
					2	26	0.03
					3	26	0.02
					4	70	0.035
					5	63	0.018
3a	Marrowfat peas	Flour	0.39	Water	1	7	0.04
3b	Moong beans	Flour	0.07	Water	1	4.2	0.03
4a	Marrowfat peas	Almond	0.39	Water	1	13	0.04
4b	Moong beans	Almond	0.07	Water	1	6.3	0.023
5a	Marrowfat peas	Sugar	0.39	Water	1	9	0.045
5b	Moong beans	Sugar	0.07	Water	1	6.6	0.015
6	Marrowfat peas	Flour	0.39	Water	1	7	0.04
				Olive oil	1	8	0.04
				Lactose	1	6.5	0.035
7	Marrowfat peas	Almond	0.39	Water	1	13	0.04
				Olive oil	1	16	0.04
				Lactose	1	13.5	0.04
8	Marrowfat peas	Sugar	0.39	Water	1	9	0.045
				Olive oil	1	7.5	0.035
				Lactose	1	9.5	0.045

CHAPTER 5 CONCLUSIONS AND PROPOSAL FOR FURTHER WORK

5.1 CONCLUSIONS

- Diameter and mass are increased greatly with the rolling distance within a short coating distance. After this distance, the increased diameter and mass remain almost constant for each single cycle.
- Coating powder, particles and binder are the most significant factors affecting the particle coating.
- The coating process is also influenced by the system factors (equipment height)
- The mathematical model curve agrees well with the experimental data. The mathematical model for powder coating is expressed as: $Y = A * (1 - e^{-K * X})$, in which X is the rolling distance, Y is the increase in diameter, A is the max. increase in diameter or mass and K indicates the rate of increase.

5.2 PROPOSAL FOR FURTHER WORK

Due to the limited time and finance for this project, it is not realistic to carry out sophisticated experiments using a purpose built test rig discussed in Appendix A. Below are suggestions for future work.

- An investigation of how other material/binder combinations effect the model constants.
- A study to establish the specific material/binder parameters which influence the model constants, and their relative importance.

- To extend the investigation to metal powders and appropriate binders to coat ceramic particles according to the snow-ball principle.
- The investigation should be repeated with intermediate wetting of the particles when the coating effectiveness diminishes after a short rolling distance.

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APPENDIX A NEW PROCESS DESIGN

A.1 INTRODUCTION

The results presented in this thesis provided sufficient insight permitting design of possible processes for coating ceramic powder with metallic matrix material. In order to achieve the aim of a new/improved process for the coating of metal powders, the criteria for success must be established, and any candidate methods measured against these.

A.2 SPECIFICATION OF AIMS

A process with the potential to coat ceramic powders of diameter between 50 μm to 100 μm with metal/binder mixture to a diameter of about 500 μm (10 times), must meet the *basic requirements*: 1) to avoid clustering (good acceleration & separation of ceramic powders), 2) to avoid blanks (agglomeration only occur at powder/ceramic powder interface), 3) possible to coat partially coated particles, and 4) metal powder not to coat equipment itself.

It is *very desirable* for the process to be based on known working principles, to be versatile to cope with a range of particle sizes, and to have simple controls and a possibility to monitor the process.

An *ideal* process is an enclosed system with continuous production at high production rate but low capital cost (off the shelf equipment). The product should be round granules (rotating action). The process design must be compact, simple, easy to clean (to facilitate changeover of powder type) and easy to maintain.

A.3 POSSIBLE DESIGN SOLUTIONS

In the following sections, there are three kinds of candidate design solution, including two kinds of spray drying method and one kind of layering agglomeration method are discussed.

A.3.1 Ceramic Particles Coating Process

The powder

Powders have relatively large specific surface area, or surface area per given mass of material, usually measured in square meters per gram, or m^2/gm . The main advantage of a high surface area is that, when the powder is fired, it will easily sinter and become a single, large solid body. The main disadvantage of the large specific surface area is the tendency to agglomerate, or form uncontrolled lumps and clusters. Usually the higher the surface area, the greater is the tendency to be agglomerated,, other factors being equal. The proposed systems are proposed for coating ceramic particles of 50 μm to 100 μm diameter with metallic powder of size 10 μm to 20 μm diameter.

The powder is mixed with a liquid to make a flowable or fairly fluid system which can be easily shaped. In this process, the ceramic or diamond particles are mixed with alcohol, and the metallic or Cobalt powder is mixed with PEG. For the high specific surface of ceramic or diamond, powder tends to agglomerate and it is difficult to be dispersed.

Green body

The green body of the product is obtained by compacting spray dried ceramic or diamond particles coated with metallic or cobalt powder. The PEG as a binder will finally be removed during the sintering stage.

Products

The final coated product is obtained by sintering process. The higher temperature usually above 700 °C, first removes any organic additives like PEG, and then it causes sintering. Metal or Cobalt coated diamond or ceramic powder is produced. If the coating thickness is not sufficient, repeat process may be needed.

