Factors affecting the Coffee-Stability of Instant Whole Milk Powder

Thesis presented by

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Declaration

I hereby certify that the material, which I now submit for the assessment on the programme of study leading to the award of Master of Science 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 test of my work

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2. LIST OF ABBREVIATIONS

Abbreviation	Explanation
ADMI	American Dry Milk Institute
CCP	Colloidal Calcium Phosphate
ESCA	Electron Spectroscopy for Chemical Analysis
HS	Heat Stability
MTL	Moorepark Technology Ltd., Fermoy, Co. Cork
NEM	N-Ethyl-Maleimide
SDS	Sodium Dodecyl Sulphate
SEM	Scanning Electron Micrograph
SMP	Skim Milk Powder
SMP	Skim Milk Powder
TS	Total Solids
WMP	Whole Milk Powder
WPNI	Whey Protein Nitrogen Index

3. ABSTRACT

FACTORS AFFECTING THE COFFEE-STABILITY OF INSTANT WHOLE MILK POWDER

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There are growing markets in developing countries, for instant WMP with the additional specification of being stable in coffee. Coffee is considered a harsh environment for reconstitution of milk powders owing to high temperatures (~80°C) and low pH (~4.8) which may cause protein precipitation or feathering. The objective of this study was to explore a range of parameters in order to identify some key factors that contribute to instability. Initially, the physico-chemical conditions in the coffee test were scrutinised. Later, emphasis was shifted to the effects of individual process steps during pilot plant manufacture of instant WMP. Coffee-stable commercial samples were more resistant to feathering and sedimentation under conditions of low pH and high temperatures. A model test was developed to simulate the acidic effect of coffee, and to examine the sensitivity of WMP over a wider pH range. The resulting sedimentation/pH profiles were distinguishable for coffee-stable and coffee-unstable samples. Furthermore, the model test proved to be more exacting than the coffee itself. Subsequent investigations confirmed that monovalent cations such as K^+ counteract the negative effects of low pH. Water hardness and powder characteristics such as bulk density, particle size and interstitial air volume influenced coffee-stability. The low sediment volumes that resulted from prior reconstitution before addition to coffee; increased mechanical activity and addition of surface active agents suggest that the observed instability may arise from the physical behaviour of the powder itself. In an investigation of the process parameters affecting coffee-stability, preheating and agglomeration conditions were critical to product physico-chemical characteristics and functionality. Fat composition of the milk influenced coffee stability more so than protein. Storage resulted in a disimprovement in coffee-stability. An alternative production method by recombination of cream into skim milk concentrate was comparable with the conventional process in terms of powder characteristics and coffee-stability as was also the case for butter-oil fat-filled powders.

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I WISH TO DEDICATE THIS THESIS TO MY PARENTS,
EAMONN AND MARY TEEHAN.

THERE IS VICTORY TO BE GAINED IN EVERY GALLANT STRUGGLE
THAT IS MADE.

5. Introduction

The aim of dairy research in many instances is to find new and profitable ways to utilise milk in its different forms and lessen the dependence on the production of commodity dairy products, such as skim-milk powder and butter. On a global scale, whole milk powder production is increasing, particularly in Australia where increases of ~6% were recorded in 1995 (IDF, 1996). In the European Union, where milk production is restricted by quotas, WMP production decreased by 4.7% in 1995 (IDF, 1996). During 1995 in Ireland the production of whole milk powder achieved a level of 34,100 tonnes compared to 36,000 in the previous year representing a decline of 5.3% in output. However, in China where up to 90% of milk is processed into milk powder, there is potential for developing markets for WMP (IDF, 1996). WMP is used for nutritional and functional purposes and has applications in products such as sauces, chocolate, bakery and beverage products. It has an advantage over skim milk powder (SMP) in that it does not require recombination with fats or oils in order to prepare a milk substitute and so only requires packaging for the consumer market. Much of the WMP produced is marketed in Third World and developing countries where fresh milk is in limited supply. Dried milk also has advantages over fresh milk in these situations in that it can be stored for long periods while still retaining its high quality and can be transported easily at a lower cost. Many such countries also require that WMP meet the additional specification of coffee-stability.

The manufacture of instant whole milk powder was not successful until the 1970's and since then, the technology for production of WMP has evolved and undergone refinements. The essential elements such as spray atomisation of concentrated milk in the form of droplets into a hot air-stream in a drying chamber are still fundamental to the technology being used. However evolution has focused on the sub-division of drying into 2-stages, 3-stages (also known as multi-stage) and development of the straight-through method for agglomeration to produce a superior quality product at a low energy consumption.

For WMP to be useful as a coffee-whitener, the dried product must have instant characteristics. It must also be capable of storage for at least 6 months and preferably longer without deterioration in performance or flavour. The generally accepted process for producing instant WMP today is based on the straight-through process, employing either 2 or 3-stage drying with fines re-circulation to aid the formation of agglomerates, followed by lecithination to improve wettability.

This project aims initially to investigate the factors which contribute to poor coffee stability of instant WMP. This was implemented by scrutinising the coffee sediment test under various conditions. The second aim of the project was to determine the process parameters which affect the coffee stability of instant WMP in order to improve the product for this application.

6. Literature Review

6.1 Coffee Stability

Aqueous coffee solutions create adverse conditions for the reconstitution of a whole milk powder (WMP) because of low acidic pH (4.6-5.0) and high temperature (approximately 85°C). The acid content of coffee which is determined by measurement of the pH value, is influenced by the type of coffee beans, the method of their processing and the hardness of the water used in reconstituting the instant coffee (Geyer and Kessler, 1989).

Much of the literature deals with liquid and powdered coffee creamers, while very little research has been carried out on the use of milk powders as coffee whiteners. Sweetsur (1976) suggested that the instability of instant skim milk powder in hot coffee may be associated with rehydration of protein in the powder. It is possible that when skim milk powder is added to hot coffee at a low pH, possibly as low as the isoelectric point of casein, dispersion and rehydration of the protein must be rapid to allow dilution to take place before coagulation occurs. A strong positive correlation ($r = +0.81$) was found between the amount of "coffee" precipitate and the casein number (% of the total nitrogen that is precipitated at pH 4.6 by the addition of acetic acid and sodium acetate, which includes casein nitrogen and denatured whey protein) (Sweetsur, 1976). This suggested that the unstable powders had been subjected to a severe heat treatment at some stage of the process with resultant denaturation of much of the whey proteins, which were then able to complex with the casein. Sweetsur and White (1975) suggested that the greater the complex formation between β-lactoglobulin and casein in heated milk, the greater will be the extent of the heat stability curve minimum (the pH at which the milk is the least heat stable. c.f. Figure 6.6). Consequently there will be a reduction in the coagulation time maximum. Negative correlations obtained between the amount of coffee precipitate and maximum and minimum coagulation times ($r = -0.69$ and -0.48 respectively) suggests that the more unstable the powder to hot coffee, the greater the amount of whey protein complexed with casein (Sweetsur, 1976).

6.2 Coffee Whiteners

WMP is normally manufactured to a specified fat content of 26% or 28%. Fat-filled milk powders are based on the replacement of milk fat with less expensive sources such as vegetable, animal or marine oils. This type of product resembles WMP in most physico-chemical characteristics, save whatever changes may be brought about by the process of recombining and emulsification of the replacement fats into a skim milk base. Imitation coffee creamers bear little resemblance to milk and milk powders except where a milk protein such as sodium caseinate is used as the main fat emulsifier. The fat content of such products is 30-35 %, with the remainder composing of sugars such as dextrose or corn syrup, emulsifiers and stabilising agents.

If instant WMP is to be used as a coffee whitener, it must be competitive with commercial coffee whiteners as regards price, nutrition and functionality. It should also meet the same functional specifications as coffee whiteners: exhibit good flow properties, avoid clumping and caking, disperse easily in hot liquids such as coffee and be non-hygroscopic. It must be packaged properly for convenient use and if necessary to protect against moisture build-up. (Griffin and Lynch, 1968).

The requirements of a dried coffee whitener when used in coffee at extremes of pH 4.8-6.3 and temperature (50-90°C) have been outlined (Ortman, 1960, Giddey, 1967). A coffee whitener must dissolve rapidly with no insoluble particles evident after brief stirring. There should be no coagulation of the whitener or separation from the solution. When instant WMP is used as a coffee whitener, performance is based on the solubility of the powder in the coffee solution.

Coffee whiteners are used as a substitute for cream, evaporated milk or fresh milk in coffee, tea and cocoa products. Spray dried imitation coffee whiteners have been readily accepted by consumers due to lower cost, convenience, ease of handling, improved shelf life without refrigeration and preference of some consumers for vegetable fat products. Also, they are used as bodying agents and flavour enhancers in soups, sauces and puddings. Greutzmacher and Bradley (1991) reported a formulation for a spray-dried coffee whitener as follows:

Table 6-1. Formulation for a coffee whitener (Greutzmacher and Bradley, 1991).

Ingredient	Levels (% dry basis)
Partially hydrogenated vegetable oil	34.0
Corn syrup solids (42DE)	55.4
Sodium Caseinate	3.5
Di potassium phosphate	2.1
Sodium silicoaluminate	0.5
Mono and di-glycerides	1.3
Sodium stearoyl lactylate	0.1
Flavours and Colours	0.1
Moisture	3.0

In addition to the requirements already mentioned, coffee whiteners must maintain emulsion stability either as liquid or as a spray-dried powder when it is reconstituted in hot coffee solutions. Sodium caseinate is used in coffee whiteners for its good emulsifying properties, although it is regarded as expensive. Greutzmacher and Bradley (1991) recently used demineralised acid whey protein derived from cottage cheese whey, as an acceptable replacement for sodium caseinate in spray-dried coffee whiteners.

Thompson and Reniers (1982) used freeze-dried succinylated whey concentrate prepared from precipitates obtained by heat coagulation of cottage cheese whey, instead of sodium caseinate in a coffee-whitener formulation. No feathering or significant flavour differences were noted upon the addition of modified whiteners to coffee.

Reformed casein micelles have been developed and used as a complete or partial replacement for fat in a variety of food products and particularly in coffee whiteners (Melachouris *et al.,* 1992). It is claimed that such coffee whiteners have functional and organoleptic properties equivalent to conventional coffee whiteners.

6.3 Coffee creamers

Much of the scientific literature refers to liquid coffee-whiteners or coffee-creamers. This information while not directly related to instant WMP however, does provide valuable background information relating to coffee-stability.

A coffee-stable WMP should have the same properties as a liquid coffee-whitener namely, stability, limited viscosity effects, whitening ability and good flavour. The coffee-whitener must provide uniform whitening ability which is controlled by the total amount of solids present and the fineness of the dispersed phase. Because of the multitude of applications, the coffee-whitener should have a bland flavour and be odour free.

Coffee stability of coffee-cream mainly depends on fat content and viscosity of the coffee cream as well as on the acidity and temperature of the coffee solution. Visible flocculation (feathering) results from membrane surface layer protein-induced aggregation of the homogenised fat globules (Hoffman *et al.,* 1996).

Homogenisation causes proteins to adhere to the fat globules in the form of additional membrane material and enhance the opportunity for flocculation together with the fat globules in the hot coffee (Geyer and Kessler, 1989).

The whitening power of coffee cream is due to the attachment of both micellar casein and whey proteins to the fat globule membrane by homogenisation. However, whey proteins in their native state are more liable to flocculation in hot coffee than the casein fraction (Geyer and Kessler, 1989).

Salt-reduced creams were found to be less stable in coffee (Geyer and Kessler, 1989). Since the colloidal stability of milk is based on a dynamic equilibrium of milk salts and protein, a reduction in salts concentration is likely to cause disturbance. Heating causes a drop in the pH value of the cream and without the buffering effect of salts, the pH decreases rapidly towards the isoelectric point, resulting in precipitation.

For calcium-reduced creams, the stability to flocculation increases as the calcium ion concentration decreases. As the calcium ion concentration increases, the size of the casein micelles increases. According to Kirchmeier (1966), larger casein micelles are less heat stable and flocculate more easily.

In summary, the combination of fat, protein and salts in the milk in addition to processing steps, for example, preheating and homogenisation, influence the stability of coffee creamers. Therefore, it is reasonable to assume that some similar challenges arise during the application of instant WMP, as a coffee whitener.

6.4 Production of Instant Whole Milk Powder

It is difficult to find specific steps or process conditions in the literature relating to the production of instant WMP with coffee stable characteristics. Most information concerns the key steps in the manufacture of instant WMP: (a) preheating to enhance shelf-life and (b) agglomeration to give the powder instant properties. Instantization improves the rate and completeness of reconstitution of the powder without changing its solubility. The process was originally developed for milk powders by Peebles (1956), using rewet approaches. A modern two-step process (Westergaard, 1983) for the production of instant WMP is shown in Figure 6-1, for the production of the basic powder followed by lecithination and packing. The possibility of connecting these two process lines into one continuous line is also shown by a dotted line.

6.4.1 Standardisation of the milk composition

WMP is usually manufactured to either of two fat specifications: 26% or 28% fat in powder. Consequently, the first step is to standardise the milk. Because the fat content of the milk varies throughout the manufacturing season, it is necessary to standardise it with reference to its non-fat solids content (protein, lactose and ash, which may also vary in content). As the production of milk falls towards the end of the year, the concentration of lactose in the milk decreases, whereas fat and protein increases. Protein levels can reach quite high levels (up to 40% of the solids-non-fat content) resulting in problems during processing, for example, "burn-on" in evaporators and reduced powder solubility.

A proportion of the raw milk is separated into cream and skim milk before recombining either phase at the appropriate rate with the bulk milk to achieve the desired fat content. It is not normal practice to standardise the protein content, although it can be adjusted by addition of permeate (containing lactose and non-protein nitrogen) or retentate (containing concentrated protein) from ultrafiltered milk.

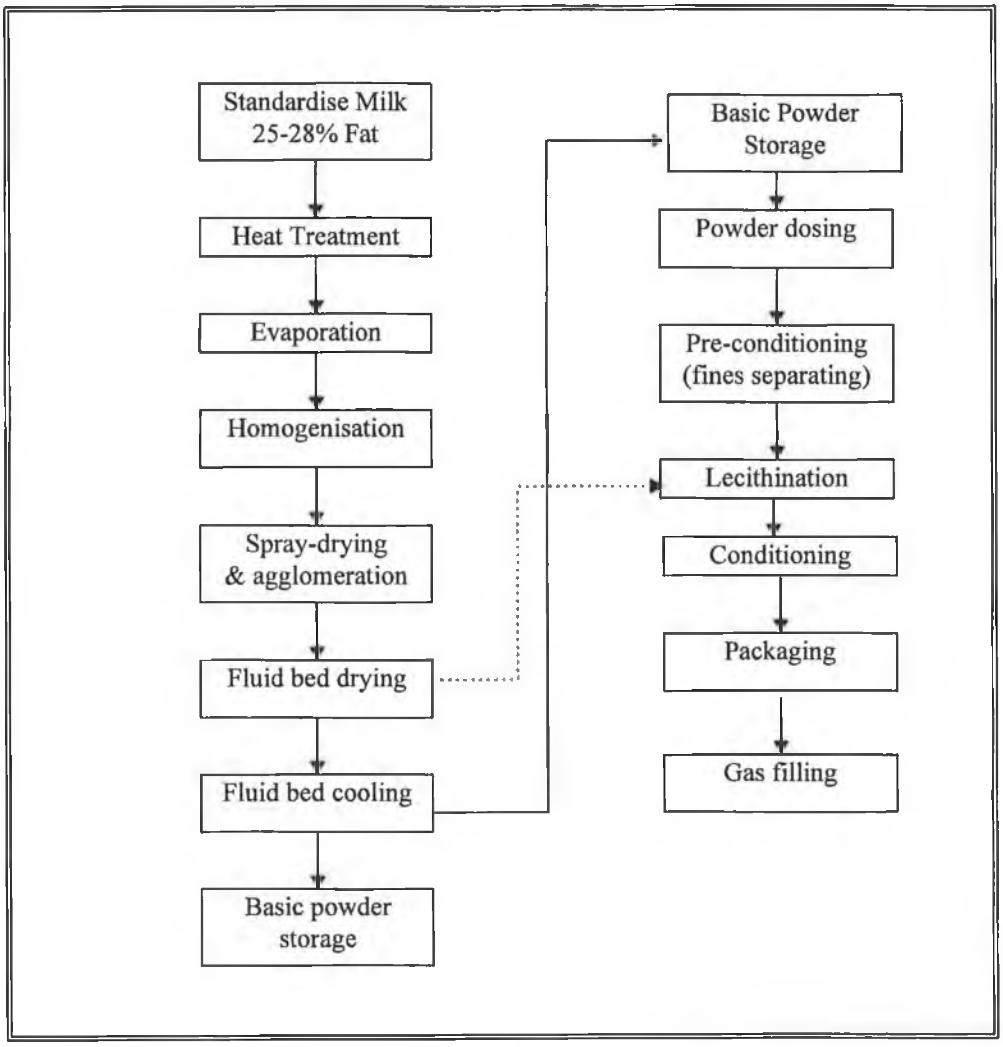


Figure 6-1. Processing scheme for the production of Instant WMP.

The heat stability (H.S) of protein-standardised 2.0 % milk does not change dramatically and has sensory qualities equivalent to that of normal milk (Peter *et al.*, 1996). Unlike fat, protein standardisation during manufacture of preserved milk products has not been recognised within the Code of Principles of the Codex Commission for Milk and Milk Products. However, recent revisions to the Codex standards for preserved milks are likely to be adopted in the near future.

6.4.2 Preheat Treatment

Many of the physical, chemical and functional properties of milk powders are determined mainly by the preheating conditions used. High preheat treatments, which retard the onset of oxidised aroma and flavour, produce under similar drying conditions WMP with a higher solubility index. The adverse effects on solubility index may be minimised by using a high preheat treatment temperature (125°C) with a moderate holding time (20s) (Baldwin and Ackland, 1991). WMP is generally classified as a medium-heat powder. Over the years a wide range of preheating temperatures have been applied, ranging from typical pasteurization (72°C for 15s); long holding-time heating known as the 'Hot Well' method (e.g. 85°C for up to 30 min) to high-temperature, short-time heating (e.g. 120°C for 2 min) using direct (steam injection) or indirect (plate heat exchanger) heating (Singh and Newstead, 1992).

6.4.3 Evaporation

Evaporation refers to the process of heating liquid to temperatures in excess of the boiling point to remove water in the form of vapour. Because milk is heat sensitive, heat damage can be minimized by evaporation under vacuum to reduce the boiling point. The basic components of this process consist of heat-exchanger, vacuum, vapour separator and condenser. Falling film evaporators are the most widely used in the dairy industry. Milk is concentrated to 44-50% total solids by evaporation, before spray-drying. Multiple stage evaporation (Figure 6-2) is usually employed to improve the energy efficiency, whereby the stages are connected in series and the vapour generated from the product in the first stage is used as a heating medium in the second. The second and subsequent effects must operate at a higher vacuum (and therefore a lower boiling temperature) than the previous. In addition, thermocompression increases the efficiency, by recompressing part of the vapour from the first vapour separator to heat the first calandria. High pressure steam connected to the thermocompressor increases the kinetic energy of the vapour from the separator, which is then used as a heating medium for the first stage of a multiple effect evaporator (Westergaard, 1983).

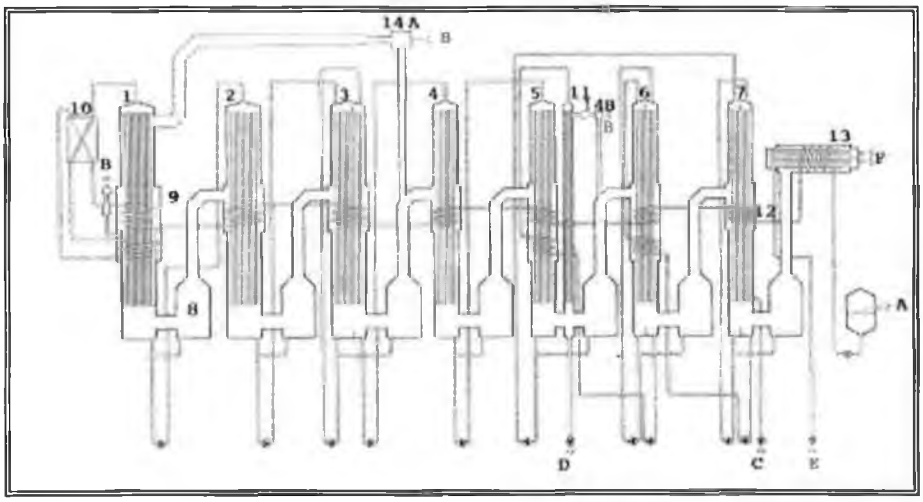


Figure 6-2. Seven effect evaporator with finisher.

A: Feed; B: Boiler steam; C: Condensate outlet; D: product outlet; E: vacuum; F: cooling water; 1: first effect; 2: second effect: 3: third effect; 4: fourth effect; 5: fifth effect; 6: sixth effect; 7: seventh effect; 8 vapour separator; 9: pasteurising unit; 10: heat exchanger; 11: finisher; 12: preheater; 13: condenser; 14a and 14b: thermocompressor. (Courtesy APV ANHYDRO A/S Denmark.)

6.4.4 Concentrate heating

Heating of concentrated milk before atomization has two purposes; (a) to obtain a final product with good bacteriological quality and (b) to achieve better efficiency and easier atomization during the spray-drying process (de Vilder *et al.*, 1979).

6.4.5 Homogenisation

Homogenisation of milk concentrate is necessary to decrease the free fat content of WMP. Fat globules without protective membranes reduce milk powder solubility and increase susceptibility to oxidative rancidity. A high free fat content in spray-dried WMP adversely affects a number of properties:

- the dispersibility or wettability during reconstitution;
- the rising of cream and foaming or scum formation in the reconstituted milk;
- the stickiness of dried whole milk powder (Buma, 1971a).

Homogenisation is the process whereby the size of fat globules is reduced from the range of 1-10μm (mean 3.5μm) commonly found in milk to less than 1μm (Banks and Evans, 1985). The newly formed smaller fat globules are stabilised by protein absorption onto their surface. The quantity of proteins being adsorbed varies with the homogenising pressure and temperature. Casein is the major protein adsorbed but whey proteins as well as the natural globule membrane are also present (Mulder and Walstra, 1974).

Homogenisation is usually carried out with a high pressure pump, composed of a high pressure piston and a special valve. The concentrated milk is forced through the valve. This has the effect of reducing the size of the suspended particles, increasing the surface area to volume ratio of the dispersed phase (fat) in the continuous phase, increasing shelf life of the product and increasing the viscosity (Nistri, 1994).

Generally, 2-stage homogenisation (80 bar initially, followed by 20 bar in the second stage) of whole milk concentrate before drying is adequate to maintain free fat levels of less than 4% in WMP (Singh and Newstead, 1992). Typical free-fat levels in instant WMP are in the region of ~1.5% (Pisecky, 1990).

6.4.6 Spray-drying

Spray-drying has numerous advantages compared to other drying techniques, in that the process proceeds very rapidly and product temperature-rise is minimised by exploitation of evaporative cooling effects during the primary stages of dehydration. Oxidation does not occur during the actual process though it remains to be resolved as to whether the process (for example, the preheating stage) gives rise to pro-oxidant as well as antioxidant effects. Loss of vitamins, denaturation of proteins, lactose transformation and the other adverse effects of heat are generally low in this process. During spray-drying, it is possible to control the final properties of the powder, such as moisture, bulk density and powder particle size. It is necessary to be able to do this in order to produce powders with different functional specifications (Caric, 1994).

The principle of spray-drying (Figure 6-3) is to remove the water from the concentrate as fast and at as low a temperature as possible, so as to minimise heat damage to the milk solids. This is achieved by spraying the milk concentrate in the form of very fine droplets into a hot dry air-stream (e.g. 190 °C) and thus exposing a large evaporative surface. The more finely dispersed the milk droplets, the larger their specific area will be and the more effective the drying. Atomizing the feed increases the specific area of 1 litre of milk by approximately 700 times.

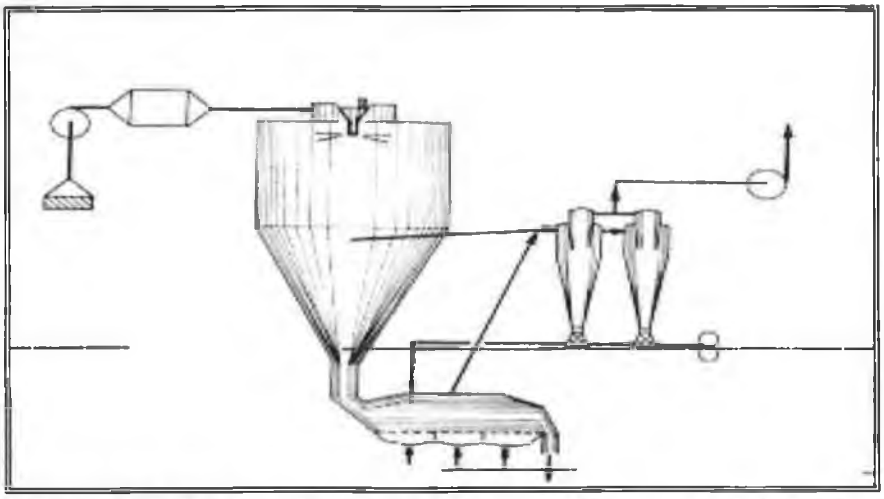


Figure 6-3. Drying chamber with conical bottom and fluid-bed.

(Courtesy APV ANHYDRO A/S Denmark).

Atomization is achieved by either a nozzle or a rotary wheel atomizer, and it should ideally produce uniformly sized particles, as smaller particles may overheat and larger particles take longer to reach the desired moisture level (Singh and Newstead, 1992). With disc atomization, the properties of the product are controlled by the speed of rotation of the disc. In a stationary nozzle which discharges the milk in the same direction as the airflow, the milk feed pressure determines the particle size. The feed can also be presented counter-flow to the drying air. With pressure atomization, the powders that are produced have higher particle densities than those generated with wheel atomization. Higher particle densities contribute to higher bulk densities, improved reconstitution properties and better shelf life of fat-filled products like whole milk powder (Boersen, 1990).

6.4.6.1 Agglomeration of Whole Milk Powder

Before use, food in the form of powder is generally dispersed and/or dissolved in water or in aqueous liquid. Water wets very fine powders only very poorly because of its high surface tension. In order to improve the wettability and accelerate dispersion and dissolution, such powdered products are often made instant or are agglomerated. Agglomeration is a process of forming larger units or clusters from a number of smaller ones, while still retaining the original particles in identifiable form (Figure 6-4). Conditions which contribute to the enhanced stability of particles are promoted. Reconstitution properties of the powder are also dramatically affected by the agglomeration process. It has the effect of shortening the time for capillary penetration of the liquid into the powder to wet uniformly without lump formation (Schubert, 1993).

Agglomeration is applied to improve the flowability of powders (Boersen, 1990). Instantizing or agglomeration of WMP causes an increase in the amount of air incorporated in between the powder particles. During reconstitution, the air is replaced by water and the powder particles are wetted and dispersed before dissolution starts. Incorporated air enables a large quantity of water to come in contact with powder particles during reconstitution. This prevents formation of a viscous layer around grouped particles, which could hinder further water penetration and slow the dissolving time (Wulff, 1980).

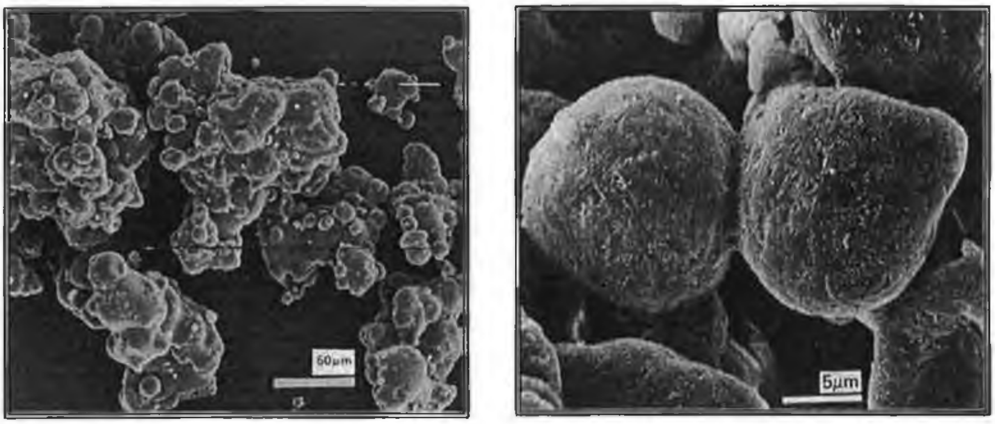


Figure 6-4. SEM of agglomerated WMP at different magnifications (Caric, 1994).

There are two basic types of instantizing: the "rewet" process, whereby instantizing is carried out after the powder is obtained in dry form, and the "straight-through" process, where instantization is accomplished during drying.

6.4.6.2 "Rewet Process"

Instantization by the "rewetting" method includes two main agglomeration techniques: droplet agglomeration and steam agglomeration. In the droplet agglomeration technique, concentrated milk is spray-dried into the drying chamber. Some distance below the atomizer, water mist or another desired liquid is atomised into the drying chamber to wet the powder particles. In the steam agglomeration technique, steam is mixed with the milk powder in the drying chamber. The powder is dispersed and wetted in the wetting chamber up to a water content of 5-10%, causing the formation of powder particle agglomerates (Caric, 1994), which should be in the region of 100-250µ in diameter. There should be as few non-agglomerated particles as possible (Pisecky and Westergaard, 1972). The choice between droplet and steam agglomeration is dependant on the desired final properties of the powder.

6.4.6.3 "Straight-Through" Process

In the "straight-through" instantization process, agglomeration is carried out during drying and immediately after powder particles have been formed. This process exploits the benefits of recycling fines generated in the final stages of drying to the atomisation zone by creating a powder "core" to which moisture droplets and wet particles can adhere. Water from the droplets evaporates on contact with the hot air stream (~ 190°C). At this stage the particle reaches the cooler region (< 90°C) of the dryer where exhaust air conditions are adjusted, so that the discharged powder contains a moisture level of 6-7 %. If the drier is fitted with an integrated fluid-bed (static bed), then a higher moisture content (9-10%) in the powder is permissible. The powder is subsequently transferred through a vibrating fluidised-bed drier, where excess water is removed, resulting in a powder of 2.8-3.0% moisture. The vibrating drier or fluidised-bed drier consists of a casing with a perforated bottom (Figure 6-5). The casing is spring mounted and can be vibrated by means of a motor. The powder from the drying chamber is admitted into the first section where it is humidified. The vibrations convey the powder through the drying sections, where air at a gradually decreasing temperature is admitted through the powder bed. Agglomeration takes place in the first stage of drying, when the particles adhere to each other. The water is evaporated from the agglomerates during their passage through the drying sections. The screened and instantized particles are conveyed by the cooling air to the cyclones, where they are separated from the air and packaged. After separation, the fines are fed back to the atomization zone to be agglomerated with the wet powder (Singh and Newstead, 1992; Caric, 1994). Excess water is removed in the fluidised bed, resulting in a powder of 2.8 - 3.0% moisture. Whole milk powder produced by this method produces mechanically more stable agglomerates than the rewet-agglomeration process (Woodhams and Murray, 1974).

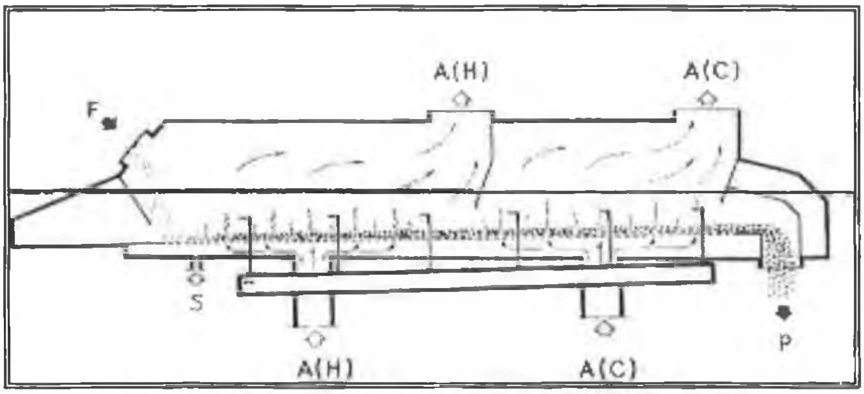


Figure 6-5. Fluidised bed drier (instantiser).

F-feed, A[H]-hot air, A[C]- cooling air, S-steam, P-Product. Courtesy Alfa Laval.(Caric, 1994).

6.4.7 Lecithination

A major factor responsible for the low level of acceptability of WMP as a consumer product was the difficulty of reconstituting the powder in cold water. Considerable mechanical agitation was required to dissolve the powder and essentially, warm water was necessary for reconstitution. Therefore there was a need for a grade of WMP which was not alone instant but could also be readily reconstituted in cold water (Baldwin and Sanderson, 1973).

WMP particles which are covered in a layer of free-fat are water-repellent. For a powder to be instant, it should have a good wettable surface, which is determined by the free fat content (discussed in section 5.6.5). The water-repellency of the particles caused by their fat-coating may be overcome by application of a surface active agent such as lecithin dissolved in butter oil. As a result, wetting of the powder can take place immediately after a suitable interfacial tension has been reached. Lecithin is particularly acceptable, because it is a naturally occurring milk component. Lecithin (L-α-lecithin is the naturally occurring form, also known as phosphatidyl choline) is a phospholipid mixture with hydrophobic and hydrophilic properties and can therefore affiliate with both water and lipids. On reconstitution of WMP, it acts as an emulsifier (Aurand and Woods, 1973). Lecithin for this purpose is sourced from soyabean oil.

For lecithin to work well in these applications, it must be applied in a thin uniform layer to the surface of the powder. Most lecithins are moderate to heavy viscous fluids which are difficult to spray in undiluted form at room temperature. In such cases, the lecithin can be treated to get a viscosity suitable for spraying, by heating or thinning with additional oils such as butter oil.

Lecithination consists of two stages; initially, the lecithin/butter oil solution is sprayed onto the powder granules, generally with two fluid nozzles, after which the suspension must be spread out to complete the coating. This process is called conditioning and is carried out in a fluidised bed. A final lecithin concentration and application in WMP should be 0.2% (w/w) with a coating of 0.01-0.15 μ in thickness (Pisecky and Westergaard, 1972).

6.5 Properties of Instant Whole Milk Powder

6.5.1 Particle Size

The particle size distribution of a powder is a measure of the average particle diameter and of the spread of sizes on either side of this average (Woodhams and Murray, 1974). The particle size of powders affects appearance, reconstitution and flow characteristics. It depends on the atomization conditions and on viscosity of the concentrate, for example, at high atomizing pressures and low viscosities, the particle size decreases (Woodhams and Murray,1974). The average size of particles produced in disc atomizer driers is decreased by increasing the peripheral speed of the atomizer wheel or by reducing the feed rate of the concentrate. For powders produced by spray driers fitted with nozzle atomizers, the average particle size is decreased by increasing the pressure at which the concentrate is supplied to the nozzle and by increasing the swirl and axial velocities of concentrate through the orifice (Woodhams and Murray, 1974). It was also found that high inlet air temperatures produced a larger mean particle size and that a high atomizing pressure produced a smaller size, for a nozzle atomised feed

(Bloore and Boag, 1982). The mean particle size for an agglomerated WMP should not be smaller than 180µ, the fraction below 125µm not greater than 20% and the fraction above 500µ not over 10% (Pisecky, 1990).

6.5.2 Bulk Density

Bulk Density is a measure of the mass of powder which occupies a fixed volume. It is normally expressed in grams per millilitre (g/ml). A typical bulk density for an agglomerated WMP is ~ 0.45g/ml (1250 taps). The bulk density depends on three factors:

1. the density of the milk solids,
2. the amount of occluded air within the particles,
3. the way in which the particles pack together.

The processing variables which have a major effect on the bulk density of the powder are (i) the total solids content of the concentrate and (ii) the temperature difference between the air and the droplet in the drier (Woodhams and Murray, 1974). Inlet air temperature has the greatest effect on the bulk density of the powder, with higher temperatures giving rise to lower bulk densities. Concentrate flowrate also has a strong influence: high flowrates increases bulk density. The effect of increasing the atomizing pressure is generally to reduce bulk density but it also increases the effect of inlet air temperature and reduces flowrate (Bloore and Boag, 1982).

6.5.3 Particle Density

Particle density is an expression of the content of occluded air. A high content of occluded air may result in the creation of foam on the surface of a reconstituted solution. Particle density should be 1.18g/cm^3 for instant WMP (Pisecky, 1990). The particle density is determined by the drying conditions. A high particle density is required because it ensures:

- good sinkability of the particles
- absence of foam or scum
- ease of gas packing (Verhey, 1972).

The atomization system influences the content of occluded air. Rotary atomizers cause a higher occluded air content than pressure nozzles. Other factors involved include the feed properties and droplet drying technique. Air being whipped into the concentrate is the primary source of occluded air (Pisecky, 1990).

6.5.4 Moisture Content

The moisture content of a powder is estimated by evaporating the residual water from a known mass of powder in an oven under precisely controlled time and temperature conditions or by distilling the water heated in toluene (Woodhams and Murray, 1974). Typical values for moisture in instant WMP are in the region of 2.7-3.0 %. An excessively low moisture content can have an adverse effect on the free fat content while a very high value can effect shelf life, especially if the product is being transported to tropical countries, as lumps can develop in tins (Pisecky, 1990).

The moisture content of powder is controlled by the relative humidity of the exhaust air, which in turn, is controlled by the temperature of the air, or by controlling the flowrate of the concentrate into the drier or by adjusting the inlet air temperature. Because there is some gain in moisture due to pneumatic conveying and blending and to a lesser extent during storage, it is normal to produce powder from the drier at a lower moisture than required, so that the final powder is within specification (Woodhams and Murray, 1974). According to Bloore and Boag (1982), if the feed flowrate is reduced at the same time as inlet temperature is increased, the moisture content of the powder drops sharply. Increased inlet temperature reduces the moisture content of powders, while increasing the total solids of concentrate also causes a slight decrease in moisture levels.

6.5.5 Free Fat Content

The free fat content of WMP is a measure of the amount of fat that can be extracted from food powders by an organic solvent. It is meant to represent fat on the surface, however, it also includes fat which originates from the pores and capillaries within the particle (Buma, 1971b). A technique called Electron

Spectroscopy for Chemical Analysis (ESCA) was developed by Fådlt *et al.* (1993), which can accurately determine the fat, protein and carbohydrate coverage on the outer surface of the powder particles. The chemical composition of the surface of whole milk powders produced in laboratory, pilot and production plants were similar and consists of 55% fat, 30% protein and 15% lactose (Fåldt and Sjöholm, 1996).

High levels of free fat are normally caused by abrasion which damages the protective coating of phospholipids and protein surrounding the milk fat globules. Production of powders with a high free fat content can be avoided by reducing pumping of the fluid milk to a minimum, homogenising the concentrate prior to drying and handling the powder in a manner which minimises abrasion until after the powder temperature has been reduced to below 30°C (Woodhams and Murray, 1974). Free fat levels in instant WMP vary depending on the type of drying system used. For example, a tall-form dryer produces powder of 1.43% free fat compared to a 2.38% for a multi-stage dryer (Pisecky, 1990).

6.5.6 Flowability

Coarse agglomerated powders have better flowability than powders of small mean particle size. Therefore, powders with larger particle sizes are preferred. The flowability of marketed powders is between 20-250s, good flowability however is less than 80s (Pisecky, 1990).

6.5.7 Heat Classification-Whey Protein Nitrogen Index

This is influenced by the heat treatment prior to concentration. According to the ADMI-method, High Heat powders have <1.5 g WPN/ g powder, Medium Heat powders have 1.51-5.99g WPN/g powder and Low Heat powders have > 6.0g WPN/ g powder. (ADMI, 1971b).

6.5.8 Heat Classification-Heat (Casein) Number

Heat number (also referred to as casein number) is a more accurate method than WPNI for determination of the heat classification of milk powders. The powder is categorised into one of four heat classes; Low Heat

(≤ 80.0), Medium Heat (80.1 - 83.0), Medium-High Heat (83.1 - 88.0) and High Heat (≥ 88.1). The Heat Number is used as a reference method whereas the WPNI is used for routine analysis (IDF, 1982)

6.5.9 Reconstitution Properties

Reconstitutability refers to the ease with which a powder can be dissolved in water. Factors affecting reconstitutability include:

1. Structure and physical properties of the powder, for example, particle size, shape, density, porosity and specific volume;
2. Chemical properties of the powder, for example, free fat, extent of protein denaturation;
3. Conditions of reconstitution, for example, temperature and quality of water, time and speed of agitation;
4. Age of powder and storage conditions (Walstra and Jenness,1984a).

The reconstitution properties are also affected by the processing conditions e.g. the type of dryer equipment, system of atomization, preheat treatment of milk concentrate, total solids and heating, outlet air temperature, storage time and temperature (Woodhams and Murray, 1974; Bloore and Boag, 1982). Instant WMP should also produce a well reconstituted solution, when dissolved in hot water or hot beverages such as tea or coffee (Pisecky, 1980).

The complete reconstitution of a mass of milk powder is difficult. This is because of the wetting of powder particles not only on the water surface but also lying above the surface, as water is drawn towards them by capillary attraction. The replacement of interstitial air by water through capillary penetration is often incomplete, which results in air bubbles between the wetted particles. Three phases then co-exist with products of varying concentrations. This co-existence is very dangerous, as the space between the particles fills with dissolved products. This results in a jelly with islands of unwetted powder and residual air. In addition lumps are created, which are wet on the outside, dry inside and are impervious to water. This penetration of water into the powder can be prevented, when the powder consists of large agglomerates and high interstitial air volumes (Wulff, 1980). The process of reconstitution involves the following phenomena:

Reconstitution Phenomena	**Milk Powder Properties**
Wetting	Wettability
Penetrating	Penetrability
Dispersing	Dispersibility
Sinking	Sinkability
Dissolving	Solubility

6.5.9.1 Wettability

One the most important properties that instant powder should possess is good wettability. Instant milk powders when poured onto cold water, should pass the surface at a reasonable rate and produce a liquid equivalent in quality and appearance to whole milk, after mixing (Mohr, 1960). Wettability depends mainly on the nature of the surface of the particle and the presence of fat at the surface inhibits wetting of the particle. It is possible to overcome this water repulsion by treating the surface with a surface active agent such as lecithin dissolved in pre-butter oil (Pisecky and Westergaard, 1972).

The melting point of fat in the powder also greatly influences the wettabilty. Milk powder which contains a butter-fat fraction melting at 19-21 °C, has better wettability than a powder prepared using butter fat with a melting point of 33-35 °C (Baker *et al.*, 1959). Bullock and Winder (1958) observed that WMP, when first removed from the drier, exhibits a greater wettability than the same powder after storage at room temperature for a short period.

6.5.9.2 Penetrability

Penetrability is the ability of powder particles to penetrate the surface of the liquid. This property will depend on the structure and porosity of the powder and also on the wettability (Wulff, 1980).

6.5.9.3 Sinkability

Once wetted, the powder particles start dissolving, dispersing and simultaneously sinking into the water. Sinkability is the ability of the powder particles to overcome the surface tension of the water and sink into the water after passing the surface (Singh and Newstead, 1992). In order to achieve good sinkability, the density of the particles has to be greater than that of the water, so the content of occluded air should be low (Pisecky and Westergaard, 1972). A large particle size enhances sinkability (Woodhams and Murray, 1974).

6.5.9.4 Dispersibility

Dispersibility reflects the ability of the wetted, aggregated particles to become uniformly dispersed on contact with water. This property decides whether or not a powder is instant. It is related to the rate at which lumps and agglomerates fall apart. If too much fine material is present, there is a tendency for lumps to be cemented by a thick layer of wetted particles. Without these fine particles the water penetrates the lumps and causes dispersion more readily. In general, large particles are easier to disperse than small particles (Baldwin and Woodhams, 1974). The dispersibility of a powder can be improved by; (i) keeping the preheat treatment to a minimum; (ii) minimising the holding time and heat treatment of the concentrate; (iii) increasing the particle size (Singh and Newstead, 1992). Dispersibility was also found to improve, if the butter-fat fraction has a melting point of 19-21°C, compared to powders produced from butter-fats with higher melting points (Baker *et al.*, 1959).

6.5.9.5 Solubility

Solubility is a measure of the final conditions to which the powder constituents are brought in a stable suspension. The solubility index is used to determine the undissolved residue, which usually consists of denatured whey protein, caseins and minerals. Typically, a low solubility index may be the result of a high solids concentration or due to excess heat treatment during the production process (Singh and Newstead,1992).

The solubility index is an indirect measure of the extent of denaturation of the proteins and protein-salt complexes in milk powders. The greater the solubility index, then the greater the amount of denaturation. The solubility index for an agglomerated WMP according to ADMI methods of analyses is 0.2ml (ADMI, 1971a). High solubility indices may be caused by:

- Poor Milk Quality,
- Excessive burn-on in the evaporator,
- Excessive holding times for milk concentrates especially at elevated temperatures,
- Subjecting partly dried powder particles to high temperature air,
- Failure to cool the powder before silo storage or packing,
- Single-stage drying with high exhaust air temperature (Woodhams and Murray, 1974).

Inlet air temperature clearly has a dominant effect with higher temperatures giving rise to higher solubility values. Higher concentrate flowrates and atomizing pressures reduce solubility index, while increasing total solids concentration causes only a very small increase. An increase in total solids requires an increase in concentrate temperature, if viscosity is to be held constant (Bloore and Boag, 1982).