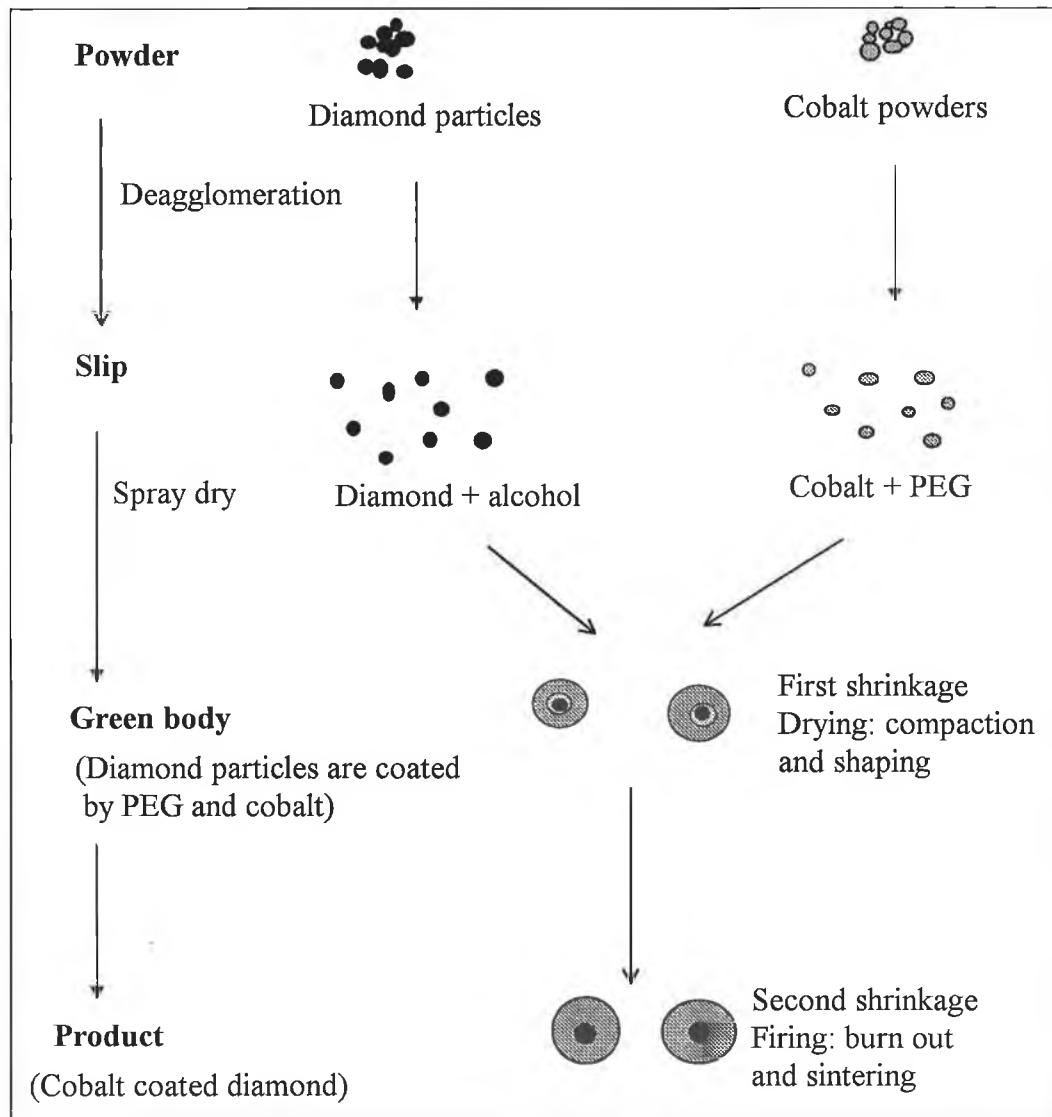


Figure A.1. Schematic Diagram of Cobalt Coating Diamond Process.

The main design objective is to develop a spray dryer system, to encapsulate diamond particles singly. The ideal thickness of the coat is about ten times the size of the particle itself. This can not be achieved at one stage, so two or three stages may be needed. The following are some ideas to develop the DIAMOND COATER DC-1 process.

A.3.2 Spray Drying with Pre-mixed Slurry of Metal Powder and PEG

The first alternative process is to pass the diamond particles over a vibrating table. Figure 2.