6.6 Compositional Factors that influence the Heat Stability of Milk and Milk Concentrates

There are no reports in the literature relating to the thermal stability of instant WMP in coffee. However, much work has been carried out on the heat stability of milk and milk concentrates and its influence on powder properties. Heat stability refers to the relative resistance of milk to coagulation when it is heated to sterilization temperatures (~140 °C). Instant WMP is added to coffee at temperatures of ~ 80 °C, which is much lower than sterilization conditions. However, the literature dealing with the effects of composition and processing (c.f. Sections 6.6 and 6.7) on heat stability may be relevant to the production of a coffee stable instant WMP.

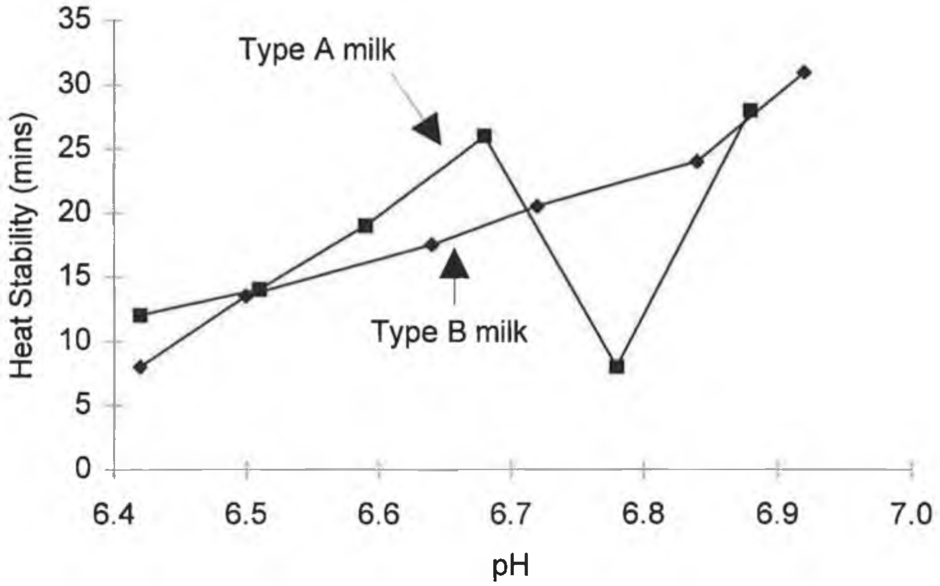


Figure 6-6. pH-heat stability of individual milk. (Tessier and Rose, 1964)

6.6.1 pH

Tessier and Rose (1964) found that the milks from individual cows could be classified as one of two types (Figure 6-6). Type A milk shows a pronounced maximum and minimum in the Heat Stability (H.S.)- pH profile, whereas the H.S. of type B milks increases gradually throughout the pH range. Most of the milks tested in Ireland are of Type A. The H.S. of concentrated milk is much lower than that of unconcentrated milk. However, the pH of maximum H.S. for unconcentrated milk is very similar to that for concentrated milk, which usually occurs in the pH range 6.4-6.6. However, concentrated milk remains unstable at all pH values greater than 6.8 and therefore does not possess a H.S. minimum.

Lactose is a major source of acidity when milk is heated. Such acidity is responsible for approximately half the decrease in pH. The acidity is mainly due to the thermal decomposition of lactose. Also, the acidity derived from the primary and secondary effects of the Maillard reaction between lactose and the ε-amino group of lysine residues will contribute to the pH decrease (Sweetsur and White, 1975).

6.6.2 Season/Lactation

In dairying countries with seasonal production patterns, e.g. New Zealand and Ireland, the problem of low heat stability occurs primarily at the beginning and end of lactation. During this period, the pH of the milk may or may not coincide with the pH of the maximum in the pH-H.S. profile (Singh and Newstead, 1992).

The urea content of milk is known to have an effect on the H.S. of milk and it has been suggested that as much as 80% of the seasonal variation in the H.S. is due to changes in the urea levels (Holt *et al.,* 1978b).

It was observed that milk powder prepared from summer milk is more wettable than powder made from winter milks (Bullock and Winder, 1958; Baker *et al.*, 1959). This has been attributed to the lower melting point of the fats in summer milk.

¯Et3b0s12v1P6.6.3 Milk Salts

For many years, variations in the H.S. of milk were attributed to differences in the salt balance (Sommer and Hart, 1919). However, it has since been proven that this is not the case, since colloidal salts such as colloidal calcium phosphate (C.C.P.) are important for the structure of casein micelles and its removal can markedly improve the H.S. of whole milk (Pyne and McGann, 1960, Rose, 1962). Soluble salts such as calcium phosphate, also influence the H.S. of milk. Newstead (1977), showed that a reduction in the concentration of soluble salts increases the H.S. of concentrated milks.

6.6.4 Milk proteins

Newstead *et al.* (1977) showed that the H.S. of concentrated milk decreases with increasing whey protein concentration. Muir and Sweetsur (1978) also found that concentrated milks with high levels of β-lactoglobulin were significantly less stable than those with less β-lactoglobulin.

Rose (1962) demonstrated that the reaction between denatured β-lactoglobulin and κ-casein was a major factor responsible for the complex H.S.-pH pattern of most milks. Further work by Tessier and Rose (1964) suggested that the H.S. patterns of milk are controlled by the proportions of micellar surface κ-casein and soluble β-lactoglobulin.

Muir and Sweetsur (1978) also showed that the addition of κ-casein to milk before concentration increased the H.S. of the resulting concentrate over the entire pH range.

6.6.5 Urea

The ability of urea to increase the heat stability of raw milk is well established (Pyne, 1958, Robertson and Dixon, 1969, Muir and Sweetsur, 1976, 1977, Holt *et al.,* 1978a). Fox *et al.,* (1980) suggested that the most likely mechanism by which urea increases the H.S. is via heat-produced ammonia from urea which neutralises the acidity produced during heating. It has been suggested that as much as 80% of the seasonal variation in H.S. could result from changes in the urea level (Holt *et al,*1978b). However the ability of urea to improve the H.S. of skimmed milk was decreased due to increased severity of homogenisation (Sweetsur and Muir, 1983a). However, in contrast to normal milks, urea does not increase the heat stability of concentrated skim milk (Muir and Sweetsur, 1977).

6.6.6 Lactose

Generally, lactose is considered to be a destabilising factor in the H.S. of milk. Also, urea was found to stabilise milk only in the presence of lactose (Kudo 1980, Shabali and Fox, 1982a) The effects of lactose on the H.S. of concentrated milks, have not yet been reported.

6.7 Changes in Milk Components during the Manufacture of Milk Powder

6.7.1 Standardisation

This has a negligible effect on milk components (Singh and Newstead, 1992).

6.7.2 Preheating

It has long been known that preheating milk increases the heat stability of the resulting concentrate (Webb and Holm, 1932, Webb and Bell, 1942). However, Rose (1962) showed that the true effect of preheating on the heat stability of unconcentrated milk was due to a shift in the pH of maximum stability. Preheating increased stability where the natural pH of milk was lower than the pH of the maximum stability but reduced it if the pH of the milk was on the alkaline side of the pH of maximum H.S. (Griffin *et al.*, 1976).

The main factor influencing the H.S. of reconstituted powders is the level of heat treatment applied during the preheating stage. Preheat treatments of 80-90°C for 10-20 min are commonly used. However, it has been shown that preheating temperatures in excess of 100°C with shorter holding times produce more stable powders. Heat treatments in the region of 110-125°C for 30-240s are favoured in New Zealand for the manufacture of heat-stable milk powders (Singh and Newstead, 1992). Increasing preheat temperatures (> 110°C) enhanced the heat stability of skim milk powder even more when urea levels in the milk were supplemented (total urea content 70-90mg/100ml) (Kelly, 1982). Preheating of milk results in many physical and chemical changes including: (1) destruction of bacteria, (2) inactivation of enzymes, (3) denaturation of whey proteins, (4) formation of a complex between κ-casein and β-lactoglobulin, (5) transfer of soluble salts to the colloidal phase, (6) modification of casein micelle structure, (7) Maillard browning reaction between protein and lactose, (8) decrease in pH, (9) development of antioxidant activity, (Singh and Newstead, 1992).

6.7.2.1 Inactivation of enzymes

Inactivation of enzymes by heat treatment usually depends on conditions such as pH, ionic strength and water activity, as well as the time and temperature of heating. Milk contains naturally occurring proteases and lipases which degrade protein and fat and may contain similar enzymes derived from contaminating micro-organisms and thus heat treatment is required for their inactivation (Singh and Newstead, 1992).

6.7.2.2 Protein denaturation

Whey proteins (principally, β-lactoglobulin and α-lactalbumin) which account for approximately 20% of the total milk protein, are heat labile, while the caseins are less heat sensitive. During heating to temperatures in excess of 65°C, denaturation of the whey proteins occurs and they become attached to the casein micelles, via κ-casein/β-lactoglobulin interaction. During heat treatments, the side chains of some amino acids become reactive and combine with other proteins or with other constituents of milk. During denaturation, whey proteins complex with the casein micelles, probably via interaction with κ-casein and α_{s2}-casein. The extent of denaturation is also dramatically affected by pH, ionic strength, and the concentrations of protein and calcium (Hillier *et a.l,* 1979, Park and Lund, 1984; Dannenburg and Kessler, 1988).

Apart from the decomposition of casein during heating, polymerisation of caseins as a result of condensation reactions e.g. Maillard-type, also occurs (O'Brien and Morrissey, 1989)

6.7.2.3 Casein/Whey Interactions

The formation of a complex between β-lactoglobulin and κ-casein has long been recognised (Zittle *et al.,* 1962; Long *et al.,* 1963; Sawyer *et al.,* 1963). Direct interaction between α-lactalbumin and κ-casein when heated is limited, if it occurs at all, but the complex formed between α-lactalbumin and β-lactoglobulin is able to interact with κ-casein (Elfagm and Wheelock, 1977). This results in aggregation and precipitation of the protein complexes (Singh and Newstead, 1992).

When milk is heated above 90°C, whey proteins denature and interact with κ-casein on the surface of the casein micelles, forming thread-like appendages (Creamer and Matheson, 1980; Mohammad and Fox, 1987). The extent of association of denatured whey proteins with casein micelles, is markedly dependant on the pH prior to heating. If $pH < 6.7$, a greater quantity of denatured whey protein associates with the κ-casein micelles. If $pH > 6.7$, whey protein/κ-casein dissociate from the micelle surface, apparently due to dissociation of κ-casein (Singh and Fox, 1985, 1986). The interactions between denatured whey proteins and casein micelles markedly affect the H.S. of reconstituted milk powder.

Newstead *et al.* (1977) showed that concentrated milk from which the whey proteins had been largely removed, was heat-stable irrespective of whether the milk was preheated or not. However, the detrimental effect of whey proteins on the heat stability of the caseinate system of concentrated milk may be reduced by heat treatment of milk prior to concentration. Preheating before concentration, probably causes both the complexation of β-lactoglobulin with the casein micelles and some precipitation of calcium phosphate. Thus, casein micelles coated with β-lactoglobulin and calcium phosphate may be less susceptible to dissociation during subsequent concentration and sterilisation than micelles in non-preheated milk.

6.7.2.4 Distribution of Salts

Heating milk reduces ionic calcium, soluble calcium and phosphate, by converting them to the colloidal phase. Therefore, because of its association with casein micelles, precipitated calcium phosphate does not sediment at preheating temperatures up to 85°C (Evenhuis and DeVries, 1956). On subsequent cooling, some of the precipitate may redissolve (Kannan and Jenness, 1961). Heat treatment has little effect on the monovalent ions of sodium, potassium or chlorine (Kannan and Jenness, 1961). However, *Hardy et al.*, (1984) reported that changes in the mineral equilibria influence the heat stability of sterilized concentrated milk, probably by differences in the concentrations of soluble calcium.

6.7.2.5 Modification of casein micelle structure

Preheat treatment causes the denaturation of whey proteins and their interaction with the casein micelles. The extent and type of association between whey proteins and casein micelles depends on the severity of heat treatment. (Hostettler *et al.,* 1965; Josephson *et al.,* 1967). The increase in size of the casein micelles is thought to be due to the deposition of denatured whey proteins on to the micellar surfaces of the casein micelles and the precipitation of calcium phosphate (Singh *et al.,* 1989).

6.7.2.6 Interactions of Lactose and Proteins

Heat treatment catalyses the Maillard reaction in milk, which involves the condensation of lactose with the free amino groups of protein, leading to the formation of brown pigments. These pigments result in development of off-flavour, loss of available lysine, reduced digestibility and solubility in addition to discoloration on milk powders. The reaction rate is strongly dependent on pH, time and temperature of heating, water activity and temperature during storage (Burton, 1983).

6.7.2.7 Decrease in pH

The pH of milk decreases on heating due to release of H^+ ions, caused by the precipitation of calcium phosphate and the formation of acids from lactose. Pyne and McHenry (1955) attributed 50% of total acidity to lactose decomposition, approximately 30% to casein dephosphorylation and the remainder to changes in phosphate equilibria. Heat induced changes in pH due to shifts in milk salt equilibria are largely reversible but re-establishment of equilibrium conditions on cooling is slow, especially following severe heat treatments.

6.7.2.8 Development of antioxidant activity

The development of reducing substances, such as reactive sulphydryl groups which may prevent oxidised flavour development, occurs during preheating (Gould and Sommer, 1939, Thomas, 1954). Free thiol (SH) groups act as free radical scavengers and thus, as antioxidants in biological and other systems. Whole milk powder produced using high preheat treatment in order to generate a high level of sulphydryl groups is resistant to oxidation (Harland *et al.*, 1952).

6.7.2.9 Changes in antigenicity

For milk powders used in baby-milk formulations, the destruction of antigenic properties is essential. The antigenic properties of the immunoglobulins and serum albumins are destroyed by heating at 70-80°C for 15min, but β-lactoglobulin and α-lactalbumin remain active after heating up to 100°C/15mins. Caseins retain their antigenicity on heating up to 120°C for 15min (Hanson and Mansson, 1961). Thus, it would seem that the antigenic properties of the major milk proteins are not destroyed by the commonly-used preheat treatments.

6.7.3 Evaporation

In addition to concentration, evaporation causes numerous changes in the milk system which is also dependent on preheat treatment, temperature during concentration and time elapsed after concentration (Singh and Newstead, 1992). This process results in : (1) concentration of solids; (2) some further denaturation of whey proteins; (3) increase in colloidal salts; (4) increase in casein micelle size and (5) further decreases in pH.

The pH of milk decreases during concentration from an average initial value of 6.7 to approximately pH 6.3 at 45% solids (Howat and Wright, 1934). This is partly due to changes in the salt equilibria, as more calcium phosphate is transferred from the soluble to the colloidal phase, with a concomitant release of hydrogen ions, which lowers the pH.

Concentration of milk causes closer packing of the casein micelles, which are themselves about two-thirds water and higher concentrations of whey proteins, lactose and colloidal salts. Casein micelles may increase in size due to the increase in colloidal calcium phosphate or due to the coalescence of micelles (Walastra and Jenness, 1984a). Also the sensitivity of casein micelles to heat increases with increasing concentration (Webb and Holm, 1932).

The viscosity of milk increases with increasing concentration and the effect is most marked when the concentration of milk solids exceeds 45%. Concentrate viscosity can have a major influence on the size of the droplets formed during atomization and hence, on the rate of drying and final powder particle size distribution. These factors in turn, affect the physical properties of the dried product (de Vilder and Marten, 1974; King *et al.,* 1974; Baldwin *et al.,* 1980). Generally, raising the preheat temperature increases concentrate viscosity and the effect is greater for longer preheat holding times.

6.7.4 Concentrate heating

Concentrate heating (50-80°C) tended to decrease the solubility index and viscosity, but had virtually no effect on free fat, volume of occluded air, bulk density or particle density of WMP (non-agglomerated) when the dry matter content was < 45% TS. At higher dry matter contents, increasing the concentrate temperature increased the free fat levels (de Vilder *et al.,* 1979).

6.7.5 Homogenisation

During homogenisation, the surface area of the fat is greatly increased and is covered by plasma components, the major material adsorbed being protein. The quality of protein adsorbed varies with the homogenising conditions such as pressure and temperature. The major protein adsorbed is casein but whey proteins as well as the natural globule membrane material, are also present (Mulder and Walstra, 1974). Mol (1975) showed that the adsorption of casein onto the fat globule during homogenisation of the concentrate, adversely influences the solubility of the final powder because casein micelles adsorbed onto the fat globules are less stable during drying than those normally present in milk plasma (Singh and Newstead, 1992).

Sweetsur and Muir (1982b) confirmed the destabilising influence of homogenisation at high pressures on the heat stability of milk concentrates. Marked seasonal differences were also shown in the effects of homogenisation on the heat stability of non-preheated whole milk before concentration. Summer milks (May-July) were found to be more stable than winter (October-December) milks and to be more readily stabilised by added phosphate or citrate. Soluble salts, especially calcium, rather than seasonal variation in fat content, were shown to be a major determinant factor affecting the heat stability of concentrated, homogenised whole milks (Sweetsur and Muir, 1982a).

Homogenisation of milk is also known to produce sulphydryl compounds, denature proteins (Harper, 1976) and effect changes in milk salts such as citrate and phosphate (Rose, 1963), factors that can also influence HS. Sweetsur and Muir (1983a) investigated the effect of concentrate temperature and pressure of homogenisation on the heat stability of milk. Their results indicate that milks with better heat stabilities were produced at moderate homogenisation pressures, regardless of the temperature of the concentrate.

Snoeren *et al.,* (1984) found that homogenisation of full cream milk concentrate increases its viscosity. Increases in homogenisation pressure applied to the concentrate resulted in an increase in moisture content and decreases in the free fat content and solubility of spray-dried milk obtained from it. These effects appeared to be due to the increase in viscosity of the concentrate. According to de Vilder *et al.* (1979) homogenisation of the concentrate decreased the free fat content of the powder and also the viscosity of the redissolved powder; however, this was true only for concentrates of < 50% dry matter content.

Ideally, concentrate viscosity should be constant and as low as possible to ensure the best quality instant dried whole milk powder. High viscosity may be due to poor milk quality, high protein content, high homogenisation pressure or preheating of concentrate to >80°C. This, in turn, may lead to impaired product quality (Anderson,1986).

6.7.6 Spray-drying

The native properties of the milk components are essentially unmodified by moderate drying conditions. The normal size distribution of the casein micelles and their heat stabilities are substantially recovered on reconstitution of spray-dried milk (King,1965). However, *Muir et al.*, (1978) found that spray-dried skim milk powder had improved heat stability compared to concentrated skim milk.

During spray-drying, an increase in protein denaturation and aggregation is likely to depend on the temperature of the air into which the milk is sprayed (inlet air temperature), the degree of concentration and the temperature of the concentrate prior to drying, the size of the drying droplets and temperature of the drying air/powder mixture exiting from the drier (outlet air temperature). The temperature of the droplets approaches that of the outlet air as the drying process nears completion (Parry, 1974). Therefore, the outlet air temperature is a critical parameter controlling heat damage to dry milk products. However, modern spray drier designs based on the multi-stage principle are maintaining outlet air temperatures as low as possible. At relatively high outlet temperatures, the protein may become insoluble. The insoluble components of dry milk are mainly casein, denatured whey proteins and calcium phosphate. Under normal spray-drying conditions, whey protein denaturation is negligible (Walastra and Jenness, 1984a). Some crystallization of lactose occurs in addition to a re-orientation of the more soluble milk salts, concentrating chlorides and salts of sodium and potassium in particular, in the surface layer of the powder (Bockian *et al.*, 1957). The amino-acid composition of the milk proteins is not greatly affected by the drying process; normal drying conditions cause only small losses of lysine (Renner,1983).

6.8 Process Improvements for the Production of Instant Whole Milk Powder

Sweetsur and Muir (1982b) set up a study to improve the H.S. of homogenised concentrated milk and they found that the most effective changes were brought about by (a) high temperature forewarming and (b) two-stage homogenisation. The heat stability of milk can also be manipulated by use of additives to the milk before processing or to the concentrate before drying.

6.8.1 High temperature fore-warming

The effect of forewarming has largely been attributed to serum protein denaturation (Rogers *et al.*, 1921; Sommer and Hart, 1926; Ramsdell and Hufnagel, 1953) resulting in the formation of a complex with κ-casein (Davies, 1959) and significant changes in mineral equilibrium (Leighton and Mudge, 1923; Sommer and Hart, 1926; Rose and Tessier, 1959). Forewarming also enhances the heat stabilising effect of traditional stabilisers such as sodium phosphates and citrates. Traditionally forewarming consisted of heating milk concentrate to 90°C/10mins, but in later years, it has been shown that higher temperatures (140-150°C) result in milks of higher H.S. (Sweetsur and Muir, 1981). Sweetsur and Muir (1982b) subsequently found that 145°C /5 s was the most effective forewarming method for improving heat stability of homogenised concentrated milk.

6.8.2 Two-stage/multiple pass homogenisation

First-stage high homogenisation pressures may result in some of the disrupted fat particles re-aggregating to form clumps, which may be broken up by a second homogenisation pass at a lower pressure (Jones and Harper, 1976). It was observed also that two-stage and multiple-pass homogenised concentrated milks are more heat stable than single-stage homogensied milks. Such milks were more heat-stable if pasteurised (74°C, 15s) rather than forewarmed (90°C, 10min). This is in contrast to single-stage homogenisation where forewarming was more effective than pasteurisation, in improving heat-stability of concentrated milk (Sweetsur and Muir, 1982b).

Homogenisation usually occurs after evaporation and before spray-drying; therefore, it recommended that the contents of the balance tank between the evaporator and the dryer be kept at the lowest level possible, in order to minimise residence time. If the residence time is prolonged, aggregation of protein may occur, due to a rise in temperature of the concentrate and a subsequent increase in the concentrate viscosity (Singh and Newstead, 1992).

6.8.3 Use of additives

The H.S. of milk may be modified by several chemical reagents although there are only a few legally permitted food grade additives that can be used commercially to increase the H.S. Additives that modify the heat stability are given below.

6.8.3.1 Detergents

Anionic detergents such as sodium dodecyl sulphate (SDS) increase the heat-stability of milk, as do cationic detergents such as cetyltrimethylammonium bromide (Fox and Hearn, 1978, Shabali and Fox, 1982b). Non-ionic detergents such as Triton X and Tween 60 added to milk have no significant effect (Shabali and Fox, 1982b).

6.8.3.2 Aldehydes, Ketones and Sugars

Aldehydes such as formaldehyde and other low molecular weight aldehydes increase the heat-stability of both concentrated and unconcentrated milks throughout the pH range (Nelson, 1954, Holt *et al.,* 1978b, Singh and Fox, 1985b). Urea acts synergistically with aldehydes in stabilising such systems (Muir *et al.,* 1979, Shabali and Fox, 1982a). The increase in H.S. on addition of aldehydes is due to modification of the ε-amino group of lysine (Holt *et al.*, 1978b) and to cross-linking of polypeptide chains. Arginine residues are thought to be important in H.S. (Shabali and Fox, 1982c).

Addition of simple sugars e.g. glyceraldehyde and erythrose, increases the heat stability of both normal and concentrated milks, throughout the pH range (Holt *et al.*, 1978b).

6.8.3.3 Addition of salts

Sweetsur and Muir (1982b) demonstrated that in order to ensure the heat stability of concentrated milk, stabilizing techniques should be employed before homogenisation. Traditionally, salts such as sodium phosphates and citrates are added to milk concentrates to improve their H.S. (Sweetsur and Muir, 1980). They were found to be effective only if the milk has been forewarmed. The stabilising effects of such salts is probably due to their ability to modify the pH of the concentrated milk to the value at which its heat stability is at a maximum (Pouliot and Boulet, 1991).

6.8.3.4 SH-acting compounds

It has been found that addition of sulphydryl blocking agents e.g. N-ethyl maleimide (NEM) or oxidising agents e.g. KIO_3 (Potassium Iodate) cause a large increase in the heat stability of homogenised concentrated whole milk (Sweetsur and Muir, 1983b). NEM negates the heat-destabilising effect of homogenisation. Cu (copper) which is an oxidising agent also prevents homogenisation-induced destabilization (Sweetsur and Muir, 1983b). Therefore, destabilization of milk concentrates due to homogenisation can be counteracted by preventing sulphydryl group interaction between milk proteins.

6.8.3.5 Other additives

κ-carrageenan and soyabean lecithin have both been shown to increase the H.S. of full cream concentrated milk (Hardy *et al.,* 1985). Addition of β-lactoglobulin to milk before preheating reduces the heat-stability of concentrates prepared from it and homogenisation accentuates the destabilizing influence of β-lactoglobulin (Muir and Sweetsur, 1983b).

6.9 Alternative Process for the Production of Instant Whole Milk Powder

Traditionally, the production of a WMP involves standardising the whole milk to a designated fat content (typically 3.6% (w/v)), heat treating and concentrating the milk to 45-50 % (w/v) total solids and spray drying to produce a powder of approximately 3% moisture (w/w). This method has disadvantages in that;

1. The evaporator is subject to fouling, leading to decreased efficiency and product loss (Hols and Van Mil, 1991).

2. High heat treatment before evaporation for several minutes may be required. The aim of this treatment is to reduce the autoxidation of the fat in WMP during storage by means of the antioxidant activity of the thiol (SH) groups (Kirchmeier *et al.,* 1984), whose concentration increases on heating milk at 85-90°C for several minutes. These groups are formed in the whey proteins (particularly β-lactoglobulins) but also, in the membrane proteins of the fat globules (Walstra and Jenness, 1984a).

Hols and Van Mil (1991) attempted to address these problems by an alternative process method, whereby the raw milk was separated into cream and skim milk fractions and given a separate heat treatment. Thus, for thiol group (SH) protection against oxidation of the fat, the cream is given an optimal heat treatment. The skim milk can be given a low heat treatment and the whey proteins are not unduly denatured, resulting in a less cooked flavour. An additional advantage is that the viscosity of the concentrated milk should be lower which would improve the liquid flow in the evaporator pipes and calandria. Furthermore, the lower viscosity of the concentrated whole milk when atomised in the spray-dryer will give smaller droplets and thus better powder quality (Van Mil *et al.,* 1988).

The results show that both the traditional and alternative processes produce similar powders except for the whey protein nitrogen index and free fat content. The traditional process produced a powder with greater denaturation and higher free fat content.

6.10 Fat-Filled Agglomerated Powders

In the 1960's, fat-filled powders were developed for use in coffee creamer formulations. Fat-filled powders are produced by recombining an oil e.g. coconut or palm kernel oil with skim milk concentrate. The concentrate/oil mixture is then homogenised and dried in a similar fashion to whole milk concentrate. They are preferred over WMP as they give better dispersion, are acid-stable and have better whitening ability (Hayman, 1995). However, whole milk powder does have advantages over fat-filled powders.

In whole milk, the natural emulsifying constituents in the milk appear at the surface between the fat and other milk components. However, the fat globules are covered by proteins in fat-filled powders, which are orientated towards the globular surface as a result of homogenising fat in the concentrate. This reduces the H.S. of the proteins, causes risk of denaturation in the protein fractions when drying and agglomerating and may decrease powder solubility (Hansen, 1980). A modification of the normal agglomeration technique where fines are recirculated to the cone of the spray-drying plant rather than the atomising zone, was successful in achieving powders with improved solubility (Hansen, 1980).

Melting point is an important factor when choosing an oil, as it can influence powder properties such as free fat, dipersibility and wettability (Baker *et al*, 1959; Baker and Samuels, 1961; Hansen, 1980). A low melting point coconut oil causes a higher free fat content than the same oil with a higher melting point (Hansen, 1980). Powders with better dispersibility and wettabilty were produced from low melting point butterfat (Baker and Samuels, 1961; Baker *et al*, 1959). However, powders produced from low melting point fats may have poor flow properties that result in problems during handling (Hansen, 1980).

7. MATERIALS AND METHODS

All reagents used were Analar Grade.

Distilled reverse osmosis-treated water was used for all analysis, unless stated otherwise.

7.1.1 New Zealand Dairy Research Institute Coffee Sediment Test

2g of WMP was added to 100ml of coffee (8g/l) at 80°C. A single batch of Nescafe Gold Blend reconstituted in distilled deionised water was used throughout. The solution was stirred using a spoon for 6 sec (6 turns clockwise and 6 turns anti-clockwise) and allowed to stand for 10 min, before transferring into the ADMI (American Dry Milk Institute) Solubility Index tubes. After standing for a further 5min, the sediment volume (ml) was measured following centrifugation (Super-Quatro, Funke Gerber GmbH, Berlin, Germany) at 164g (force of gravity) for 5 min (New Zealand Dairy Research Institute, 1983). As a general guideline coffee-stable WMPs yielded sediment volumes of ≤ 0.5 ml, while coffee-unstable powders yielded volumes of ≥ 1.0ml. (Figure 7-1).

Figure 7-1. Coffee-stable WMP (left) and coffee-unstable WMP (right).

Test reproducibility was checked using one sample each of coffee-stable and coffee-unstable WMP (Appendix A, Table A2). The standard deviations were 0.11ml and 0.15ml for the coffee-stable and unstable samples respectively.

The S.D. values were substantially higher than the figure of 0.05ml, which was quoted within a draft of the same coffee test for dried milk and milk products, soon to be published by the IDF.

7.1.2 Application of the coffee sediment test

Initially, it was decided to assess the application of the coffee test in detail. Twenty instant WMPs, defined by the industrial collaborator, as being coffee-stable and coffee-unstable, were subjected to a testing routine involving variation of several parameters:

1. Temperature of the coffee solution/acidified water;
2. pH of the coffee/acidified water;
3. Effects of surface active agents;
4. Mechanical effects;
5. Particle size classification of the test powders and
6. Water hardness/effect of NaCl and KCl

7.1.2.1 Temperature of Coffee Solution

In order to simulate behaviour in coffee solutions, 2g of WMP was added to acidified water at temperatures within the range 20-90°C. The dosage of 1 M HCl used (600 μl) was based on a measurement of the volume required to give a final pH of ~ 6.2, which typically resulted from the combination of WMP and coffee. The effect of reconstitution of 2g WMP in acidified water (to attain a final pH ~ 6.2) before heating to temperatures in the 20-90°C range was also determined (Teehan and Kelly, 1996b).

7.1.2.2 pH

The effect of pH on sediment formation was determined directly by prior dosage of 1 M HCl at incremental levels to the test water at 80°C and indirectly by varying the coffee concentration (Teehan and Kelly, 1996b).

7.1.2.3 Surface Active Agents

A variety of surface active agents were added to the test water along with coffee at 80°C, before addition of the WMP. The final pH of the coffee-WMP supernatant was also noted, in addition to the sediment volume. Anionic surface active agents used included Lauryl Sulfate (Sodium dodecyl sulfate) supplied by Sigma Chemical Co. (USA) and Artodan™ (Sodium stearyl-2-lactylate) supplied by Grindsted Products Ltd., (Denmark). Non-ionic surfactants such as Tween 60 (polysorbate 60) (Quest International, UK) and SP50 (E473) (Sucrose stearate-palmitate ester) Sisterna, (Roosendaal, The Netherlands) were also assessed for their effect on the coffee stability of samples of WMP with known coffee sediment volumes (Teehan and Kelly, 1996b).

7.1.2.4 Mechanical Effects

WMP was combined with the coffee by stirring with a spoon for 6s according to the Coffee test. An attempt was made to introduce a more objective means of agitation using a mechanical mixer under controlled conditions. A laboratory mixer with digitally-controlled propeller speed (Janke and Kunkel, *Ika Werk*, Staufen, Germany) was evaluated initially in a calibration test to simulate the degree of sedimentation of the spoon. The stirrer was used instead of the spoon over a range of propeller speeds (550-3600rpm) for a 6s duration in the test. A Solubility Index Blender (Labinco B.V. Breda, The Netherlands) with a programmed propeller speed of 3600 rpm for 90s was also used to investigate the effect on dispersion of WMP in coffee (Teehan and Kelly, 1996b).

7.1.2.5 Prior Reconstitution of WMP

2.0g of WMP was added to 14.0g water at 50°C (12.5% milk solids) and subsequently combined with the coffee solution (9.3g/l), prepared in the remaining 86g test distilled water. Sediments were recorded for coffee-stable and coffee-unstable WMPs (Teehan and Kelly, 1996b).

7.1.2.6 Particle Size

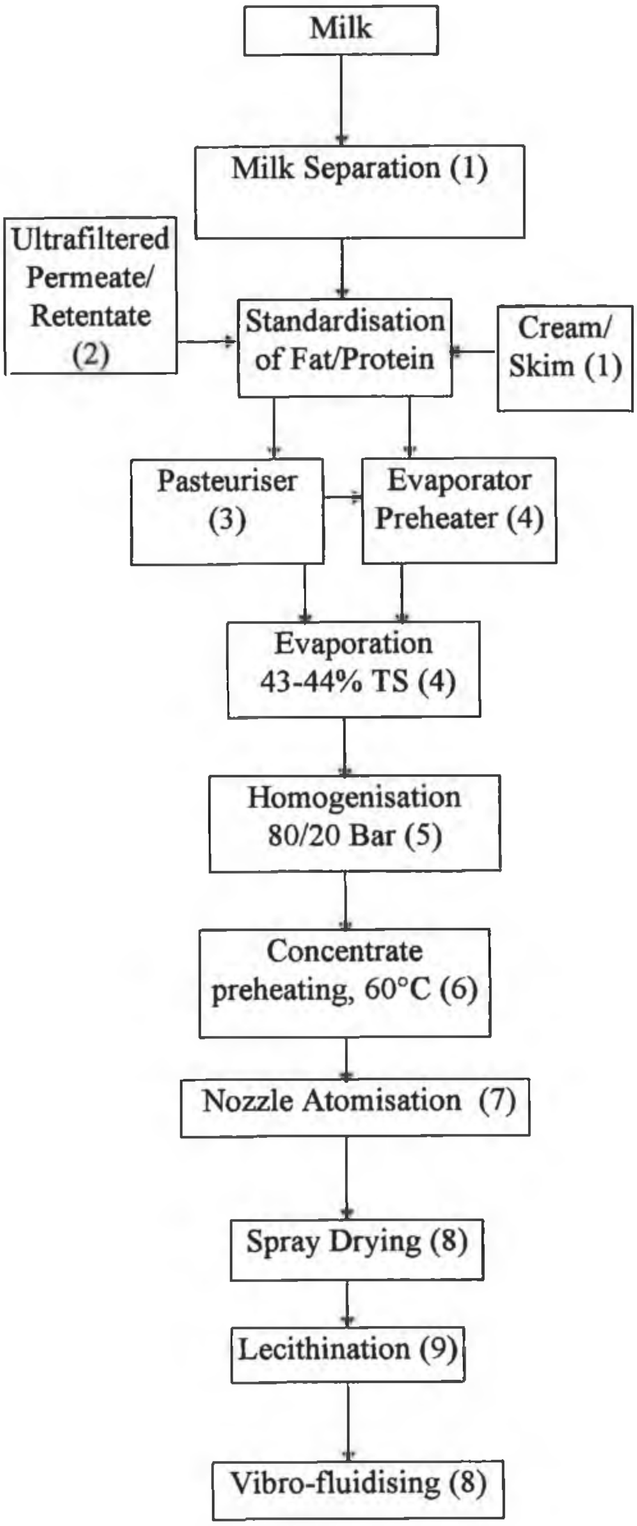
A Malvern Mastersizer (Malvern Instruments, Malvern, U.K.) was used to estimate the particle size distribution profiles for coffee-stable and coffee-unstable WMP. Sieve fractions were also prepared using a Sieve Shaker (Endecotts Octagon 200, London, UK) or an Alpine Jet sieve (APV Co. Ltd., Essex, UK). Using the Sieve Shaker, 100 g of WMP was sieved for 5 min and in the case of the Alpine Jet Sieve 20 g of WMP was sieved over a 3 min interval. The sieves were 45 µ, 106 µ, 160 µ and 250 µ in mesh size (Haver and Boeker - DIN 4188 sieves). Powder remaining on the sieves was weighed and 2.0g of each fraction was used in the coffee-test to determine the effect of particle size on coffee-stability (Teehan and Kelly, 1996b).

7.1.3 Pilot-scale production of instant WMP

The effect of the following variables on instant WMP powder characteristics and coffee-stability was investigated:

1. Preheat treatments, (a) 94°C x 30s (evaporator), (b) 72°C x 2 min (Evaporator), (c) combination heating 85°C x 2min (pasteuriser) followed by 94°C x 10s (evaporator); (d) 85°C x 2min (pasteuriser), cool (10°C), 94°C x 10s (evaporator);
2. Fat percentage of 24%, 26%, 28% and 30% in the powder;
3. Protein percentage of 25%, 28% and 31% in the powder;
4. Lecithination of 26% and 30% fat powders at 0.2% (powder basis);
5. Nozzle combinations (No: orifice x core): (a) single nozzle: 56x21, (b) triple nozzle combination: 67x20 (c) triple nozzle combination: 69x20. (Spraying Systems Ltd., Surrey, England).

Preheat treatment was generally undertaken as an in-line step (Niro-direct contact heater) before entry of milk into the first calandria of the evaporator. The preheater (Figure 7-4) consisted of an indirect heater which elevated the temperature from 4°C to 65°C. Using a combination of recovered flash vapour (infusion heating) and direct steam injection, the milk was heated from 65°C to the final temperature required. Additional heating was occasionally carried out in a regular plate heat exchanger (pasteuriser) when combination heating experiments were carried out (Teehan and Kelly, 1996b).



1. Westfalia Separator MSD 50-01-071, Germany.
2. Plate and Frame UF-36, DDS, Silkeborg, Denmark, (Membrane GR61PP Molecular cut off size: 20,000Da.)
3. Pasteuriser-Eurocal 28MP Ernst P. Fischer, Ebriechdorf, Austria.
4. Niro 3-Effect falling-film evaporator with thermal compression , Soeborg, Denmark.
5. Niro SOAVI NS2066P, Parma, Italy. 2-stage Homogeniser.
6. Niro Consistator 22A, Series 21, Soeborg, Denmark.
7. Niro DPS Gas dispenser nozzles, Soeborg, Denmark.
8. Niro TFD-0025-N, Soeborg, Denmark. C Evaporation capacity 100kgs dry matter/hr.
9. Niro, Soeborg, Denmark (Lecithin: Butter Oil; 33% :66%)

Figure 7-2. Schematic for the experimental production of lecithinated WMP at Moorepark Technology Ltd., Fermoy, Co. Cork.

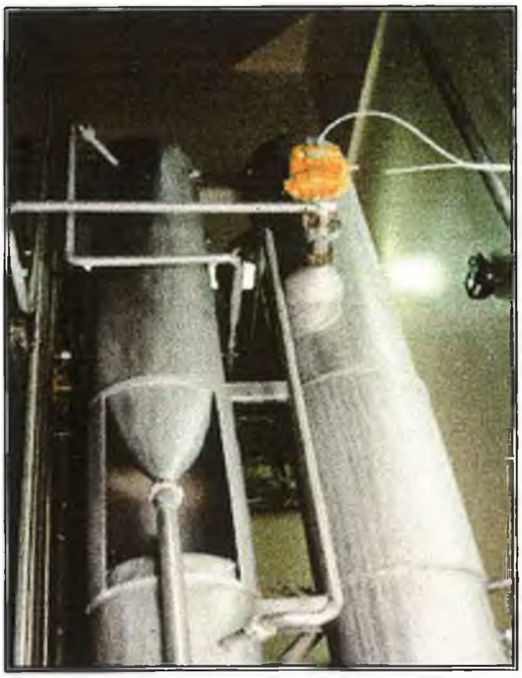


Figure 7-3. Heat-regeneration section of the evaporator in Moorepark Technology Ltd., Fermoy, Co. Cork.

Figure 7-4. Preheating and holding section in the evaporator at Moorepark Technology Ltd., Fermoy, Co. Cork, Ireland.

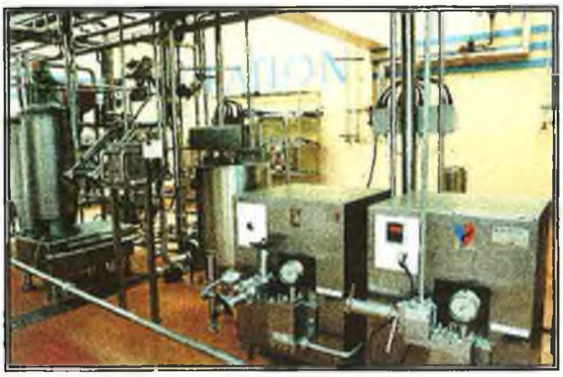


Figure 7-5. Homogenizer and High Pressure Pump section of Moorepark Technology Ltd., Fermoy, Co. Cork, Ireland

Figure 7-6. Vibrofluidiser, Moorepark Technology Ltd., Fermoy, Co. Cork, Ireland.

A schematic for the production of instant WMP is shown in Figure 7-2. Fresh milk was supplied from Dairygold Co-op, Mallow, Co. Cork and its composition analysed (Milkoscan 133B Foss Electric, Denmark). The milk was separated at 45°C and the cream or skim was used to standardise the milk to the required fat level. Standardisation of the protein level was achieved by addition of ultrafiltration permeate or retentate prepared from the same milk after cream separation. A number of individual preheat temperatures and combination heating/cooling/heating regimes were evaluated using the pasteuriser and the Direct Contact Heating system of the Niro evaporator (Niro 3-effect falling-film evaporator with thermal compression) in Moorepark Technology Ltd. (MTL) (Figure 7-3 and Figure 7-4). The milk was concentrated to 43-44% T.S. (w/w) (% T.S. measured using Labwave 9000™ CEM Corporation, Mattews, North Carolina 28106, USA) in the Niro evaporator and heated to 60-65°C using an in-line scraped-surface heat exchanger. Following homogenisation in two stages at 80/20 bar (Figure 7-5), the concentrate was delivered at 2 bar to the high pressure pump and nozzle atomised at 180-220 bar. Spray drying was carried out in the Tall-Form drier at MTL, with vibro-fluidising system (Figure 7-6). Lecithin was applied using butter oil (55-60°C) (Anhydrous milkfat, Dairygold, Cork, Ireland) as a carrier (Figure 7-6).

Table 7-1. Evaporator and Spray-Dryer Operating Conditions.

Processing Parameter	Value
Concentrate total solids	43 % (w/w)
Concentrate heater	60°C
Spray Drier	
Inlet temperature (chamber)	195°C
Outlet temperature (chamber)	70°C
Temperature fluidised bed No.1	60°C
Temperature fluidised bed No. 2	30°C

7.1.4 Small scale production of concentrate

Sixty litres of milk was standardised to produce instant whole milk powder with a fat content of 26%. The milk was preheated using the indirect heating section of an APV heat exchanger (UHT-Pasilac, Silkeborg, Denmark) (flowrate=150 l/h), and concentrated in a single-stage evaporator (Type F1-Lab 3 (evaporation capacity 45 Kgs/h) Anhydro, Copenhagen, Denmark). 5kg batches of concentrate were adjusted to 43, 44 and 45% TS (% TS measured using Labwave 9000™ CEM Corporation, Mattews, North Carolina 28106, USA) and homogenised in a two stage homogeniser (APV-Gaulin D2400, Lübeck, Germany) at 75/25 bar. A control was prepared at 43% TS and homogenised at 80/20 bar for comparison. The homogenised concentrate was heated to 60°C in a batch pasteuriser. Samples of concentrate, homogenised concentrate and heated homogenised concentrate were tested for coffee-stability. Sufficient concentrate was added to the coffee to yield a final milk solids of 2% in the coffee (Appendix C).

7.1.5 Fat-filled powder

Whole milk powder was produced using an alternative method (Hols and van Mil, 1991). Whole milk was separated into skim-milk and cream using the system in Figure 7-2. The skim-milk was concentrated to 40% TS. The % fat in the cream was determined according to the Irish standard described by the Institute of Industrial Research and Standards (1968). Cream was heat-treated to 90 °C x 30s and cooled to 45°C. The cream and skim concentrate (~ 40% TS) were recombined to yield the % fat powder required (i.e. 13% fat in the concentrate) for a 26%. After confirmation of the desired fat content, the % TS was adjusted to 43% and the concentrate homogenised and spray-dried under the conditions for instant WMP (Table 1). Similarly butter oil (Dairygold, Mallow, Co. Cork. Ireland) at 45°C was recombined with skim concentrate using a Silverson Mixer (Model GX -Immersion Type, Waterside, Chesham, Bucks, England) and dried as for instant WMP. An instant WMP control containing 26 % fat was prepared from the same milk.

7.1.6 Evaluation of powder properties

7.1.6.1 % Moisture

During production, the % moisture was measured using the Dickey John (GAC III, Dickey John Corporation, USA). The oven method (A/S Niro Atomizer, 1978a) was used to confirm the results: 1.0g of powder was weighed into an oven dish and allowed to dry for 4.5 hours at 102°C (Harvard/Lte Vulcan Laboratory Oven, Gelman Filtration systems, Oldham, Lancashire, UK). The dishes were cooled in a dessicator before weighing. The difference between the initial and the dried weights of the sample is defined as the % moisture.

7.1.6.2 Bulk Density

100 g of powder was weighed into the glass graduated cylinder and tapped using the Engelsmann Ludwigshafen tapping device (Germany). The bulk density was recorded as Kg/m^3 at 100 and 625 taps (IDF, 1995).

7.1.6.3 Wettability

10g of instant WMP was evenly spread on the surface of distilled water at 25°C, using the apparatus shown in Figure 7-7. The time taken for all the particles of the sample to become wetted was observed and recorded (IDF, 1979).