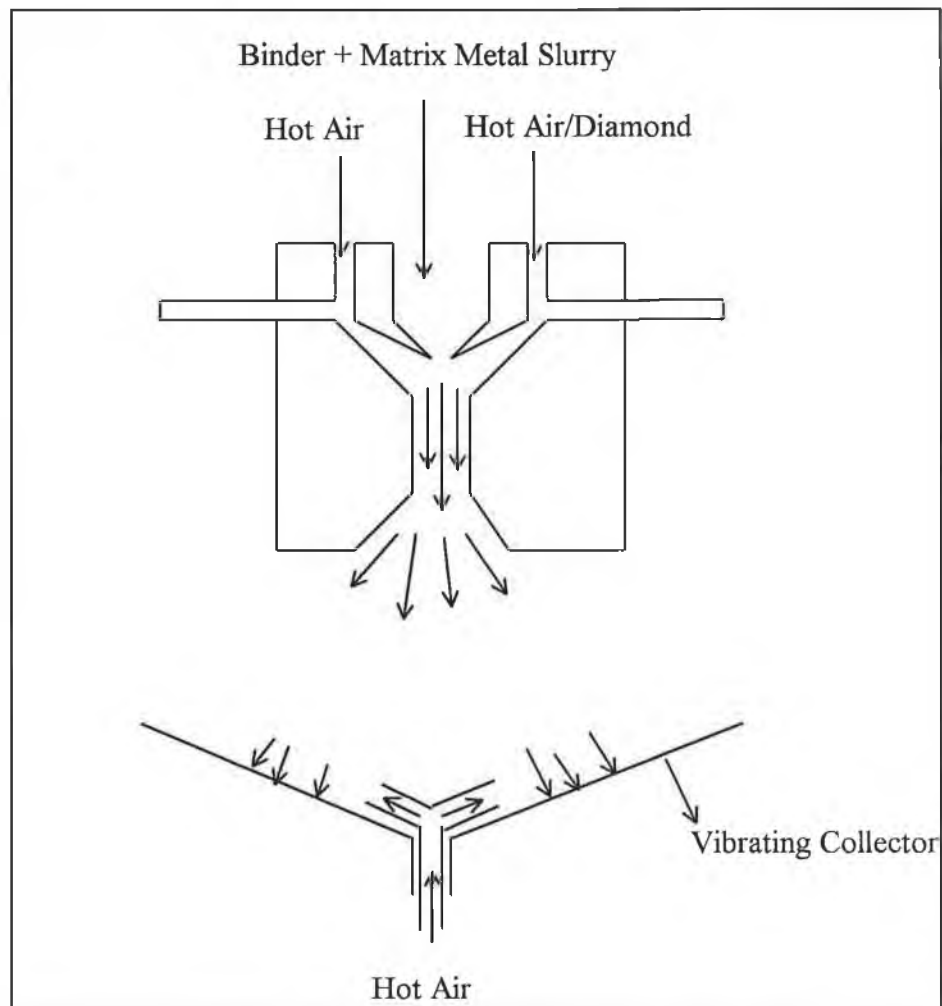


Figure A.2. The First Candidate Design Solution

The particles could be sprayed while rolling down the table, and re-circulated through a drying step. The advantages would be that there would be a large area for spraying-thus enabling high production rates. The particles accelerate and thus separate

as they are vibrated down the table - thus making clustering of the diamonds unlikely, and enabling smaller particle sizes to be coated. In the process, the incline angle and vibration intensity of the vibrating table can be adjusted as is already quite common in the diamond industry.