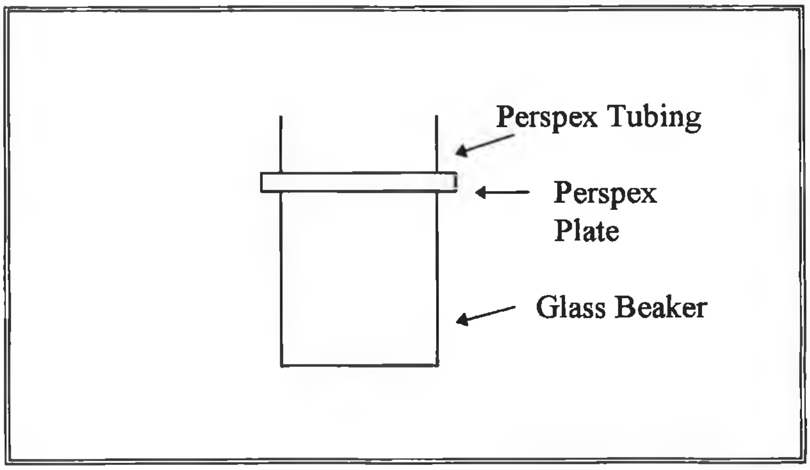


Figure 7-7 Apparatus for determination of wettabilty of instant WMP (IDF, 1979).

7.1.6.4 Dispersibility

In order to determine the ability of milk powder to disperse in water at 80°C, 26g of WMP was added to 200ml water at 80°C and stirred with a fork for approximately 30 turns over a 15 s period. The solution was then immediately passed through a 160 μ wire guage sieve of diameter 50mm. The number of lumps was then compared with the dispersibilty standard scale. On a scale ranging from 1-9 the high values, indicate poor powder dispersibility. (The chart and method was sourced from industry and therefore cannot be referenced for reasons of confidentiality).

7.1.6.5 Solubility Index

According to the ADMI method, 13g of WMP was added to 100ml of water at 24°C. This was mixed in a Solubility Index mixer (Labinco B.V., Breda, The Netherlands) at 3600rpm for 90sec. After standing for 15min, the solution was transferred to 2 solubility index tubes (50ml) and centrifuged (Funke Gerber GmbH, Berlin, Germany) at 164 g (g force gravity) for 5 min. Using a vacuum pump the sediment-free liquid was removed from above the 5ml mark and the tube refilled with 24°C water up to the 50ml mark. The sediment was dispersed into the water phase and re-centrifuged. The sediment volume was recorded per 100ml. (ADMI, 1971a)

7.1.6.6 Particle Density, Occluded air and Interstitial air

The particle density (expressed as mass in g having a total volume of 1 cm^3) was calculated by measuring the air-free volume of a known weight of powder using the Accupyc 1330 (Micromeritics, USA). Using this data and the bulk density (100 taps) of the powder, the volume of occluded air and interstitial air was derived (A/S Niro Atomizer, 1978b). The occluded air represents the volume of air within powder particles, while the interstitial air refers to that trapped between the particles.

7.1.6.7 Vacuum packing and CO_2 equilibration of WMP

WMP (2g) was vacuum packed into sachets, which were opened under the level of the coffee using a scalpel blade so as to prevent contact with the atmosphere. The air was removed by vacuum-packaging (Swissvac Transmatic, Transvac-Maschein AG, Luzern, Switzerland).

10g of WMP was equilibrated in a balloon of carbon dioxide overnight. The 12 x 26cm bags (vacuum-pack quality) were heat sealed on introduction of the carbon dioxide. The bags were then shaken with the carbon dioxide for 1 min and stored overnight before testing for coffee-stability. The sachets were opened under the level of the coffee, during the coffee-test

7.1.6.8 Analysis of the surface composition of WMP

Electron spectroscopy for chemical analysis (ESCA) is a technique used to analyse the surface composition of complex powder surfaces (Fäldt *et al.* 1993). Using this technique the percentage coverage of components such as protein, fat and carbohydrate can be determined. 4 samples of instant WMP (2 industrial WMPs-one coffee-stable and one coffee-unstable WMP; and 2 experimentally produced WMPs-one coffee-stable and one coffee-unstable WMP) were analysed using ESCA analysis, by YKI (Swedish Institute for Surface Chemistry), Stockholm, Sweden.

7.1.7 Evaluation of powder chemical properties

7.1.7.1 % Fat

Fat content of the powder was determined according to the Rose Gottliebe Method (IDF, 1987): 1g of powder was dispersed into 10ml distilled water at 50 °C. The fat was extracted from the powder using 2ml NH_3 -solution (35% specific gravity 0.88, BDH Laboratory Supplies, Poole, England), 10ml absolute ethanol (extra pure, Merck, Darmstadt, Germany), 25ml diethyl ether (Alkem Chemicals, Dublin, Ireland) and 25ml Petroleum ether (boiling point 40-60 °C, Alkem Chemicals, Dublin, Ireland) for the first extraction. Solutions were mixed with a vortex for 30s after addition of each solvent. For the second and final extraction, 5 ml of ethanol, 15ml diethyl ether and 15ml petroleum spirits were used. After each extraction the contents of the Rose Gottliebe tubes were centrifuged for 5 min at 600rpm and the ether (top) phase siphoned into a weighed beaker. The ether was evaporated over a boiling water bath. The beakers were placed in an oven at 102°C for 1 h after the final extraction and then weighed after cooling. The fat remaining in the beaker was weighed and calculated as % total fat of the powder.

7.1.7.2 % Free Fat

The content of free fat on the surface of milk particles was defined as the evaporation residue remaining after 15min extraction with carbon tetrachloride (BDH Laboratory Supplies, Poole, England): 10 g of WMP was added to 50ml of carbon tetrachloride and shaken for 15min (Griffin flask shaker, Griffin and George Ltd., England). This solution was filtered through Whatman No. 1 filter paper (Whatman International Ltd., Maidstone, England) and the filtrate evaporated on a hot plate. The content of surface fat was expressed as a % of the powder (A/S Niro Atomizer, 1978c).

7.1.7.3 % Protein

Total Protein in the powder was determined by a modification of the Kjedahl method. 1 g of a 10% TS solution of the WMP reconstituted at 50°C was used for the analysis (IDF, 1993). The sample was digested (Kjeltec 2020 digestor, Tecator, Perstorp Analytical Ltd., Bristol, England) with concentrated sulphuric acid (98% nitrogen free, Alkem Chemicals, Dublin, Ireland.) using potassium sulphate and copper sulphate as a catalyst (1000 Kjeltabs CX, Thompson & Capper Ltd., Cheshire, England). Organic nitrogen is converted to ammonium sulphate which is not volatile at the digestion temperature (450°C). Treatment of the digest with 40% NaOH results in the conversion of ammonium sulphate to ammonium hydroxide which is distilled (Kjeltec 1002 distillation unit, Tecator, Perstorp Analytical Ltd., Bristol, England) over into 50ml of a 4% boric acid, where it is converted into ammonium borate. The ammonium borate is then back titrated with HCl to determine the concentration of N. The % crude protein is given by the % nitrogen x 6.38.

Total Protein was also evaluated using the Leco® FP428 (Leco Instruments, USA).

7.1.7.4 Determination of lecithin

In order to evaluate the % lecithin, the fat in the WMP was initially extracted using cellulose extraction thimbles (25 mm x 80 mm) (Whatman International Ltd., Maidstone, England) petroleum ether (38 °C- 48 °C) (Alkem Chemicals Ltd., Dublin, Ireland), and a Quick-Fit Soxhlet extractor (AGB Scientific, Dublin, Ireland). 20ml of the extract was evaporated to dryness in a crucible and dried in an oven for 1hr, before charring with MgO and ashing in a muffle furnace at 800°C for 2 hrs. The ash was dissolved in 5N HCl and brought to 100ml in a volumetric flask. The phosphorus content of the solution was determined spectrophotometrically. All glassware for the analysis was acid washed in a 10% nitric acid bath overnight. The % lecithin is given by mg phosphorus/L x 0.007926. (Wewala and Baldwin, 1982).

7.1.7.5 Assessment of Heat Class-Whey Protein Nitrogen Index

The heat classification of the powder was determined by a modification of the WPNI method (ADMI, 1971b) (2.7g WMP was reconstituted in 10ml distilled water at 50°C. 1ml filtrate was added to 10ml saturated NaCl and two drops 10% HCl added, before measuring % transmission on the Hitachi U-1100 Spectrophotometer, Tokyo, Japan). See Table 7-2 for heat classification of milk powder (ADMI, 1971b).

7.1.7.6 Assessment of heat class-Heat Number reference method.

The casein and heat-denatured milk serum protein in 30ml of 10% TS solution of WMP (reconstituted at 50°C) was precipitated at a final pH of approximately 4.8 by adding acetic acid (10% w/v) and sodium acetate (1 M) solution. The total nitrogen of the filtrate and that of the original reconstituted WMP solution were determined by the Kjeldahl method (as for 7.2.5.3 % protein above). The heat number can be calculated from these results and its heat classification determined (Table 7-2) (IDF, 1982).

Table 7-2. Heat classification of milk powders

Heat Number	Heat Class	WPNI (mg WPN dried milk)
80.0 or less	Low Heat	≥6.0
80.1 to 83.0	Medium Heat	4.5 to 5.9
83.1 to 88.0	Medium-High Heat	1.5 to 4.4
88.1 or more	High Heat	≤1.4

7.1.7.7 Determination of Total Calcium in WMP

Following precipitation of a 10 % TS solution of WMP (reconstituted at 50°C) using 15% trichloroacetic acid (BDH Laboratory Supplies, Poole, England), the filtrate was analysed for calcium using atomic absorption spectroscopy (Spectra AA-20, Varian Techtron Pty. Ltd., Mulgrave, Victoria, Australia) (IDF, 1984). All glassware for the analysis was acid washed in a 10% nitric acid bath overnight.

7.1.7.8 Determination of Ionic Calcium in WMP

Total and ionic calcium levels were measured in the same WMP sample solution (10% TS). Ionic calcium was measured with an Orion Calcium electrode (ATI Orion Research Inc., Boston, USA.). The electrode was calibrated with standards of 20 mg/l and 100mg/l Ca^{++} (Calcium standard 922006, Orion Research Inc., Boston, USA). All standards and samples were kept in a water bath at 25°C, before measurement. Glassware for the analysis was acid washed in a 10% nitric acid bath overnight.

7.1.7.9 Determination of total water hardness

Total water hardness was determined according to the Standard method of the American Public Health Association (1992): 25ml of sample were initially diluted to 100ml with distilled water; 2ml of ammonia buffer and an indicator tablet were added and the mixture titrated with 0.02N EDTA until the end point was reached (purple to blue colour change).

To investigate the effect of variation in water hardness on the coffee stability of instant WMP, different dilutions were prepared using a stock solution of $MgCl_2$-$6H_2O$ and $CaCl_2$ (Greutzmacher and Bradley, 1991).

7.1.7.10 Buffering capacity

The buffering capacity of 30 ml samples of 10% TS solution of WMP at 20°C was determined by titration from the natural pH of the solution to pH 2.0 with 0.5 M HCL and then back titrated with 0.5M NaOH to pH 11.0 (Lucey, 1992). Acid or base was added in 1 ml increments by a Mettler DL21 Autotitrator (Mettler, Greifensee, Switzerland). A computer programme was developed to calculate buffer indices, according to Van Slyke (1922), for each addition of titrant and buffering curves were prepared by plotting these indices as a function of pH (Lucey, 1992). The buffering capacity (dB/dpH) was calculated by the following equation (Van Slyke, 1922):

$$\frac{dB}{dpH} = \frac{(\text{ml of acid or base added}) \times (\text{normality of acid or base})}{(\text{average volume of sample}) \times (\text{pH change produced})}$$

7.1.7.11 pH measurements

All pH measurements were carried out using a Corning 240 pH Meter. (Corning Science Products, Corning Glassworks, Corning, NY 14831, USA) .

7.1.7.12 Effect of Storage

The coffee sediment and the bulk density of the powders were after storage at 15°C and for 1 month.

7.1.7.13 Note on drawing of graphs

In the case of Figures 8-9 and 8-10 and in Appendices A, E and G, an attempt was made using Microsoft Excel software, to produce more smooth-line curves. However, manipulation of the data tended to misrepresent the findings, therefore the data has been plotted using the experimental values found.

8. AIMS AND RESULTS

8.1 Aims

The aims of this project were (a) to examine the influence of factors such as temperature, pH and water hardness, which are directly associated with the coffee environment, on the stability of instant WMP; and (b) to investigate the effects of individual steps in the production process on powder properties and subsequently coffee-stability. The results of the investigation are divided into two sections. The first sections deals with data relating to the application of the coffee sediment test i.e. measurement of flocculation associated with instability upon addition of WMP to hot coffee. Follow-up studies at pilot processing level and their effects are considered in the second section.

8.2 Part A: Application of the Coffee Sediment Test

8.2.1 Temperature of reconstitution

8.2.1.1 Solvent pre-heated before reconstitution of WMP

The stability of WMP when added to acidified water at increasing temperatures is shown in Figure 8-1. The sediment volumes for coffee-stable and unstable powders were ≤0.5 ml at temperatures up to 50°C. Above 50°C, higher sediments were produced with increasing temperature for both coffee-stable and -unstable, but sediment levels were consistently higher at all temperatures for the unstable WMP (Teehan *et al.,* 1995; Teehan and Kelly, 1996b). The % protein in the supernatant following centrifugation of the solution, decreased with increasing temperature and sediment (Figure 8-1).

8.2.1.2 Heating after reconstitution of WMP

WMP was reconstituted at 24°C in acidified water and heated to various temperatures. Sediment volumes were recorded. Both coffee-stable and coffee-unstable WMPs yielded very low sediments (<0.1ml) at temperatures up to 75°C (Figure 8-2). However, a coffee-unstable WMP produced a sediment volume of

3.0 ml at 90°C compared to <0.1 ml for a stable WMP (Teehan *et al.*, 1995; Teehan and Kelly, 1996b).

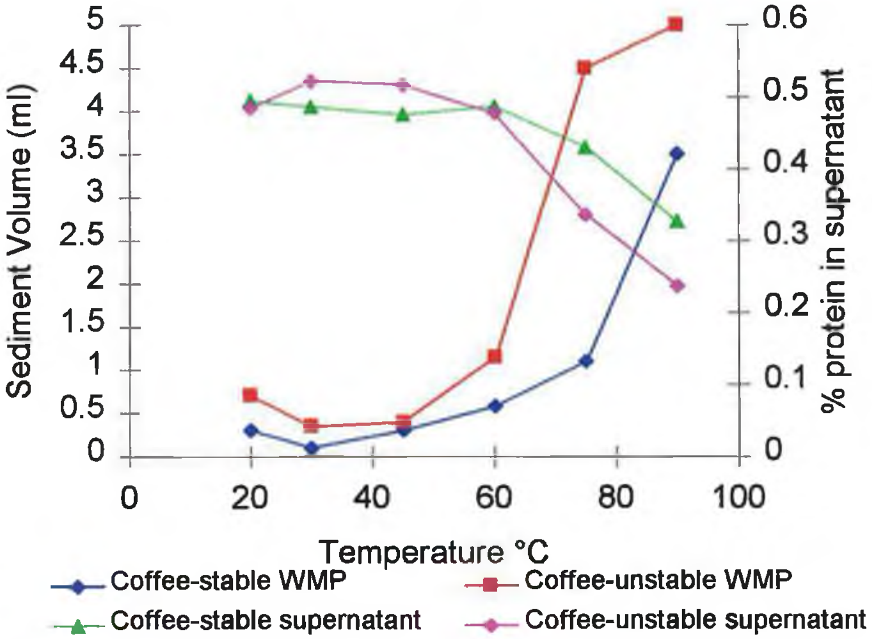


Figure 8-1. The effect of reconstitution temperature on sediment formation and supernatant protein when coffee-stable and coffee-unstable WMP were added to acidified water (final solution pH ~

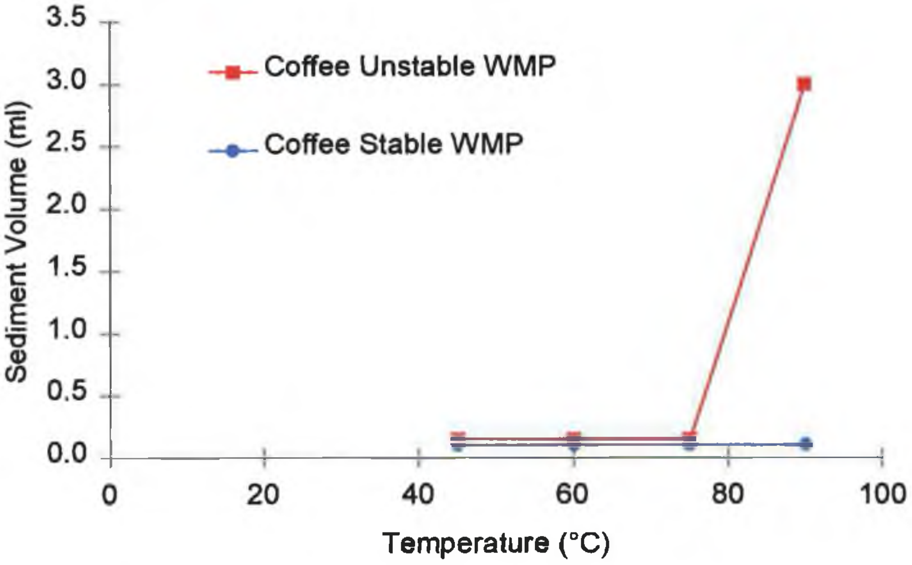


Figure 8-2 The effect of heating on sediment formation, following reconstitution of coffee-stable and coffee-unstable WMP in acidified water (final solution pH ~6.2).

8.2.2 pH

The acidity of coffee solutions challenges the stability of milk powder when reconstituted. The typical pH value of an aqueous coffee solution containing 0.8 % Nescafe Gold Blend coffee solution is 4.8 at ambient temperature. After reconstitution, the coffee/WMP mixture has a pH of ~ 6.2. Thus, milk powder exerts a strong buffering effect on the coffee/WMP solution. The sensitivity of WMP to pH on addition to coffee was simulated by replacing coffee with different levels of acid (1 M HCl) in water at 80°C.

Coffee-unstable WMP showed a gradual increase in sediment as the pH of the mixture decreased from 7.0 to 6.6 at 80°C (Figure 8-3) (Teehan *et al.*, 1995). However, sediment formation increased rapidly from < 1.0 ml to ~ 4.0 ml when pH was lowered from 6.5 to 6.2 for coffee-unstable powder compared to a maximum sediment of ~2.5 ml at the lower pHs for coffee-stable samples. The % protein in the supernatant decreased as the pH was decreased (Figure 8-3).

Acidified water did not distinguish between coffee-stable and coffee-unstable WMP at pH 6.0 or below as sediment levels reached > 5.0 ml. An alternative means of pH manipulation such as increasing the level of coffee during reconstitution gave rise to lower amounts of sediment formation (Figure 8-4) compared with those obtained with acidified water (Figure 8-3). Sediment levels in coffee increased steadily as pH was lowered, but at all times were much lower than in acidified water. For example at pH 6.2, the coffee-stable WMP formed a sediment of 0.5 ml compared with 2.0 ml for the coffee-unstable WMP. However, in acidified water at pH 6.2, the sediments for the coffee-stable and coffee-unstable WMP were 2.0 ml and 4.5 ml respectively (Teehan *et al*, 1995; Teehan and Kelly, 1996b). Therefore, it would appear that some of the constituents of coffee may contribute to stabilising the system against the adverse pH effects, as the sediments for both coffee-stable and coffee-unstable WMP are lower in reconstituted coffee than in acidified water at pH 6.2.

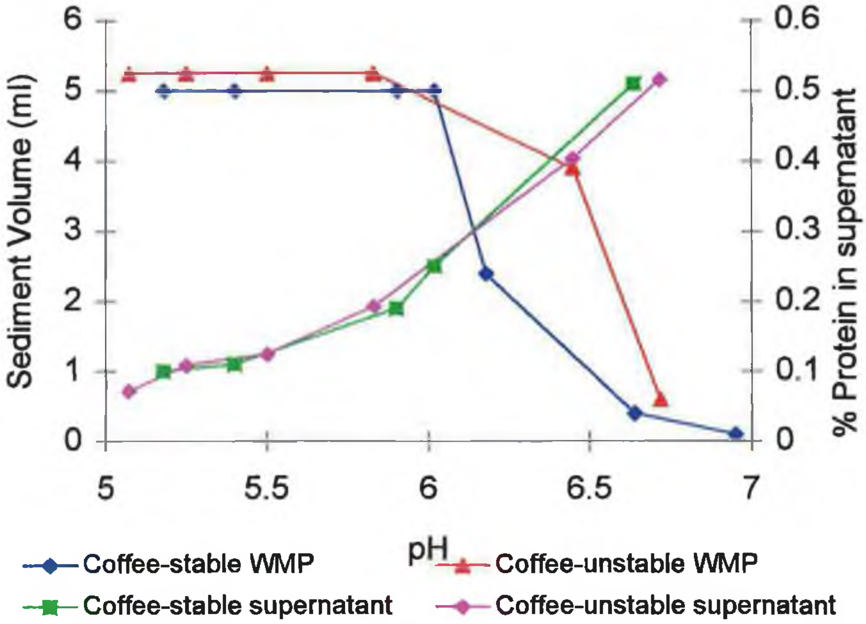


Figure 8-3 The effect of pH on sediment formation and supernatant protein for coffee-stable and coffee-unstable WMP added to acidified water at 80°C

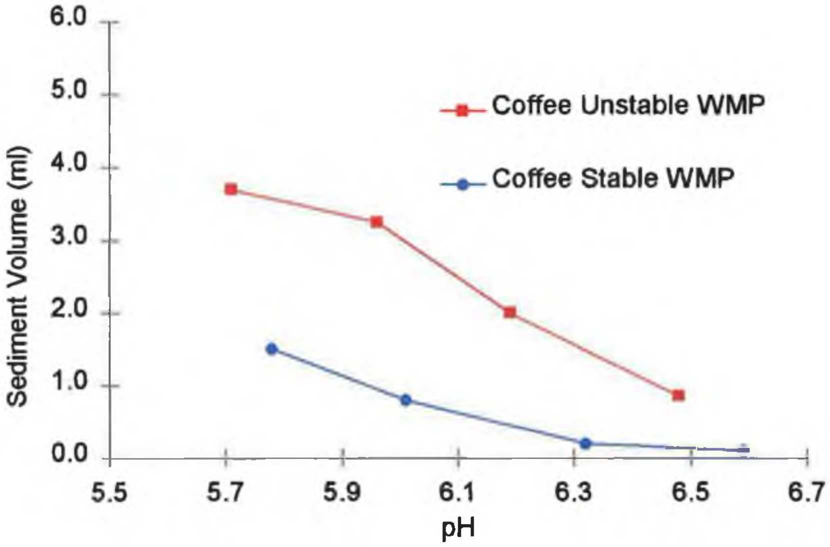


Figure 8-4 The effect of pH change (due to incremental addition of coffee) on sediment formation of coffee-stable and coffee-unstable WMP at 80°C.

8.2.3 Mechanical Effects

In comparing sediment volumes produced with different agitation speeds for 6s, it was found that a propeller speed of 550 rpm was equivalent to hand mixing using a spoon over a similar time. Increasing propeller speed from 500 to 1500 rpm decreased sediment volumes significantly (Figure 8-5). The reduction was greater for coffee-unstable WMP. On the other hand, high speed agitation at 3600 rpm for 90s in a Blender (Labinco Solubility Index Mixer) completely dispersed both stable and unstable WMP into coffee. For example, agitation at 3600 rpm for 90s reduced sediment formation of an unstable WMP from 1.0 ml to < 0.1ml (Teehan and Kelly, 1996b).