A.3.3 Spray Drying without Pre-mixed Slurry of Metal Powder and PEG

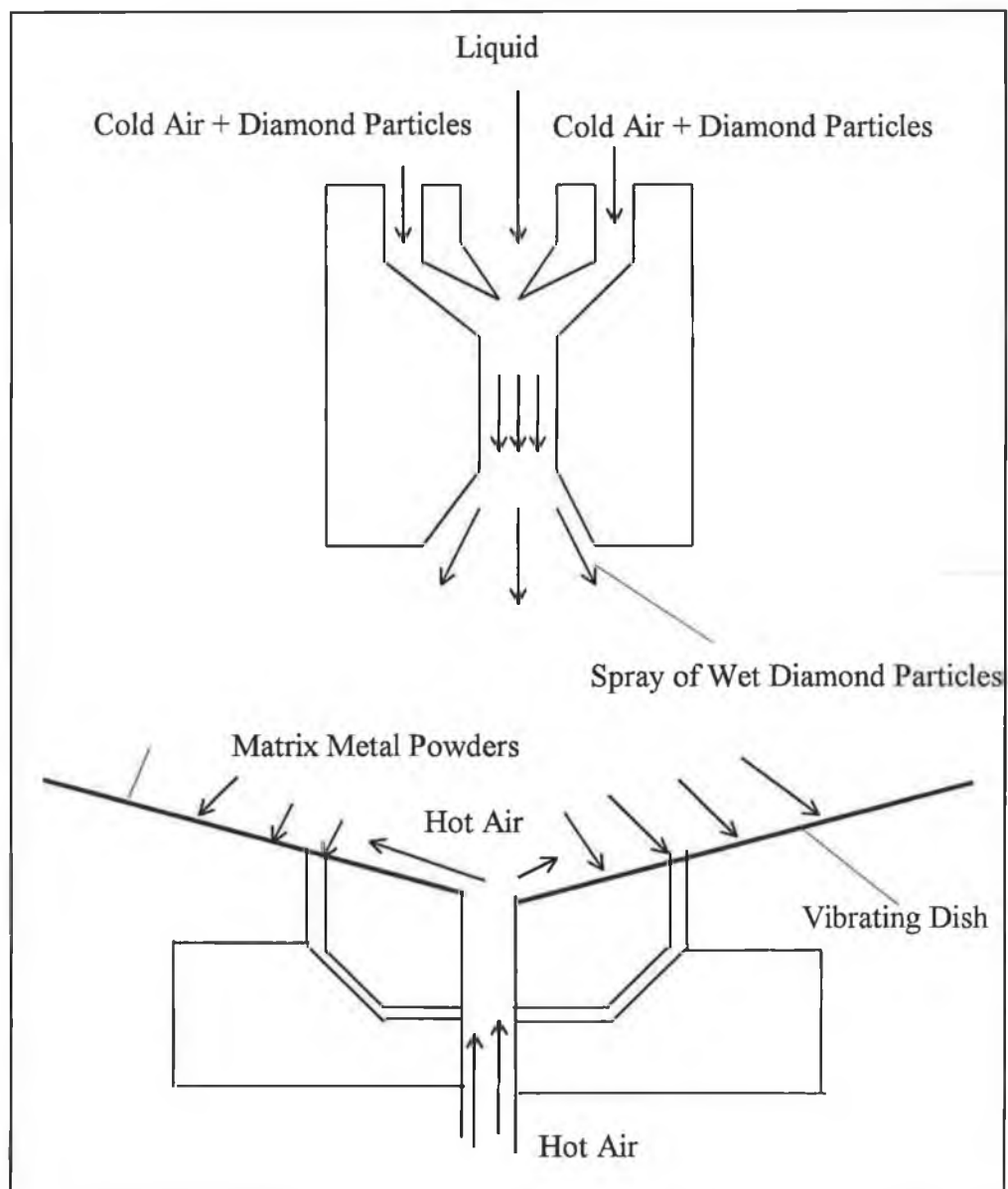


Figure A.3. The Second Candidate Design Solution

In this process it is proposed that wet diamond particles will be sprayed on to a vibrating table containing a dry mixture of binder and metallic powders. The shape of the vibrating plate will be so designed so that each wet particle will get coated as it progressively moves along the surface of the plate towards the hole in the centre of plate. (Figure 3)

A.3.4 Layering Agglomeration Method

A third alternative process might be the tumbling laying agglomeration process. It may need multiple steps to achieve the required thickness of the coating layer.

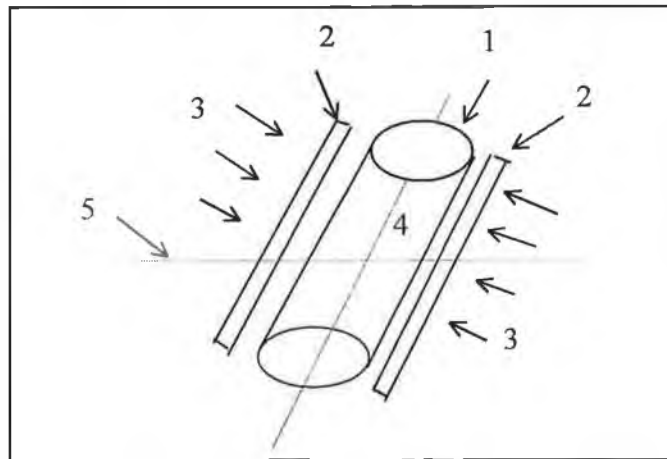


Figure A.4. Representation of the Agglomeration Apparatus

1, tilted cylinder; 2, heating elements; 3, cooling medium; 4, load; 5, rotation axis

The binder-coated diamond particles and fine metal powder are loaded in a brass cylinder internally coated with Teflon and tilted at an angle to the horizontal axis of rotation as shown in Figure 4. The tumbling ensures a good mixing and a uniform

movement of the diamond particles and of the coating powder. Upon heating the cylinder, the binder reaches a viscous state which allows the formation of a fine powder coating on the diamond cores (Figure 5.). in a subsequent step, heat treatment can be carried out to remove the binder and to sinter the powder layer which is then coherently bonded to the diamond core.

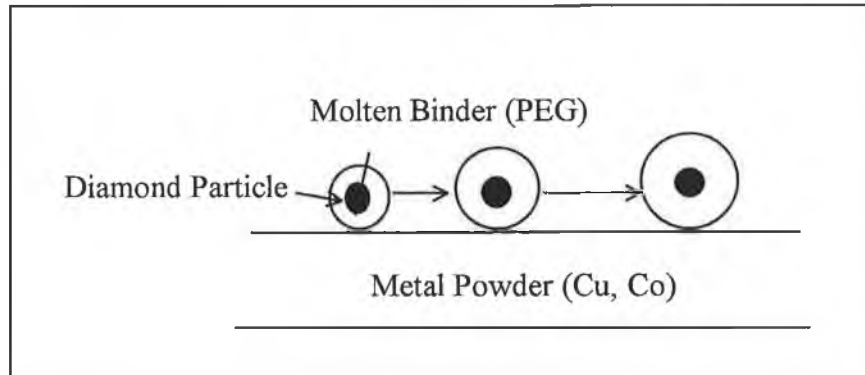


Figure A.5 Mechanism of Diamond Agglutination Coating.