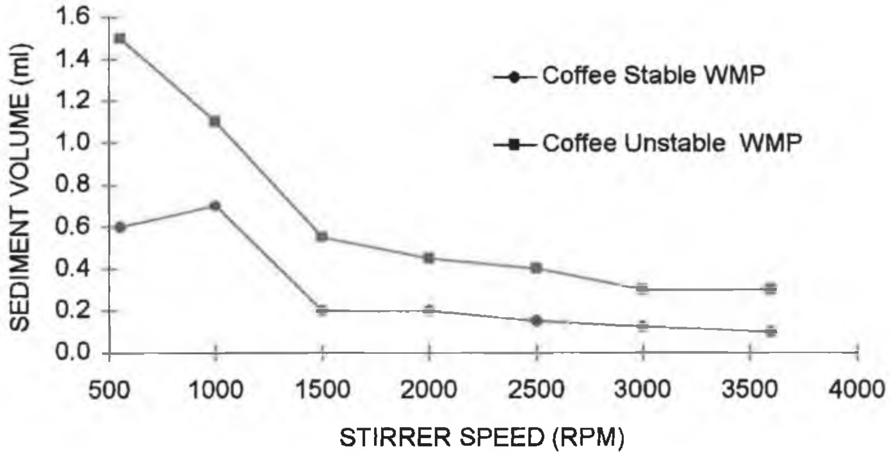


Figure 8-5 The effect of stirrer speed on sediment formation for coffee-stable and coffee-unstable WMP

8.2.4 Surface Active Agents

The objective of using surface active agents was to determine whether solvent repellent conditions prevailed at the interface between the surface of the powder particles and the coffee solution. Both ionic and non-ionic surfactants were explored. Anionic surfactants such as sodium stearyl-2-lactylate (SS-2-L) and sodium dodecyl sulphate (SDS) improved the stability of an unstable WMP in coffee. Addition of 0.4% (w/v) SDS reduced sediment values from 3.8 to

0.1ml (Table 8-1) while 1.0% (w/v) SS-2-L reduced sediment from 3.8 to 0.2ml. A shift in pH to higher values (pH 6.06-6.55) resulted from incorporation of SDS, and may have contributed to the improvement. Polysorbate 60, applied at 0.25% (w/v), reduced sediment values by 50%. Further increases in the addition level did not improve the stability of a coffee-unstable WMP (Table 8-1). When sucrose stearate-palmitate ester (SS-PE) was added (0.2% w/v) to cold water before heating to 80°C, sediment was reduced from 1.4 to 0.5ml (Table 8-1). Addition of the same levels of SS-PE to the reconstituted coffee had little effect on the sediment level of the WMP (Teehan and Kelly, 1996b). As expected, no significant change in pH was observed as a result of adding non-ionic surfactants.

Table 8-1. The effect of surfactant addition on the sediment volumes of coffee-stable and coffee-unstable WMP.

Sample Code	Surfactant	Addition % (w/v)	Sediment (ml)	pH
Control: A coffee-unstable			3.8	6.06
A	SDS	0.4	<0.1	6.55
A	SS-2-L	1.0	0.2	5.87
A	Polysorbate 60	0.25	2.0	6.14
Control: B coffee-unstable			1.4	6.19
B	SS-PE	0.2	0.45	6.25
Control: C coffee-stable			0.2	6.21
C	SDS	0.4	<0.1	6.23

8.2.5 *Prior Reconstitution of WMP*

It was necessary to establish if WMP behaved differently in terms of sediment formation when added in liquid or powder form to coffee solution. WMP samples were, therefore, dissolved at typical liquid milk solids concentration of 12.5% TS (w/v), in an aliquot of the test water prior to addition to the remaining water containing coffee. Reconstitution and subsequent addition to the coffee solution resulted in a reduction in sediment formation for both stable and unstable WMPs. Values were reduced from 0.5ml and 1.0ml, to <0.1ml (Table 8-2) (Teehan *et al*,1995; Teehan and Kelly, 1996b).

Table 8-2. Coffee sediment volumes for instant WMP reconstituted prior to addition to coffee.

Sample	Control Sediment (ml)	Reconstituted WMP Sediment (ml)
Coffee-stable WMP	0.5	<0.1
Coffee-unstable WMP	1.0	<0.1

8.2.6 Water hardness

The New Zealand Dairy Research Institute Coffee Test was adapted to incorporate minerals typically associated with water hardness, such as $CaCO_3$ and $MgCO_3$. Sediment volumes increased from 0.5 to 3.5ml per 100ml for coffee-stable WMP and from 1.5 to 4.0ml per 100ml for coffee-unstable WMP, with increasing water hardness from 0 to 260 mg/l $CaCO_3$ (Figure 8-6) (Teehan and Kelly, 1996a). The sediment volumes were higher for both coffee-stable and unstable WMPs, when added to acidified water, which was used to simulate the pH effect of coffee. Sediment volumes increased also in acidified water of increasing water hardness (0 to 260 mg/l $CaCO_3$) (Teehan and Kelly, 1996a). This suggested that constituents of the coffee contributed to stabilising the WMP in an acidic environment. The stabilising effects of potassium were investigated given that potassium accounts for 3.62-5.91 % (w/w) of Brazilian instant coffee (Angelucci, 1973). Addition of 2 g/l KCl to acidified water reduced sediment values in the 0 to 100 mg/l $CaCO_3$ range (Figure 8-7). Altering the cation form to sodium (2g/l NaCl) was also found to have a similar effect in reducing the sediment for a coffee-unstable WMP, when added to the coffee (Appendix D).

The pH effect on WMP stability (at constant water hardness, 260 mg/l $CaCO_3$) was assessed by acidification using (a) 1 M HCl or (b) increased coffee concentration (8 to 32 g/l). Increasing the coffee concentration lowered the pH and yielded much lower sediments than when the pH was altered using HCl (Figure 8-8) (Teehan and Kelly, 1996a).

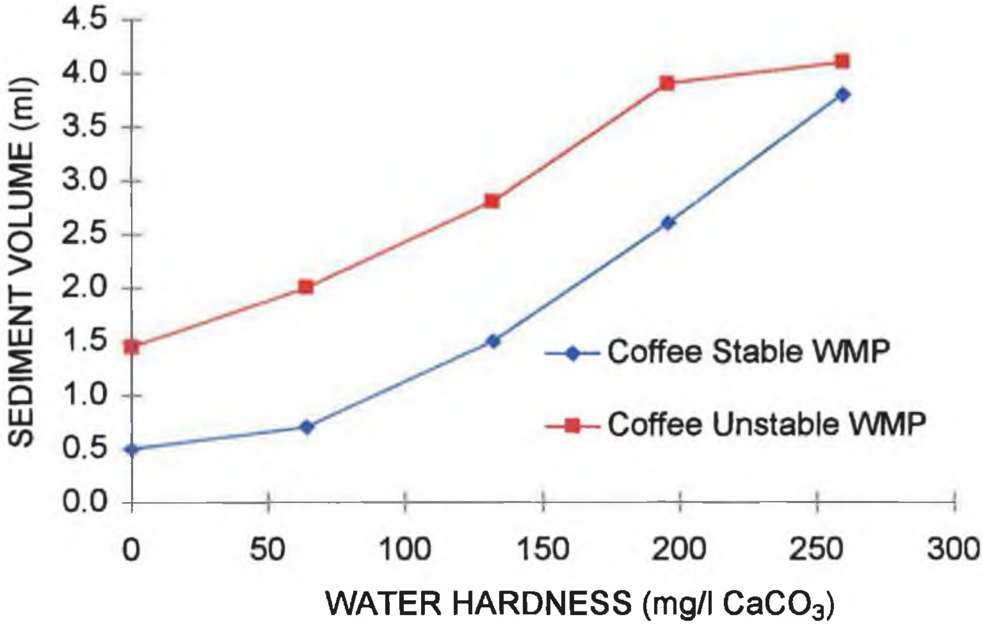


Figure 8-6 The effect of water hardness on sediment volumes for industrial samples of coffee-stable and coffee-unstable instant WMP.

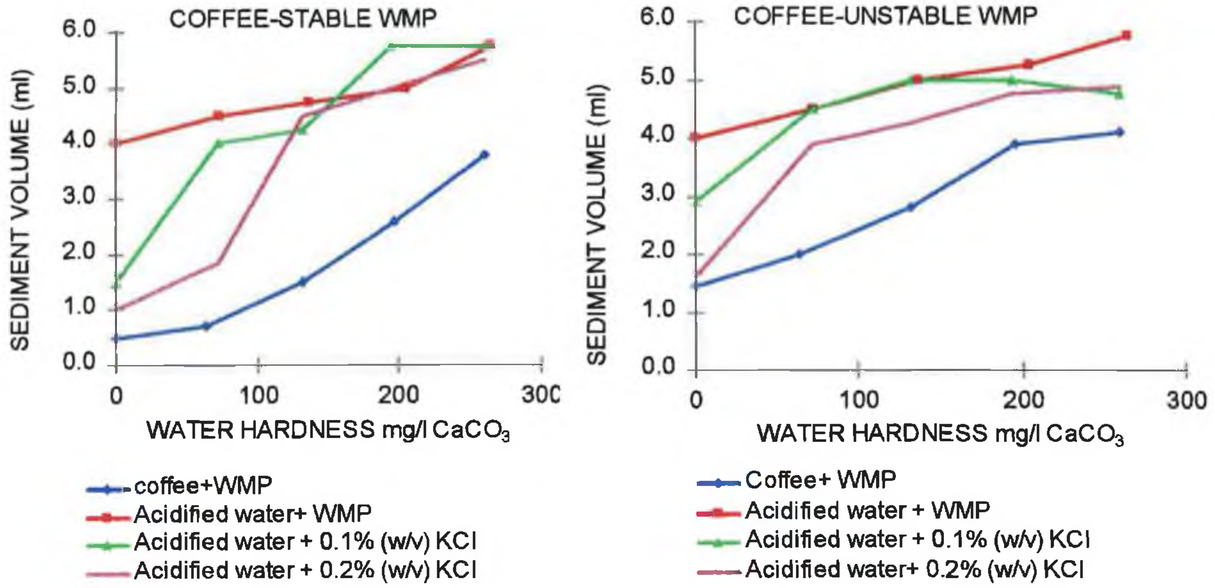


Figure 8-7 The effect of water hardness on coffee sediments for coffee-stable and coffee-unstable WMP in coffee, acidified water, acidified water + 0.1% KCl and acidified water + 0.2% KCl.

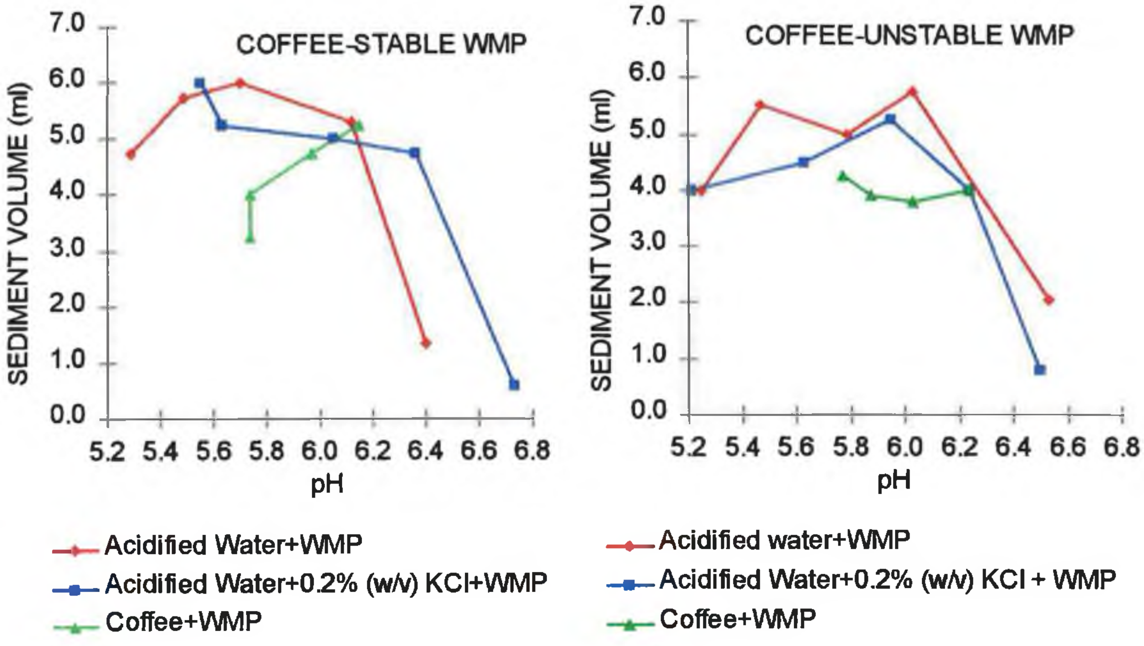


Figure 8-8 The effect of variation in pH (indirectly due to coffee concentration and directly due to HCL) on sediment volumes for coffee-stable and coffee-unstable WMP in coffee, in acidified water and acidified water + 0.2% KCL (at constant water hardness-260 mg/l $CaCO_3$.

8.2.7 Influence of physical properties of WMP on coffee-stability

8.2.7.1 Particle Size

Since the main objective of instant powder manufacture is to produce larger particles by means of agglomeration, it seemed reasonable to examine the particle size distribution of the powders in order to establish whether differences existed in their profiles. Significant differences were not found between the particle size distribution profiles, measured using the Malvern Mastersizer for experimental coffee-stable and unstable powders (n = 20 WMP samples, Appendix F). Coffee-stable and unstable WMPs with sediment values of 0.3ml and 1.2ml respectively, possessed the same proportion of particles (~71.2 %) < 163.8 μ. (163.8 μ was chosen from data as the nearest point to 160 μ). There was no correlation between the proportion of smaller particles and sedimentation values. For example, powders with sediments of 0.3 and 3.0ml had 13.11 and 13.41 % of the powder particles < 53.3 μ, respectively (Appendix E). A limitation with the estimation of particle size distribution, using the Malvern Mastersizer, is that the powder feeder system may give rise to some breakdown of fragile agglomerates. Some alternative feeding approaches were attempted without success (Teehan and Kelly, 1996b).

Coffee-stable and unstable WMPs were classified according to regular sieve fractions, with a sieve shaker in an alternative attempt to determine the effects of particle size. Contrary to expectations, the results indicated that smaller particles were more stable in coffee. However, test reproducibility was poor, possibly due to 'blinding' of the sieves by the fat-containing powder.

Size classification of experimentally produced powders (n =14), using the Alpine Jet Sieve gave more reproducible results for the amount of powder remaining on the sieve. The coffee-stable WMP had 39.5% of the particles >106 μ compared to 24.0 % for the unstable WMP. The coffee test sediments for the fractions (Table 8-3) indicated that fines <106 μ contribute to higher sediment values as once they were removed by sieving, the sediment values decreased for the coffee-unstable WMP. An overall decrease in coffee sediment volume was observed for experimental powders, as the proportion of the powder particles >160μ increased (Figure 8-9) (Teehan and Kelly, 1996b).

Table 8-3. Coffee sediment volumes for size-classified WMP and particle size distribution profiles (Alpine Jet Sieve).

Sample	Sediment (ml) Control	Sediment (ml) > 45μ	Sediment (ml) >106μ	Sediment (ml) >160μ	Sediment (ml) >250μ
A: coffee-stable	0.4	1.0	0.55	0.95	0.65
B: coffee-unstable	1.3	1.6	0.95	0.9	1.0

	% WMP >45μ	% WMP >106μ	% WMP >160μ	% WMP >250μ
A: coffee-stable	90.95	39.55	9.60	3.35
B: coffee-unstable	88.80	24.00	5.30	1.80

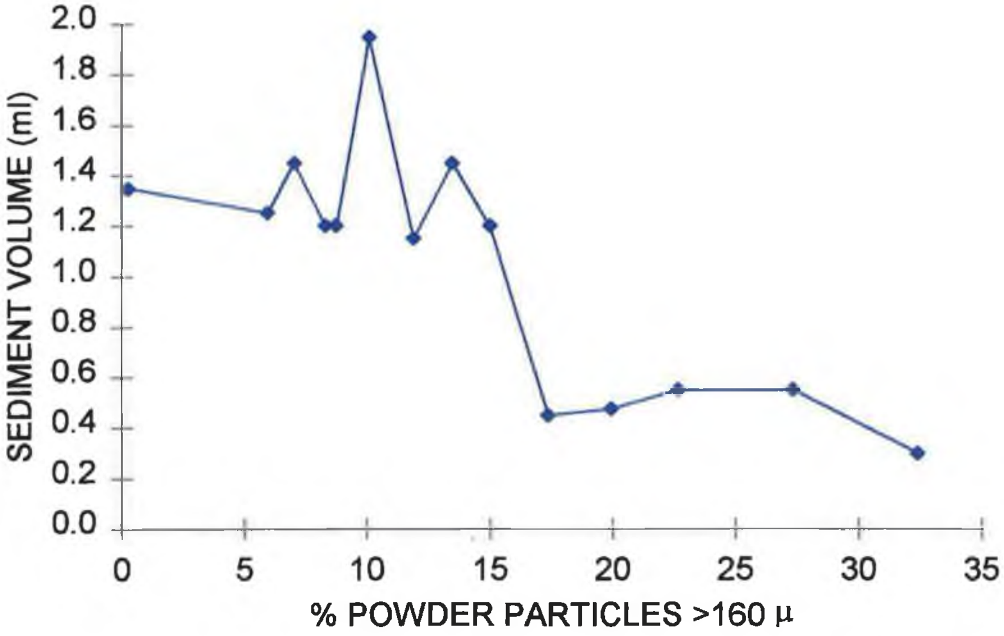


Figure 8-9. Relationship between coffee sediment and % WMP particles > 160 μ, as measured using the alpine jet sieve. Samples were prepared in MTL Ltd., Cork, Ireland.

8.2.7.2 ESCA Analysis of the surface composition of instant WMP

Electron spectroscopy for chemical analysis (ESCA) is a technique used to analyse the surface composition of complex powder surfaces. By this method the atomic concentration of each element other than hydrogen can be estimated on the powder surface. The method is very sensitive; the analysing depth is approximately 90 Å. The surface composition of 2 experimentally produced unlecithinated samples A, B and 2 lecithinated industrial samples C, D is given in Table 8-4. The analysis was performed at three different locations on the powder surface. The analysed area is a circular spot of 1.3mm in diameter. Whole milk consists of several components on the powder surfaces, but for calculation purposes it is regarded as composing of fat, protein and carbohydrate.

Sample B (coffee-unstable) had a higher coverage of fat than sample A (coffee-stable) (Table 8-4). Sample B had a lower protein coverage value while the coverage of carbohydrate on the surface was similar for both samples. The two industrial samples (C, D) had a large proportion of their surfaces covered in lecithin. Fat coverage on the surface of the two powders were 16.5% for C (coffee-stable) and 6.0% for D (coffee-unstable). However, it should be noted that it is quite difficult to distinguish between lecithin and fat, due to the fact that only 1.9 % of lecithin is pure phosphorus which is detected by the analyser. This results in a large variance in the calculated coverages when phosphorus is used to calculate the coverage of components.

Table 8-4. ESCA analysis of the surface composition of coffee-stable and coffee-unstable instant WMP

Sample	Coffee sediment (ml)	carbohydrate %	Protein %	Fat %	Lecithin %
A	0.2	17.5	28.6	53.9	-
B	1.7	18.1	21.2	60.7	-
C	0.4	21.6	5.5	16.5	56.4
D	1.45	25.8	5.9	6.0	62.3

8.2.7.3 Effect of air and carbon dioxide on coffee-stability

Air is more hydrophobic than fat and therefore may inhibit powder particles from dispersing adequately into coffee. Industrial samples of coffee-stable and coffee-unstable WMP were vacuum packed in an effort to remove all the air from the powder and then tested for coffee-stability (Table 8-5). Coffee-sediments for vacuum packed coffee-stable and coffee-unstable WMPs were high (approximately 3ml), compared with the untreated powders. The pH values for the coffee/vacuum-packed WMP solutions (pH 5.7-5.9) were lower than the control powders (~ pH 6.2). On observing the vacuum packed WMP being added to the coffee, it was noted that the powder formed clumps which sank to the bottom of the beaker and did not seem to disperse into the coffee.

Carbon dioxide is soluble in water and if the concentration of CO_2 in the air in the WMP was higher, it was speculated that the WMP may disperse more readily into the coffee and thus contribute to better coffee stability. Samples of both coffee-stable and unstable WMP were equilibrated with CO_2 overnight. The results are compared with the control in Table 8-6. Samples G, H and J showed a slight reduction in coffee sediment volumes; however the sediments for samples I, K and L marginally increased. Therefore, it is difficult to determine if the treatment with CO_2 favourably or adversely affected the coffee-stability of the sample. For all treated samples, the pH of the combined coffee and WMP solution decreased compared to the corresponding control.

Table 8-5. The effect of vacuum-packing on coffee-sediment volumes and pH

Sample	Control Sediment (ml)	Vacuum-packed Sediment (ml)	Control pH	Vacuum-packed pH
A	1.4	3.0	6.26	5.78
B	1.7	2.8	6.24	5.94
C	0.6	2.5	6.23	5.95
D	0.65	3.25	6.21	5.79

Table 8-6. The effect of equilibration of instant WMP in CO_2 on coffee sediment volumes and pH

Sample	Control Sediment (ml)	CO_2-WMP sediment (ml)	Control pH	CO_2-WMP pH
E	1.2	1.0	6.18	5.92
F	1.0	0.7	6.14	5.94
G	0.5	0.35	6.24	6.00
H	0.25	0.4	6.19	6.04

8.2.7.4 Particle Density, Occluded Air and Interstitial Air in instant WMP

The particle density was measured and from this the occluded air and interstitial air content of samples of coffee-stable and unstable WMP were calculated. For the industrial samples analysed, no significant correlation was found between particle density and volume of occluded air (Appendix A, Table A3). However, as the coffee-sediment volumes increased from 0 to 1ml, the volume of interstitial air also seemed to increase, suggesting that higher interstitial air values contribute to higher coffee sediments. It was also observed that above 1ml sediment, the relationship was not as obvious (Figure 8-10).