The first step consists in covering diamond cores with the binder by introducing both cores and the binder into the cylindrical container. The container is then rotated and heated until the binder (PEG 3350 melting range is between 54-58°C) becomes viscous. After a period of about 15 minutes, the binder coating operation is completed and the cylinder is allowed to cool to room temperature. The resulting binder-coated diamond particles are slightly sieved to separate those that have stuck together.

The second step consists in agglutinating metal powder to these binder-coated cores. Binder-coated diamond, the fine metal powder and a lubricant are put into the cylinder. The cylinder is then simultaneously heated and rotated leading to the agglutination process itself. After cooling, the metal-coated diamond particles are separated from minute residue by sieving. Metal-coated diamond particles are then dewaxed.

A.4 ASSESSMENT OF PROPOSED SOLUTIONS

The discussion of the three kinds of candidate process and the technologies involved are given below.

A.4.1 Spray Drying with Pre-mixed Slurry of Metal Powder and PEG

Avoid Clustering

- 1) Diamond particles are mixed with Alcohol in proportion to form slip. The slip can be conveyed by hot air in dilute phase (suspension flow: $v = 13-16$ m/s). This kind of flow can avoid clustering of diamond particles.
- 2) Some kind of dispersion (CTAB) can be added in the slip of diamond and Alcohol, to make a good disaggregation of the diamond particles in the system. After this, the slip can be conveyed by hot air in dilute phase.
- 3) The tube diameter of hot air should be about 15 mm.
- 4) A relatively long tube for conveying diamond slip can be used.
- 5) When the coated diamond particles are rolling down the vibrating table, the particles are accelerated and thus separated.

Avoid Blanks

This can be achieved by evaporating out all the Alcohol in the diamond slip when they are conveyed in the tube of hot air. The bare diamond particles have high surface energy, when they meet with PEG coated metal powders, agglomeration occurs at the diamond surface. To achieve non blank is very difficult.

Possible to coat partially coated particles

This can be achieved by repeating the spray step. The coated diamond particles are collected by a chamber under the spray-dry tower, and are again reintroduced to the spray drying tower.

Prevention of the Metal powder coating the equipment itself

Coating of the equipment is usually a serious problem in spray drying. This can be avoided by a suitable spray nozzle (angle of spray) and a large tower diameter.

Versatility to cope with range of particle sizes

This can be achieved by repeating the spray step.

Simple Controls & Possibility of Monitoring the Process

This can be achieved by using thermocouples at the inlet and the outlet of the drying air, and by using pressure detector in the middle of the tower. The feed flow rate of PEG coated metal powder slip to the nozzle can be monitored and adjusted by the rotating speed of the feed pump.

Continuous Production, High production Rate, Enclosed System and Round Granules.

These can be achieved in the spray drying process and are the advantages of this kind of process.

Capital Cost

The capital cost may be high. Spray-drying nozzle, vibrating table, thermocouple, pressure detector, air pump, air heater, feed pump (can be substituted by air pump), mixer (man can do it), air supply fan, air exhaust fan, stainless steel sheet (can be substituted by iron plate) about 2 mm thick may be needed (for the body of the tower), cyclone, sieve for sizing the products, many valves. Because the production rate is very high, the capital cost may be recouped over a short period of time.

Cleaning of Facility

Because it is an enclosed system, it is very difficult to clean and maintain. It can be improved by using one open part at the tower. When the lower/upper part of the tower is opened, the tower body and vibrating table can be cleaned.