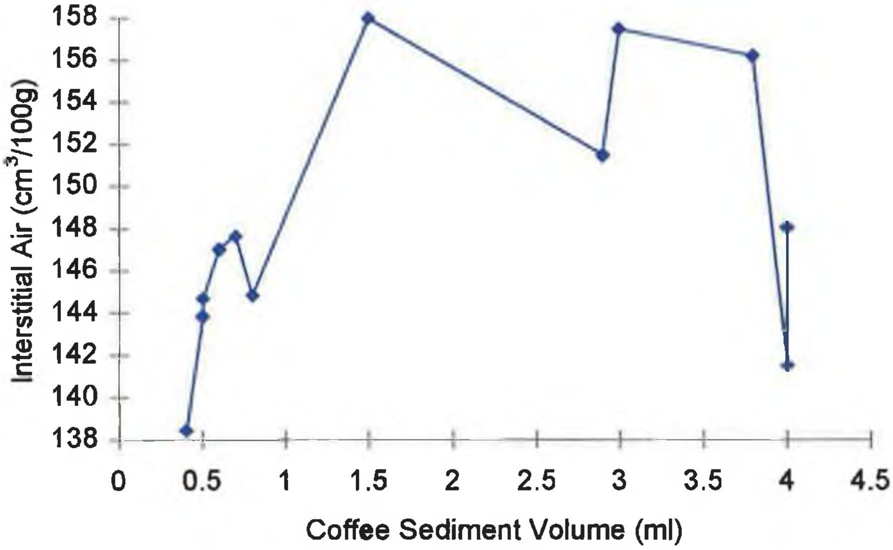


Figure 8-10. Relationship between Interstitial Air (cm^3/100g) and Coffee Sediment (ml) for industrial samples of instant WMP.

8.3 Results Part B: Investigation of processing conditions for the production of coffee-stable WMP

The manufacture of agglomerated WMP was investigated to identify the contribution of individual processing steps to the physical and physicochemical powder properties, particularly those relating to coffee stability.

8.3.1 The effect of preheat treatment on agglomerated WMP coffee stability and powder properties.

Various temperature and holding time combinations were performed on the milk feed in the pasteuriser and evaporator preheaters prior to concentration and drying. All powders may be classified (according to the ADMI WPN index, c.f. section 7.1.7.5), as medium heat, except for the powder produced from milk heat-treated at 75°C x 2 min, which was low heat. The results show that agglomerated WMPs were produced with bulk densities of 0.43 kg/m^3 or less (100taps) and typical moistures of 3.0-3.2 %, irrespective of whether preheat treatment (c), (d) or (e) is applied (Table 8-7). While combination (a) produced powder of bulk density 0.43 kg/m^3 , the coffee sediment was 0.75 ml, which is above the desired value of <0.5 ml. In an exceptional case where milk was preheated to a higher temperature (b) (97.5°C x 2 min), the bulk density (0.46 kg/m^3) and coffee sediment (1.15ml) values of the resulting powder increased slightly. Interstitial air volumes decreased as the preheat treatment was intensified (Table 8-7) (Teehan and Kelly, 1996b).

Peak buffering capacity (dB/dpH) for raw milk occurs at approximately pH 5.1 during the acidification and at pH 6.5 when the acidified milk is back titrated (Lucey, 1992). The buffering capacity curves for 10 % solutions of WMPs produced using combination (c), (d) and (e), shows a shift in the pH values at which maximum buffering occurs (Figure 8-11).

Table 8-7. The effect of preheat treatment on the physico-chemical properties of agglomerated WMP.

Heat Treatment	Coffee Sediment (ml)	WPNI (mg WPN)	Heat Number	Bulk Density (kg/m^3 100taps)	Interstitial Air cm^3/100g	Moisture %
(a) 75°C x 2 min	0.75	Low (> 7.0)	-	0.43	150.79	3.52
(b) 97.5°C x 2 min	1.15	Medium (2.3)	-	0.46	133.98	3.11
(c) Combination • 85°Cx 2min • 94°C x 10s	0.35	Medium (3.2)	87.13	0.4	169.41	3.06
(d) Combination • 85°C x 2 min • Cool (10°C) • 94°C x 10s	0.4	Medium (3.4)	86.74	0.4	168.57	3.11
(e) 94°C x 30s	0.35	Medium (4.3)	86.63	0.41	162.90	3.01

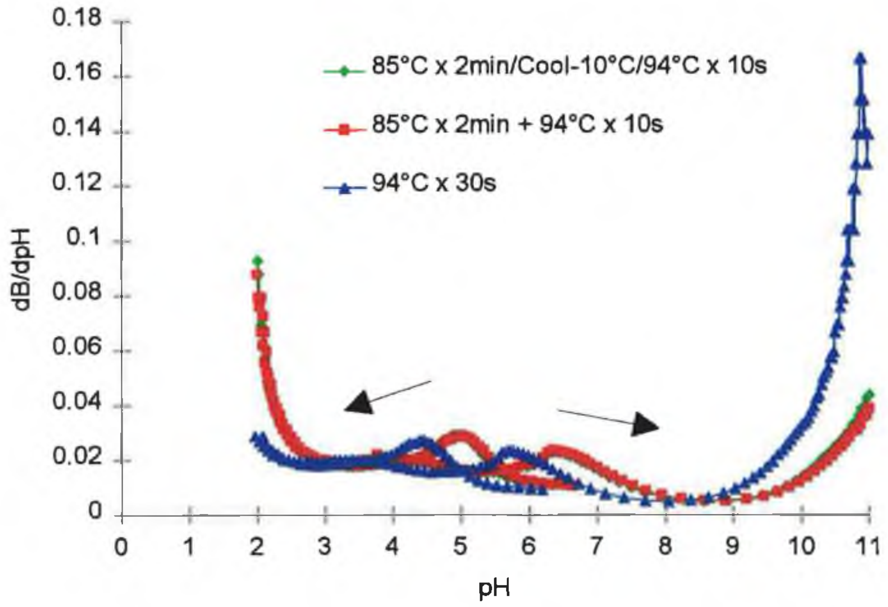


Figure 8-11. Buffering curves from initial pH (6.6) to pH 2.0 with HCl and back titrated to pH 11.0 with NaOH for 10% solutions of WMP prepared from milks preheated at 94°C x 30secs (Control - Tr1), 85°C x 2 mins + 94°C at minimum holding time (Tr2) and 85°C x 2 mins.

8.3.2 The effect of individual process steps (prior to drying) on the coffee-stability of Whole Milk Concentrate

Concentrates with varying levels of total solids produced from pre-heated milk were coffee-stable and caused no feathering. Subsequent homogenisation and heating of the concentrate to 60°C did not affect the coffee-stability of the concentrate. A slight drop in pH of the coffee and concentrate supernatant was noted for the homogenised concentrate, but pH values were restored to original values once the concentrate was heated to 60°C (Table 8-8).

Table 8-8. Coffee sediment volumes for concentrate, homogenized concentrate and heated homogenized concentrate.

% TS	Unhomogenized concentrate Sediment (ml)	pH	Homogenised Concentrate before heating (ml)	pH	Homogenised Concentrate after heating (ml)	pH	Homogenization Pressures (Bar)
43	<0.1	6.28	<0.1	6.19	<0.1	6.27	80/20-control
43	<0.1	6.28	<0.1	6.18	<0.1	6.24	75/25
44	<0.1	6.24	<0.1	6.21	<0.1	6.25	75/25
45	<0.1	6.26	<0.1	6.23	<0.1	6.24	75/25

8.3.3 The effect of variation in fat levels on the coffee stability WMP

Having adopted a preheat treatment of 94°C x 30s as a reference heat treatment (control), the effect of increasing fat level during milk standardisation so as to yield WMPs with various fat-in-powder levels was investigated. Increasing the fat from 24-30%, contributed to higher bulk densities and coffee-sediments. All powders produced were in the medium heat classification according to the WPNI and had moistures of 3.0 to 3.2%. Interstitial air volumes decreased with increasing fat content in the powder (Table 8-9). The % free fat was also found to increase with increasing fat levels in the powder, except in the case of the 30% fat powder (Appendix F, Table F2) (Teehan and Kelly, 1996b).

Table 8-9. The effect of variation in powder fat levels on coffee-stability and bulk density.

Fat %	Coffee Sediment (ml)	Moisture %	WPNI (mg WPN)	Interstitial Air Volume cm^3/100g	Bulk Density (kg/m^3 100 taps)
24.2	0.2	3.3	Medium (3.8)	147.13	0.44
25.7	0.5	3.20	Medium (3.5)	141.51	0.45
28.1	0.6	3.10	Medium (4.2)	135.96	0.46
30.9	1.25	3.11	Medium (4.8)	131.33	0.47

8.3.4 The effect of variation in milk protein content on coffee-stability

The protein content of milk was standardised to different levels by the addition of either ultrafiltration (UF) permeate or retentate, prepared from the same milk after cream separation. The fat content was maintained at the 26 % level in powder. An initial trial indicated that powders with lower protein levels were less coffee-stable. However, on repeating the trial, it was found that there were only marginal differences in the respective sediment values of 0.5, 0.35 and 0.5ml for powders of 29.3 % (Control), 34.0 % (High protein) and 25.8 % (Low protein). The volume of interstitial air was similar at ~181 cm^3/100g for the control and high protein powder, but decreased for the low protein powder (Table 8-10) (Teehan and Kelly, 1996b).

The buffering capacity curves for 10% solutions of the powders in Table 8-10 are shown in Table 8-12. As the protein level increases, the peak intensity for maximum buffering capacity increased at pH 5.0 and 6.3. The buffering capacity peaks heights were 0.026, 0.030 and 0.034 at ~ pH 5.0, and 0.021, 0.023 and 0.036 at ~ pH 6.4 for low protein, control and high protein powders respectively.

Table 8-10. The effect of variation in milk protein content on WMP properties.

Sample	Protein Leco %	Protein Kjeldahl %	Coffee Sediment (ml)	Bulk Density (kg/m^3 100taps)	Interstitial Air cm^3/100g Volume
Control	28.2	29.3	0.5	0.40	181.32
High Protein	30.8	34.0	0.35	0.40	181.04
Low Protein	24.9	25.8	0.5	0.42	157.58

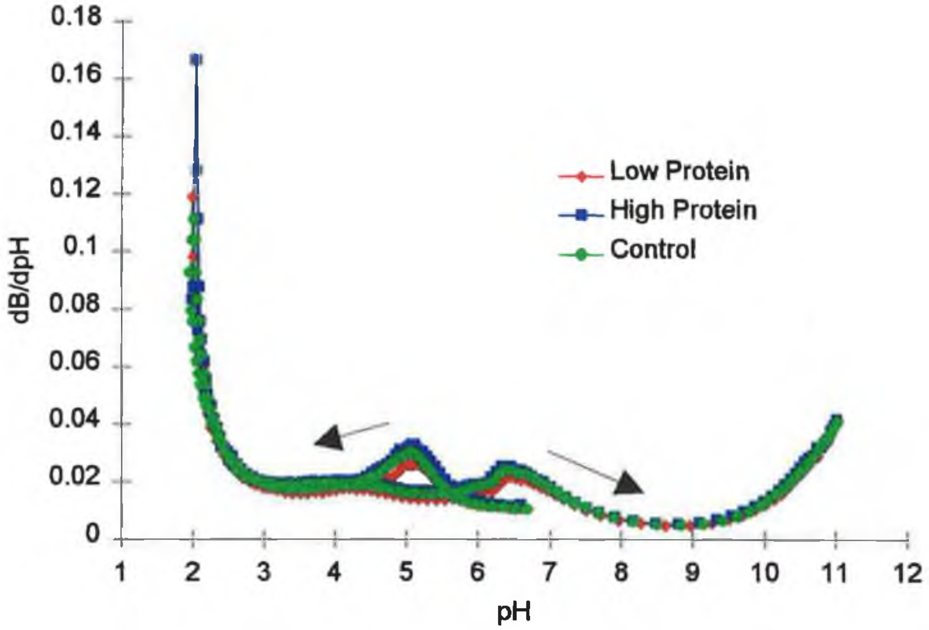


Figure 8-12 Buffering curves for 10% solutions of WMP of various protein levels titrated from the initial pH (6.6) to pH 2.0 with HCl and back titrated to pH 11.0 with NaOH.

Table 8-11. The effect of Lecithination of WMP on coffee-stability and powder properties.

Sample	Fat %	Coffee Sediment (ml)	Bulk Density kg/m^3 (100 taps)	Wettability (Seconds)	Dispersibility (Scale 1-9)	Interstitial Air Volume cm^3/100g
Control A	26.5	0.1	0.45	> 60	9	139.17
A + Lecithin	28.3	0.5	0.49	6	9	120.61
Control B	30.6	1.45	0.46	> 60	9	134.96
B + Lecithin	31.6	1.15	0.54	15	9	103.37
Control C	25.44	0.6	0.43	> 60	9	152.48
C + Lecithin	26.02	1.55	0.52	9	9	112.09

8.3.5 The effect of Lecithination on coffee stability and powder properties

Lecithination of 26% and 30% fat WMP contributed to increases in the overall fat level, powder bulk density and coffee sediments. Powder lecithin levels were 0.29% and 0.25% for samples A and B respectively. Wettability improved with lecithination but no change was recorded for dispersibility values. Interstitial air volumes decreased when the WMP was lecithinated. In order to compensate for the increase in powder fat level due to lecithination, the milk was standardised to produce a powder of 25.5% fat, to which the lecithin was applied, resulting in a final fat content of 26%. However, bulk density and coffee sediment still increased after addition of lecithin, compared to the values for the same powder before lecithination (Table 8-11) (Teehan and Kelly, 1996b).

8.3.6 The effect of nozzle combinations on powder properties

The number of nozzles and orifice/core combinations were varied to determine the effect on powder properties, based on a concentrate at 43 to 44 % T.S. (w/w). Atomisation pressure varied depending on the combination of nozzle size and number. However, for agglomeration of a 26% fat WMP, atomisation pressures of 180-220 bar were needed. Using a single large nozzle (56 x 21), agglomeration was poor and resulted in high powder bulk density (0.52 kg/m^3), low interstitial air volumes and high coffee sediment (1.9ml). Using a three-nozzle combination (67 x 20) the powders produced were lower in bulk density (0.42-0.46 kg/m^3), higher in interstitial air volumes (139-158) and had lower coffee sediment (0.1 to 0.6ml). An alternative triple nozzle combination of 69 x 20 was slightly better in terms of bulk density, interstitial air volumes and coffee-stability (Table 8-12) (Teehan and Kelly, 1996b).

Table 8-12 The effect of nozzle combination on bulk density and coffee sediment volumes.

Nozzle Combination (No. nozzles /orifice/core)	Bulk Density (kg/m^3, 100taps)	Interstitial Air cm^3/100g	Coffee Sediment Volume (ml)
1 x 56 x 21	0.52	129.0	1.9
3 x 67 x 20	0.42-0.46	139-158	0.1-0.6
3 x 69 x 20	0.40-0.41	162-169	0.3-0.4

Table 8-13 The effect of protein variation and powder storage on coffee sediment and bulk density of agglomerated WMP

Sample	Protein % (Kjeldahl)	Coffee Sediment (24hrs)	Coffee Sediment (1 month)	Bulk Density (kg/m^3, 100 taps, 24hrs)	Bulk Density (kg/m^3, 100taps 1 month)
Control	29.3	0.5	0.9	0.38	0.45
High Protein	34.0	0.35	0.9	0.38	0.44
Low Protein	25.8	0.5	0.35	0.42	0.43
Control (C)	26.44	0.6	0.85	0.43	0.47
(C) + Lecithin	25.88	1.55	1.4	0.52	0.52

8.3.7 The effect of storage on coffee stability and powder properties.

All powders were stored at 15°C and analysed after one month to investigate the effect of storage on bulk density and coffee-sediment values. Generally, these parameters increased slightly on storage (Table 8-13). However, for lecithinated powders, the bulk density and coffee sediments remained similar to original values (Teehan and Kelly, 1996b).

8.3.8 Fat-filled powders

Fat-filled powders were produced by combining cream or butter oil with skimmed milk concentrate (Table 8-14). The % fat in the control and cream-filled powders was lower than the required 26% fat due to inaccuracies in % fat determination in the concentrate. However, given that the powders are both ~ 24% fat, they can be compared. The cream-filled powder had higher bulk density and coffee sediment volume compared to the control. The butter oil-filled powder is comparable with other ~26% fat controls (e.g. 25.7% powder in Table 8-9) and produced higher sediment values. The results however are based on a single experimental trial which should be repeated to validate the results.

Table 8-14. Properties of fat-filled powders

Sample	Fat %	Coffee Sediment (ml)	WPNI (mg WPN)	Bulk Density (100 taps) (kg/m^3)	Free Fat %	Moisture %
Control	24.6	0.5	3.6	0.45	2.09	3.05
Cream	24.09	0.8	4.47	0.46	2.43	2.82
Butter Oil	25.51	0.8	4.3	0.42	1.9	2.75

9. DISCUSSION

Investigation of the coffee sediment test

Sediment Formation as a measure of coffee-stability

It is generally recognised that the challenges of temperature and low pH which prevail when coffee creamers are added to coffee combine to push the stability of such products near the limits of their colloidal stability. Failure to withstand such a test usually results in the formation of particles which either float on the surface or sink to the bottom and contribute to a visually unacceptable beverage to the consumer. It was found during this study that centrifugation of the dissolved coffee/WMP solution provided an objective means of qualifying the insoluble material or particles that arise. Sweetsur (1976) found that the coagulated particles which formed on addition of instant skim milk powder to coffee, consisted of casein and some denatured whey protein. In the present study, it was possible to observe this in another way by following the pattern of protein depletion in the supernatants, as the sediment volumes increased under conditions of varying temperature and pH (Figure 8-1 and Figure 8-3). For example, at sediments of 0.5ml, % protein in the supernatant was ~0.5 %, which was reduced to ~0.25% at higher sediment values (~5ml) (Figure 8-1).

In this study, the ionic calcium levels were higher in reconstituted solutions (10% T.S.) of commercial coffee-unstable WMP compared to coffee-stable powders (Appendix A, Table A1). Apart from pH and temperature effects, calcium has been recognised as a major destabilising factor in the heat stability of milk generally (Pyne and Mc Henry, 1955; Pyne, 1958; Morrissey, 1969; Fox and Morrissey, 1977) and more specifically in the case of coffee whiteners (Vakaleris and Sabharwal, 1972) and coffee creams (Geyer and Kessler, 1989). Studies by Geyer and Kessler (1989) demonstrated that coffee cream was less stable to feathering at higher calcium concentrations. In coffee-whitening applications, calcium effects are posed by the salts of milk or milk powder and any hardness content associated with the water used for reconstitution (see separate discussion).

In addition there would appear to be some suggestion that whey protein denaturation has an affect on coffee-stability according to Sweetsur (1976) who found that a higher level of denaturation was associated with greater coffee-instability in instant skim milk powders. Whey protein denaturation reflects the degree of heat treatment, particularly at the preheating stage, that milk is exposed to during processing. In the present study, no relationship could be established between WPNI and coffee sediment volume of industrially-produced instant WMP (Appendix A, Figure A1). However, as a measure of denaturation, WPNI is a less sensitive technique than the Heat Number Method used by Sweetsur (1976).

Appraisal of the Coffee Simulation Test

Useful information was obtained as a result of developing a model test to simulate the pH effect of coffee. By using acidified aqueous solutions, it was possible to extend the pH range about the typical value (e.g. pH 4.8) of a coffee solution. The resulting approximately curvilinear relationships based on sediment volumes and end-point pH values (acidified water/WMP solutions) in the range pH 5.0 to 7.0 for industrially produced powders highlighted interesting distinctions between coffee-stable and unstable samples. Coffee-stable samples produced lower sediments (2.0 to 0.1ml) in the pH range 6.2-7.0 compared with the unstable samples (5.0 to 0.5ml) (Figure 8-3) (Teehan *et al.*, 1995; Teehan and Kelly, 1996b).

However, when the pH was manipulated by increasing the concentration of coffee, it was interesting to note that sediment values were considerably less than at the corresponding pH values obtained with the model system. For example, a coffee-stable WMP yielded a sediment volume of 0.5ml in the coffee compared to 2.0.ml in acidified water at the endpoint pH of 6.2. The effect was even more dramatic in the case of the coffee-unstable sample where sediment values of 2.0 and 4.5ml correspond to the coffee and model tests, respectively (Teehan *et al.*, 1995; Teehan and Kelly, 1996b) (Figure 8-4). This raised a question as to why the model system should be more severe than the coffee itself. The subsequent line of investigation speculated on a potential contribution by the relatively high ash (9-10% according to Clifford, 1975) content of the coffee in countering its negative acidic effect. Potassium was reported to range from 3.62 to 5.91 % for instant coffees manufactured in Brazil (Angelucci, 1973). Addition of potassium to the model solutions succeeded in reducing considerably the sediment values of the coffee-stable powders (Teehan *et al.*, 1995; Teehan and Kelly, 1996b).

The model system was also useful to study the effect of reconstitution temperature on sediment formation. Both coffee-stable and -unstable WMP produced increasing sediment volumes at T > 50°C. Again, there was a distinct curvilinear relationship between temperature and sediment formation especially in the temperature range 59-90°C, with the coffee-stable samples displaying greater resistance to feathering at the higher temperatures (Figure 8-1). Interestingly, where WMP was reconstituted in the acidified water under ambient conditions before heating of the simulated coffee/milk solution, the sediment values were virtually undetectable (< 0.1ml) (Figure 8-2) (Teehan *et al.*, 1995; Teehan and Kelly, 1996b). This observation led to the speculation that the rapidly dissolving powder is more exposed to a type of 'acid shock' at temperatures prevailing in hot coffee and thereby results in increased sedimentation.

Buffering effects

A strong buffering effect by the WMP was evident when a final pH of 6.2 was obtained, after addition to a coffee solution with an initial pH of 4.8. Application of buffering tests typically used for milk (Lucey, 1992) proved challenging: the concentration of milk in a mixed coffee solution was at a much lower concentration (2% milk solids) compared to milk (typically 12-13% total solids) and thus made it more difficult to detect buffering peaks during acidification and back-titration with NaOH. For this reason, it was necessary to run the titrations at much higher solids (10% total milk solids) to compare buffering capacity curves for coffee-stable and -unstable WMP. No significant relationship was established between buffering capacity peaks and coffee sediment values due to conflicting evidence (Appendix B).

Physical effects associated with coffee-whitener reconstitution

A number of different approaches succeeded in distinguishing between the physical effects of whole milk powder and the thermostabilty of the milk itself in relation to coffee-stability: (a) Prior reconstitution of WMP before addition to hot coffee, reduced sediments for both coffee-stable (value 0.5ml) and coffee-unstable WMP (value 1.0ml) to < 0.1ml; (b) Increasing the mechanical activity by means of controlled stirring of the mixed coffee/WMP solution demonstrated a sizeable reduction in sediment formation, as well as providing comparable results with the rather subjective spoon stirring technique that is a feature of the coffee test; (c) The success (ionic > non-ionic) of surface active agents added to the test water of the coffee solution, in reducing sedimentation suggested a possible reduction in the water repellent forces on the surface of WMP particles e.g. free fat (Teehan and Kelly, 1996b).

Surface active agents also feature in imitation coffee-whitener formulations where their stabilising role is attributed to complex formation with protein (Greutzmacher and Bradley, 1991; Leo and Betscher, 1970) or fat emulsification (Doan, 1931; Tracy and Ruehe, 1930).

Because the number of WMP samples analysed by the ESCA technique (Fåldt *et al.*, 1993) at the Institute for Surface Chemistry (Stockholm, Sweden) was limited, some observations of a general nature were noted: (a) the percentage coverage of fat on the surface of coffee-stable powder was lower (by 6.8%) than for the unstable sample; (b) there were only slight differences in the surface content of carbohydrate and protein; (c) it was difficult to distinguish accurately between fat and lecithin; and (d) the total chemical composition of the surface of the experimentally-produced unlecithinated WMP was similar to that found by Fåldt and Sjöholm, (1996) - 55% fat, 30% protein and 15% lactose.