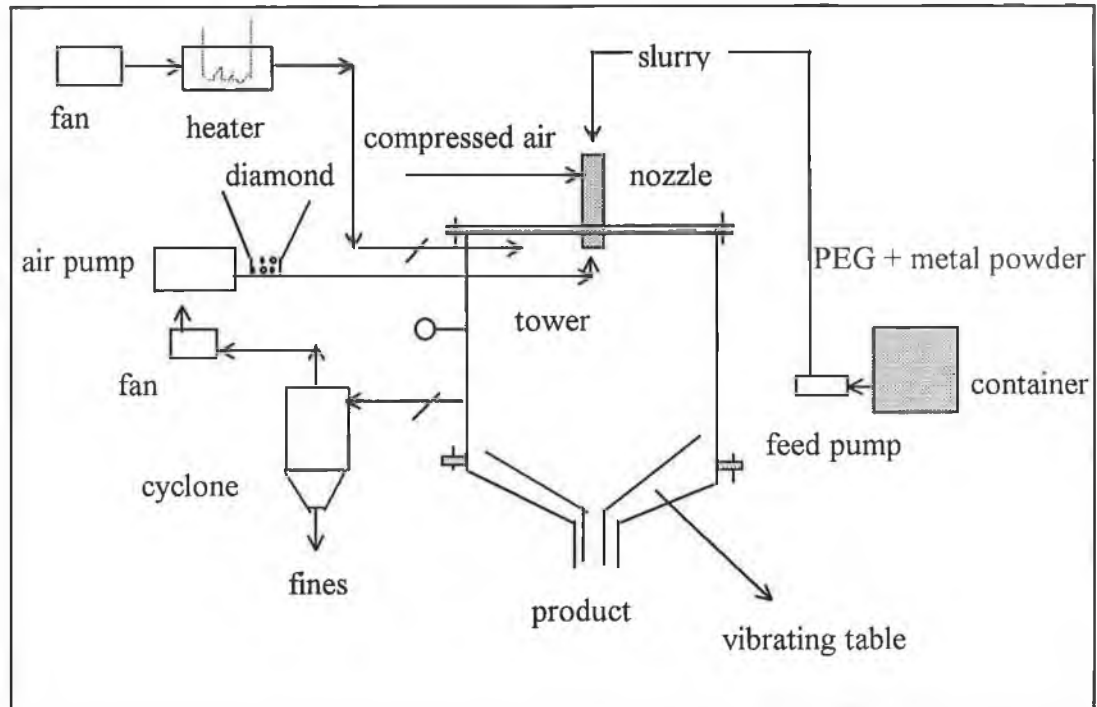
Compact Design

Figure A.6. Spray Dry and Vibrating Table for Coating Diamond Particles
(/ : Thermocouple; O— : Pressure Detector)

A.4.2 Spray Drying without Pre-mixed Slurry of Metal Powder and PEG

Avoid Blanks

Wet diamond particles will be sprayed on to a vibrating table containing a dry mixture of binder and metallic powders, all diamond particles will be coated by PEG and metal powder. Blanks can be avoided better than the first candidate design solution.

Round Granules

The round granule shape will not be better than the first candidate design solution.

Other factors may be equal with the first candidate design solution.

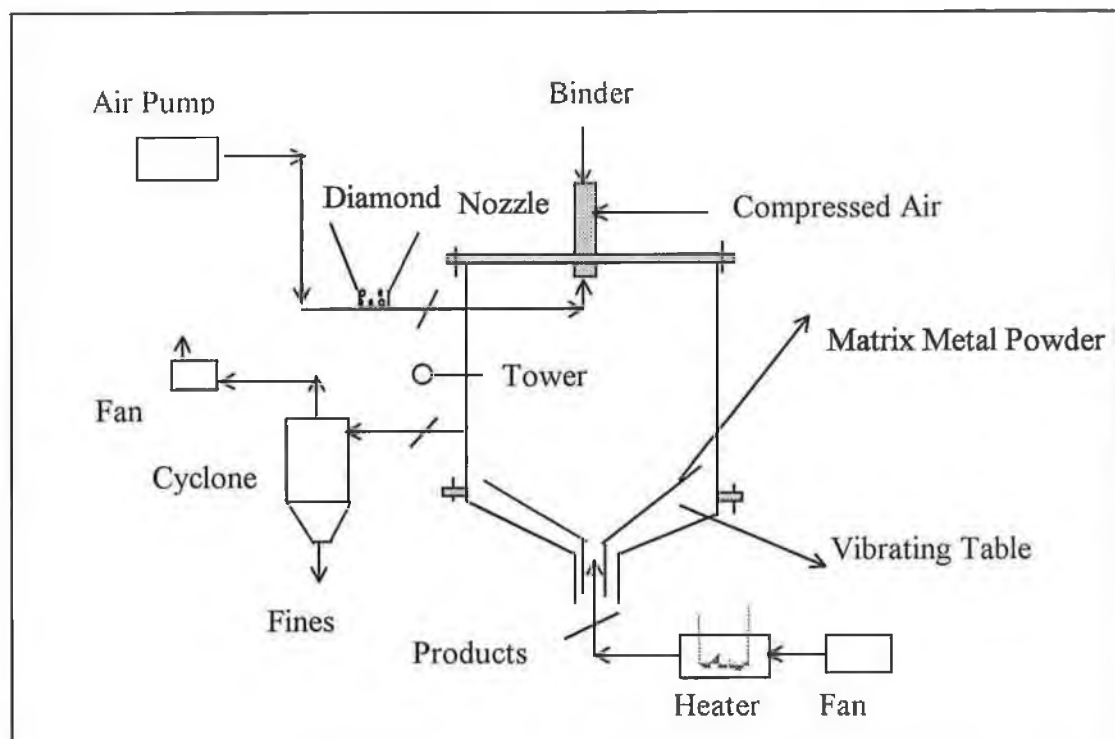
Compact Design

Figure A.7. Spray Dry and Vibrating Table for Coating Diamond Particle
(/: Thermocouple; ○: Pressure Detector)

A.4.3 Layering Agglomeration Method

Avoid Clustering

Because the amount of metal powder that ought to be agglutinated around diamond particles is high, a large amount of binder must be deposited on the surface of cores. This step consisting of covering diamond particles with the binder is of prime importance. Heating up to a maximum temperature range of 51-51.5°C (the softening temperature of PEG) for about 15 minutes is the most appropriate cycle to coat diamond particles with a fairly large amount of binder and to avoid sticking of particles together.

Possible to coat partially coated particles

By repeating the same fabrication steps involving mixtures of diamond particles with metal powder, a larger coating thickness can be achieved. It also can be coated with different metal powder.

Avoid Blanks and Metal Powder not to coat equipment itself

It is found that 95% of the core particles are coated by layering agglomeration method [22]. Because the cylinder is made of brass with internally coated Teflon and tilted at an angle to the horizon, the problem of coating equipment is not serious.

Versatility to cope with range of particle sizes

This can be achieved by repeating the same fabrication steps.

Simple Controls & Possibility to Monitor Process

This can be achieved by using thermocouple and a glass mirror on the rotating container.

Continuous Production and High production

The main disadvantage of this kind of process is that it can not provide continuous production. The two steps are separate. So the production rate is very low.

Enclosed System

This can be achieved by layering agglomeration method.

Low Capital cost

The capital cost of this kind of process is very low. A container, a electric motor, an axle of metal, a shelf of metal, a thermal couple, a glass mirror, few pieces of wheel, a sieve are needed. The low production rate makes the capital cost higher.

Round Granules

Though the metal powder was almost coarse, a thick and uniform metal powder coating may be obtained by using the agglutination process.

Easy to Clean and Simple Design

This can be achieved by layering agglomeration method.

Compact Design

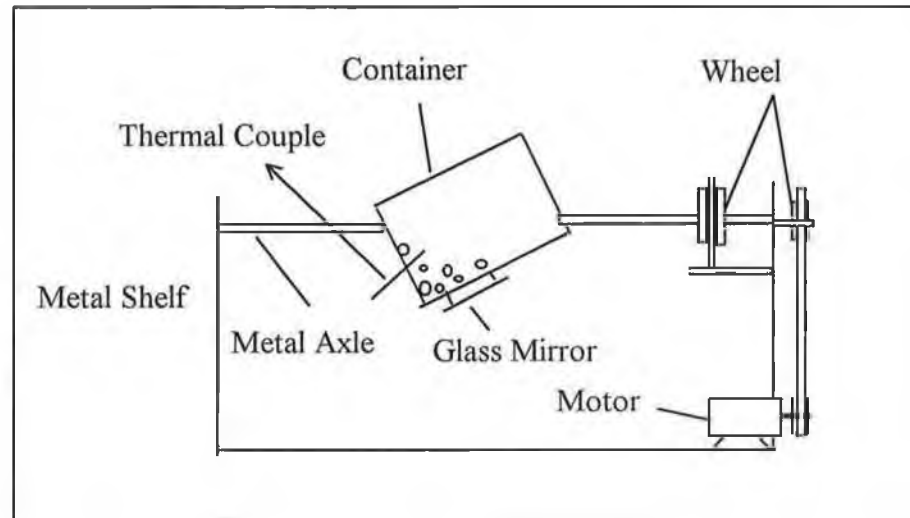


Figure A.8. Tumbling Agglutination Coating Apparatus

Conclusion

This kind of coating method is flexible and simple. To achieve a uniform and dense powder coating it only requires fine powders whose particle size depends on the size of particles to be coated.

APPENDIX B EXPERIMENTAL DATA FOR GROUND ALMONDS + WATER + MARROWFAT PEAS

B1

Ground Almonds + Water + Marrowfat Peas

(Results for cycle 1)

No.	x mm	mm	mm	mm	mm	g	mm	mm	mm	mm	g	mm
1	25	d01a	d01b	d01c	d01	weight d01	d11a	d11b	d11c	d11	weight d11	d1
		5.620	8.615	10.123	8.119	0.39	8.010	9.710	10.123	9.281	0.48	1.161
2	25	d02a	d02b	d02c	d02	weight d02	d12a	d12b	d12c	d12	weight d12	d2
		6.595	7.660	9.763	8.006	0.39	7.850	9.215	9.763	8.942	0.48	0.936
3	50	d03a	d03b	d03c	d03	weight d03	d21a	d21b	d21c	d21	weight d31	d3
		5.620	8.615	10.123	8.119	0.39	9.320	10.925	8.45	9.565	0.49	1.445
4	50	d04a	d04b	d04c	d04	weight d04	d22a	d22b	d22c	d22	weight d22	d4
		6.595	7.660	9.763	8.006	0.39	7.856	9.313	9.864	9.011	0.50	1.005
5	75	d05a	d05b	d05c	d05	weight d05	d31a	d31b	d31c	d31	weight d31	d5
		5.620	8.615	10.123	8.119	0.39	8.100	10.490	11.100	9.896	0.51	1.777
6	75	d06a	d06b	d06c	d06	weight d06	d32a	d32b	d32c	d32	weight d32	d6
		6.595	7.660	9.763	8.006	0.39	10.508	9.864	8.950	9.774	0.52	1.768

Equipment height h: 300 mm. Drying temperature (room): 21 °C. Drying time: 10 min.

APPENDIX B

Ground Almonds + Water + Marrowfat Peas

(Results for cycle 1)

No.	x mm	mm	mm	mm	mm	g	mm	mm	mm	mm	g	mm
7	100	d07a	d07b	d07c	d07	weight d07	d41a	d41b	d41c	d41	weight d41	d7
		5.620	8.615	10.123	8.119	0.39	8.108	10.492	11.101	9.900	0.52	1.781
8	100	d08a	d08b	d08c	d08	weight d08	d42a	d42b	d42c	d42	weight d42	d8
		6.595	7.660	9.763	8.0060	0.39	10.506	9.867	8.953	9.775	0.52	1.769
9	150	d09a	d09b	d09c	d09	weight d09	d51a	d51b	d51c	d51	weight d51	d9
		5.620	8.615	10.123	8.119	0.39	8.112	10.493	11.103	9.902	0.52	1.783
10	150	d010a	d010b	d010c	d010	weight d010	d52a	d52b	d52c	d52	weight d52	d10
		6.595	7.660	9.763	8.006	0.39	10.509	9.868	8.954	9.777	0.52	1.771
11	200	d011a	d011b	d011c	d011	weight d011	d61a	d61b	d61c	d61	weight d61	d11i
		5.620	8.615	10.123	8.119	0.39	8.116	10.496	11.108	9.906	0.52	1.787
12	200	d012a	d012b	d012c	d012	weight d012	d62a	d62b	d62c	d62	weight d62	d12i
		6.595	7.660	9.763	8.006	0.39	10.500	9.870	8.960	9.776	0.52	1.770

Equipment height h: 300 mm. Drying temperature (room): 21 °C. Drying time: 10 min.

APPENDIX B

B2

Ground Almonds + Water + Marrowfat Peas

(Results for cycle 2)

No.	x mm	mm	mm	mm	mm	g	mm	mm	mm	mm	g	mm
1	25	d01a	d01b	d01c	d01	weight d01	d11a	d11b	d11c	d11	weight d11	d1
		10.900	10.900	8.270	10.023	0.52	11.090	11.850	11.685	11.541	0.68	1.518
2	25	d02a	d02b	d02c	d02	weight d02	d12a	d12b	d12c	d12	weight d12	d2
		10.723	9.505	9.620	9.949	0.52	10.986	11.672	11.704	11.454	0.66	1.504
3	50	d03a	d03b	d03c	d03	weight d03	d21a	d21b	d21c	d21	weight d31	d3
		10.724	9.628	9.621	9.991	0.52	11.250	12.850	11.050	11.716	0.73	1.725
4	50	d04a	d04b	d04c	d04	weight d04	d22a	d22b	d22c	d22	weight d22	d4
		10.700	9.512	9.546	9.919	0.52	12.170	11.250	11.200	11.540	0.74	1.620
5	75	d05a	d05b	d05c	d05	weight d05	d31a	d31b	d31c	d31	weight d31	d5
		9.634	10.718	9.633	9.995	0.52	13.550	12.506	11.104	12.386	0.82	2.391
6	75	d06a	d06b	d06c	d06	weight d06	d32a	d32b	d32c	d32	weight d32	d6
		9.443	10.806	9.549	9.932	0.52	13.876	11.853	11.063	12.264	0.80	2.331

Equipment height h: 300 mm. Drying temperature (room): 21 °C. Drying time: 10 min.

APPENDIX B

Ground Almonds + Water + Marrowfat Peas

(Results for cycle 2)

No.	x mm	mm	mm	mm	mm	g	mm	mm	mm	mm	g	mm
7	100	d07a	d07b	d07c	d07	weight d07	d41a	d41b	d41c	d41	weight d41	d7
		10.732	9.648	9.630	10.003	0.52	13.993	12.068	11.045	12.368	0.81	2.365
8	100	d08a	d08b	d08c	d08	weight d08	d42a	d42b	d42c	d42	weight d42	d8
		9.340	9.620	10.850	9.936	0.52	13.878	11.856	11.064	12.266	0.80	2.329
9	150	d09a	d09b	d09c	d09	weight d09	d51a	d51b	d51c	d51	weight d51	d9
		9.620	10.830	9.523	9.991	0.52	13.972	11.854	11.232	12.352	0.80	2.361
10	150	d010a	d010b	d010c	d010	weight d010	d52a	d52b	d52c	d52	weight d52	d10
		9.620	9.338	10.842	9.933	0.52	13.994	11.764	11.039	12.265	0.81	2.332
11	200	d011a	d011b	d011c	d011	weight d011	d61a	d61b	d61c	d61	weight d61	d11i
		10.893	8.273	10.997	10.054	0.52	13.986	11.698	11.524	12.402	0.82	2.348
12	200	d012a	d012b	d012c	d012	weight d012	d62a	d62b	d62c	d62	weight d62	d12i
		10.806	9.339	9.621	9.922	0.52	13.882	11.862	11.069	12.271	0.80	2.349

Equipment height h. 300 mm. Drying temperature (room): 21 °C. Drying time: 10 min.