Influence of water hardness on coffee-stability

Sedimentation in both coffee-stable and -unstable WMP increased as water hardness was raised in coffee solutions (Teehan and Kelly, 1996b). This was consistent with the findings of Greutzmacher and Bradley (1991) who encountered similar problems during the development of whey based coffee whiteners. At constant temperature and pH, increasing the level of water hardness resulted in higher sedimentation levels and thus supported the views of Vakaleris and Sabharwal (1972) that calcium concentration is a significant additional factor associated with feathering.

Water hardness manifested itself even more severely in model test studies. Again, it was possible to demonstrate in the simulated coffee system, the positive effects of added K^+ (0.2% KCl) in stabilising WMP against feathering, especially at low levels of water hardness (Figure 8-7). Examination of the end-point pH sensitivity at high levels of water hardness resulted in very high sediments (~ 5.0ml). (Figure 8-8). One exception was a slight reduction in sediment volumes for both coffee-stable and -unstable WMP, as pH was lowered (Teehan and Kelly, 1996a). A possible explanation may be a decrease in casein micelle voluminosity with falling pH (Walstra and Jenness, 1984b) giving rise to a more dense sediment.

Influence of Particle Size on the coffee-stability of instant WMP

The Alpine Jet Sieve was used to successfully estimate particle size distribution profiles, and to also classify coffee-stable and unstable WMP according to a range of particle size fractions for further testing. The fraction containing particles > 106μ contributed to lower coffee sediment volumes for coffee-unstable WMP. Coffee-stable WMP had a greater proportion of particles >106 μ than the coffee-unstable, suggesting that a high proportion of fines <106 μ in size, contributed to the instability of WMP in coffee. Also, it was found that experimentally-produced WMP with a higher proportion of particles > 160μ, had lower coffee sediment volumes (Figure 8-9) (Teehan and Kelly, 1996b). Therefore, from these results, it appeared that WMP should have a high proportion of particles > 160μm and a low % < 106μ in size, for coffee-stability.

Baldwin and Sanderson (1973) reported that if the amount of fine particles less than 90 μ was greater than 15-20% by weight, lumps would form during reconstitution which decreased the dispersibility of WMP. The mean particle size for agglomerated WMP should not be smaller than 180 μ, the fraction below 125 μ not greater than 20% and that above 500 μ not in excess of 10% (Pisecky, 1990).

Modifying WMP atmosphere and its effect on coffee-stability

The particle density or volume of occluded air could not correlated with coffee-stability (Appendix A, Table A1). However, the industrial samples with high coffee sediments had high interstitial air values (Figure 8-10). In addition, it should be noted that for industrial WMP samples with sediment volumes > 1ml, the increase in interstitial air volume was less consistent with increased sediment volumes. On the otherhand, experimentally produced powders which had low sediments had high interstitial air volumes (See Results Section-Part B).

Comparisons between the commercial and experimental WMP samples analysed in this study, should recognise the differences in the respective processes i.e. preheating: 94°C (no holding time) v. 94°C x 30s; disc v. nozzle atomization, compact spray dryer v. tall form design and integrated fluidised bed v. standard chamber. The values for the interstitial air ranged from 138-157 cm^3/100 g for the commercial powders and 130-170 cm^3/100 g for the experimental powders. Wulff (1980) reported that reconstitution begins when interstitial air is replaced by the solvent and for a well agglomerated powder with high interstitial air volumes, the liquid can penetrate and hasten this process. Lump formation and incomplete reconstitution of milk powder is prevented by ensuring high interstitial air volumes (Wulff, 1980). Overall there is some tentative evidence from the study, suggesting that the volume of interstitial air in powder influences its ability to disperse in coffee.

It has already been shown that industrial samples of coffee-stable WMPs had a higher proportion of larger particles compared to coffee-unstable WMP (Table 8-3). The content of interstitial air is influenced by the particle size distribution, agglomeration conditions and the flowability of the powder

(Pisecky, 1978). Bigger particles do not pack as tightly and therefore there is more air between the particles. Therefore it may be expected that powders with high proportions of large particles should have both higher interstitial air values and lower coffee sediments. This was true for the limited number (n = 24) of experimentally produced powders but not for the commercial samples, but as already explained these powders are difficult to compare given that they were made under different conditions.

Vacuum packing of WMP caused coffee sediment values to increase dramatically and pH of the coffee/WMP supernatant to decrease. The decrease in pH was probably due to the inability of the WMP to disperse adequately in the coffee and buffer its acidic effect. This suggests that the air content of the instant WMP is of importance in order for the powder to disperse and dissolve in the coffee.

An attempt to displace the powder's air content by increasing the carbon dioxide concentration resulted in both slight increases and decreases in the sediment volumes of the treated powders compared to the controls. Therefore, no definite conclusions can de drawn as to the influence of carbon dioxide in the air within the powder, on the coffee stability. The slight decrease in the supernatant pH was probably due to dissolution of CO_2 into the aqueous phase producing carbonic acid.

Effect of processing parameters on coffee-stability

Attempts at replicating the performance of an industrial scale plant used for instant WMP manufacture was achieved successfully in a state-of-the-art pilot plant facility. The effects of preheating, concentration, homogenisation, concentrate heating, drying and lecithination were examined. Optimum conditions for the production of coffee-stable instant WMP were established. Using these processing parameters, the effect of variation in fat and protein content of the powder on coffee-stability was determined.

Influence of milk preheat-treatment on the coffee-stability of instant WMP

A number of preheat temperature and holding time combinations in single and multiple steps were evaluated and found to produce WMP of the required characteristics. However, a single-stage heating step involving a temperature of 94°C for 30 s proved to be satisfactory and more economical for application during subsequent pilot processing trials involving WMP (Teehan and Kelly, 1996b). The selection of an appropriate preheating regime for whole milk powder is influenced usually by the need to enhance the antioxidant potential of the milk. Temperatures above 90°C are usually applied in order to activate sulphydryl groups which are believed to contribute to better heat stability, lower solubility index and increased shelf stability of the powders (Baldwin and Ackland, 1991).

Two preheating combinations (e.g. 94°C x 30s and 85°C x 2min + 94°C x 10s) successfully produced powders with low coffee sediments and bulk densities, and higher volumes of interstitial air (Table 8-7). The high-temperature-long-time combination (97.5°C x 2 min) yielded powders with poor instant properties and coffee stability (Teehan and Kelly, 1996b). Other research has shown that higher preheat temperatures and longer holding times of the milk tend to be counter-productive in terms of producing powders with instant characteristics. The resultant higher viscosity of the concentrate contributes to an increase in powder bulk density, solubility index and % moisture (Mol, 1976; Andersen, 1986). The results of this study suggested an optimum preheat treatment of 94°C x 30s or 85°C x 2min + 94°C x 10s for the production of coffee-stable instant WMP.

The powders produced from milk preheated at 85°C x 2 min had higher buffering peaks (dB/dpH = 0.029) compared to those prepared at 94°C x 30s (dB/dpH = 0.027) (Figure 8-11). According to the WPNI and Heat Number reference method, both powders are classified as medium-high heat. The 94°C x 30s powders has a slightly higher WPNI and a lower heat number, than the 85°C x 2 min powders, suggesting that there was less protein denaturation during processing. The intensity of the buffering peak at ~ pH 5.0 was stronger with increasing heat treatment of the milk, which is in agreement with Lucey (1992).

The 85°C x 2 min powders also had buffering peaks at slightly higher pH values compared to the 94°C x 30s. Other researchers (Evenhuis and de Vries, 1956; Rose and Tessier, 1959) also found that moderate heating, e.g. pasteurisation caused small shifts in pH and buffering by the expulsion of CO_2 and precipitation of calcium phosphate, which is less soluble at high temperatures. According to Lucey (1992), the pH of maximum buffering of raw milk shifted from pH 5.1 for raw milk to pH 4.4 during acidification, for milk heated at 120°C x 10min.

Buffering capacity curves for samples of commercial instant WMP did not show significant differences between coffee-stable and unstable powders (Appendix B), thus leading to the conclusion that the coffee-stability problem is not related to buffering capacity.

Effect of process steps prior to drying on coffee-stability

Concentrate prepared from preheated milk or non-preheated milk was coffee-stable (sediment volumes $\leq$ 0.1ml). Subsequent homogenisation of these concentrates did not effect the stability of the concentrate in coffee. Heating of homogenised concentrates did not effect sediment values. Dilution of the concentrates to 20% TS before addition to the coffee did not alter the coffee-stability (Appendix C, Table C3). Therefore, it would seem that the individual process steps of preheat-treatment, concentration and homogenisation of the milk, have little influence on coffee stability of concentrate as such and that the most critical part of the process is the drying of the milk itself. This result strengthens earlier investigations (c.f. section 8.2.5-Prior reconstitution) that distinguish between coffee stability of milk in liquid and powder forms. However the effects of some individual steps e.g. preheating, influences indirectly coffee-stability and 'instant' characteristics for the reasons as already outlined in relation to optimum agglomeration.

Influence of atomisation conditions on instant WMP properties and coffee-stability

An atomisation pressure in the region of 180-200 bar with a three nozzle combination of 20 x 69 orifice/core was found to be most effective in producing agglomerated WMP with high interstitial air volumes, low bulk densities and coffee sediments. Other nozzle combinations resulted in slightly lower interstitial air values, higher bulk densities and higher coffee sediment volumes (Teehan and Kelly, 1996b). In general, nozzle atomisation produces powders with higher particle densities than wheel atomisation. This contributes to high bulk densities and improved reconstitution properties, better shelf life and a reduction in stack losses of spray dryers (Boersen, 1990). Tracy *et al.* (1951) found that for a constant nozzle size, bulk density increased with increasing atomisation pressure and decreased with increasing nozzle size at constant pressure. However, concentrate viscosity influences various parameters of the drying process and physico-chemical properties of the powders as a result of influencing droplet size distribution (Andersen, 1986). Unfortunately, viscosity monitoring and control is not yet a feature of modern milk drying processing plants, as numerous situations can give rise to changes e.g. preheat temperatures, protein variation and other seasonal-based compositional changes to milk, concentrate solids, age thickening and concentrate heating. Much more research is needed in this area to determine the effects of atomisation and drying conditions on the coffee-stability of instant WMP.

Effect of lecithination of instant WMP on coffee-stability

Lecithin application to WMP during manufacture caused a dramatic increase in the powder bulk density, coffee sediments and also increased the fat level in the powder. Even though WMP was subsequently produced to the correct fat specification (26%) by compensating in advance for the contribution by lecithin, bulk densities and coffee sediment volumes remained high (Teehan and Kelly, 1996b). Lump formation observed during the test is explained by Wulff (1980), as resulting from poor agglomeration and low interstitial air values, which prevent adequate penetration of the liquid to disperse and dissolve the powder. Wettabilty improved on addition of lecithin but dispersibility values remained poor and unchanged. Lecithin was used to prevent feathering in an acid-whey-based coffee creamer powder (Greutzmacher and Bradley, 1991) and improve reconstitution properties such as wettability and dispersibility of agglomerated WMP (Pisecky, 1980). Present investigations show that there was no significant correlation between wettability or dispersibility and the coffee stability of experimentally-produced or industrial instant WMP (Appendix F and G). However, further investigation may be required to study the efficacy of the lecithin application system used in experimental trials, given that industrial samples of lecithinated WMP were coffee-stable.

Influence of variation in milk composition on the coffee-stability

Increasing the fat-in-powder levels resulted in increased bulk densities and coffee sediments. For the 24% and 26% fat containing powders, sediments were within specification (i.e. ≤ 0.5ml). However, increasing fat levels above 26% fat resulted in poor coffee-stability (Teehan and Kelly, 1996b). This may be due to free fat on the surface giving rise to water repelling forces at the interface between the powder particles and the coffee, thus preventing the WMP from dispersing adequately into the coffee. The increase in % free fat with increasing fat levels would suggest that this was the case. Free fat is defined as the amount of fat that can be extracted from food powders by a solvent in a designated time, and it is meant to represent that fat on the powder surface. However is has be shown that the free fat value also includes fat which originates from the interior of the particles. (Buma, 1971b, de Vilder *et al.*, 1977, Bucheim, 1982). ESCA analysis (Fåldt *et al.,* 1993) of these powders would be required to accurately determine surface fat values.

Increasing the protein level in the milk by addition of skim milk retentate had no significant effect on bulk density or coffee-stability of the WMP produced from it (Teehan and Kelly, 1996b). This was surprising given that increased protein levels in the concentrate tend to increase viscosity and this, in turn, influences bulk density, % moisture, solubility index and dispersibility of instant WMP (Andersen, 1986; Snoeren *et al.*, 1983). Decreasing the % protein in the milk by addition of skim milk permeate produced powder with similar properties to the control, although there was a slight decrease in interstitial air volume (Table 8-10) and % free fat (Appendix F, Table F3).

The buffering capacity peaks at pH 5.1 and pH 6.5 increased with increasing protein content in the powder. Srilaorkul *et al.,* (1989) estimated that the contribution of casein, whey proteins and milk salts to the buffering of skim milk was 36.0, 5.4 and 58.6% respectively. These values are comparable to those reported by Kirchmeier (1980b) i.e. 26.3, 8.3 and 65.1%. Therefore, it is reasonable to assume that an increase in the % protein was responsible for the increased buffering capacity, given that casein and whey proteins have a considerable influence on buffering capacity of milk.

Effect of storage

After storage of the powders at 15°C for one month, both the bulk density and the coffee sediment increased (Teehan and Kelly, 1996b). Baldwin and Ackland (1991) found that during storage for 12 months, moisture and solubility index of WMP increased and the -SH group concentration decreased probably due to oxidation. Schubert (1981) reviewed the mechanisms of particle bonding in agglomerates. Usually, more rapid drying produced agglomerates with higher bonding strengths; however, drying rates which are too high can lead to crack formation in the agglomerates and reduce their strengths. It would appear that the forces holding agglomerates together degenerate during storage, resulting in the disaggregation of larger agglomerates into smaller particles and a resultant increase in bulk density. It was speculated that this, in turn, may cause higher coffee sediments. Lecithinated WMP did not change in coffee sediment or bulk density after one month storage, suggesting that lecithin may prevent destabilisation of agglomerates during storage. The relationship between mechanical stability of agglomerates and coffee-stability may be an important property requiring further examination.

Fat-filled agglomerated powders

Powder produced from concentrated skim-milk where the fat content was standardised by addition of heat-treated cream, had slightly higher bulk density, WNPI and coffee sediment compared to instant WMP produced by the traditional process with the same milk. Hols and van Mil (1991) produced WMP by this alternative process of recombining skim milk concentrate and cream and it was comparable with WMP produced by the traditional process in terms of physical properties, flavour and product losses.

The butter oil fat-filled powder was less coffee-stable (0.8ml) than instant WMP (0.5ml), although wettability and dispersibility were similar to instant WMP (Appendix G). The fat melting point is considered most important when choosing an oil for fat-filled powders and influences wettability (Baker and Samules, 1961, Hansen, 1980) and dispersibility (Baker *et al.*, 1959). In particular, fats with lower melting points have better wettabilities (Baker *et al.*, 1959) and dispersibilities. Other research (Kelly, 1996, unpublished data) has shown that agglomerated fat-filled powders produced with coconut oil or hardened palm oil were superior in terms of bulk density and coffee-stability compared to instant WMP. The melting points of the coconut oil and hardened palm oil were ~ 28°C and ~ 43°C, respectively. The precise melting point of the butteroil used is unknown however, it was summer butter oil (Irish) which according to literature would have a melting point of 30-33°C (Keogh and Higgins, 1986). Hardened palm oil which has a higher melting point than the butter oil, yielded a more coffee-stable fat-filled powder. Therefore, the melting point of the fat would not seem to influence the coffee-stability of fat-filled powder although it does affect its instant properties.

10. CONCLUSIONS

The development of a simulation test which established a relationship between pH and sediment volume for both coffee-stable and coffee-unstable powder may prove useful when examining the behaviour of unknown samples. Although coffee creates adverse circumstances for the stability of WMP through a combination of low pH and high temperature, simulated conditions using acid would appear to be more exacting. Thus, coffee would seem to possess some stabilising factors. Potassium may well be the important coffee constituent given its beneficial effects when added in the model tests. The fact that increased mechanical agitation and prior reconstitution of WMP tend to reduce the amount of sediment formation suggest that the physical characteristics of the powder have a major bearing on coffee-stability. Forces governing the re-wetting behaviour of instant powders may be at play under conditions where acid shock could be taking place. As expected high levels of water hardness adversely influence coffee-stability.

The individual process steps of concentration, homogenisation and concentrate heating did not affect coffee-stability. Various combinations of temperature and holding time during the preheat treatment of milk affected powder properties, in particular; coffee sediments, bulk densities and interstitial air volumes. Powders produced by preheating to 94°C for 30s had good coffee stability and were characterised by low bulk density, high interstitial air volume, and a particle size distribution with a high proportion of larger particles. Instant properties were also affected by nozzle atomizer combinations. Therefore, it seems that preheating and atomisation can affect agglomeration and instant powder properties, which are critical to the coffee-stability of instant WMP. Variation in fat composition of the milk more so than protein, affected coffee-stability and instant powder properties. This was surprising, given that increased protein has previously been shown by other researchers, to adversely influence powder properties, due to increased concentrate viscosity. Further research may be desirable to investigate the relationship between fortified levels of milk protein, concentrate viscosity and instant powder properties which affect coffee-stability.

In this study, a system was established for the production of coffee-stable instant WMP. However, further research is necessary. Preliminary analysis of some samples of coffee-stable and -unstable powders showed differences in surface fat coverage as determined by ESCA. Analysis of a larger number of samples is required to validate the theory that higher surface fat coverage contributes to higher coffee sediments. Further pilot plant trials are required to determine if alteration in the milk salts and whey protein content influence the coffee-stability and properties of instant WMP. Further investigations are also required to study the efficacy of the lecithin application system and the use of different types of powdered and liquid lecithin.

Appendix A

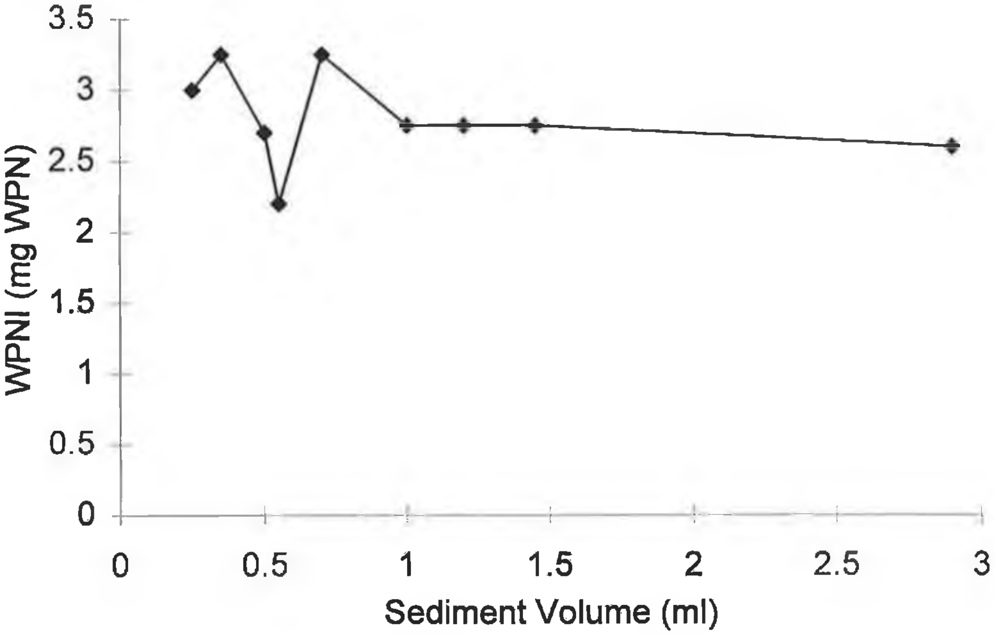


Figure A.1 Relationship between coffee sediment volume and WPNI for industrial instant WMP.

Table A-1 Coffee sediment volumes and ionic calcium levels for industrial samples of coffee-stable and coffee-unstable WMP

Sample	Coffee sediment volume (ml)	Ionic Calcium mg/l
A	4.0	80.0
B	4.0	82.4
C	0.4	69.9
D	0.5	70.9

Table A2- Statistics for Coffee Test

Sample	Sediment (ml)	x-av	x-av^2	(x-av)^2/n	Std Dev
41	0.6	0.11	0.0121	0.013	0.114018
	0.6	0.11	0.0121		
	0.3	-0.19	0.0361		
	0.4	-0.09	0.0081		
	0.5	0.01	0.0001		
	0.3	-0.19	0.0361		
	0.6	0.11	0.0121		
	0.5	0.01	0.0001		
	0.6	0.11	0.0121		
	0.5	0.01	0.0001		
Average:	0.49	Sum:	0.13		
	Sediment (ml)				
Sample	1.2	-0.025	0.000625	0.021875	0.147902
30	1.4	0.175	0.030625		
	1.2	-0.025	0.000625		
	1	-0.225	0.050625		
	1.4	0.175	0.030625		
	1.1	-0.125	0.015625		
	1.4	0.175	0.030625		
	1.1	-0.125	0.015625		
Average:	1.225	Sum:	0.175		

Table A-3. Particle density, occluded air volume and interstitial air volume for coffee-stable and coffee-unstable instant WMP.

SAMPLE	COFFEE SEDIMENT (ml)	BULK DENSITY g/cm^3	PARTICLE DENSITY g/cm^3	VOLUME OCCLUDED AIR g/cm^3	VOLUME INTERSTITIAL AIR g/cm^3
1	0.4	0.427	1.1937	5.48	138.45
2	0.5	0.405	1.1983	5.25	143.82
3	0.5	0.446	1.2109	4.31	144.69
4	0.6	0.416	1.1691	7.26	147.02
5	0.7	0.45	1.2079	4.44	147.63
6	0.8	0.438	1.1978	5.16	144.82
7	1.5	0.416	1.1229	10.84	157.86
8	2.9	0.434	1.2089	4.36	151.47
9	3.0	0.434	1.2058	4.75	157.45
10	3.8	0.44	1.1875	5.90	156.17
11	4.0	0.43	1.2088	4.47	141.49
12	4.0	0.44	1.2139	3.99	148.04

Sample Calculations
Accupyc 1330 :

Sample: No. 10

Weight: 2.0977g

Temperature: 23.4°C

Equilibration rate: 0.05 psig/min

Run	Volume (cm^3)	Density (g/cm^3)
1	1.7660	1.1878
2	1.7658	1.1879
3	1.7657	1.1880
4	1.7671	1.1871
5	1.7678	1.1866

Mean Volume: 1.7665 cm^3
Mean Particle Density: 1.1875 g/ cm^3

Standard Deviation: 0.0009cm^3
Standard Deviation: 0.0006g/cm^3

Appendix B

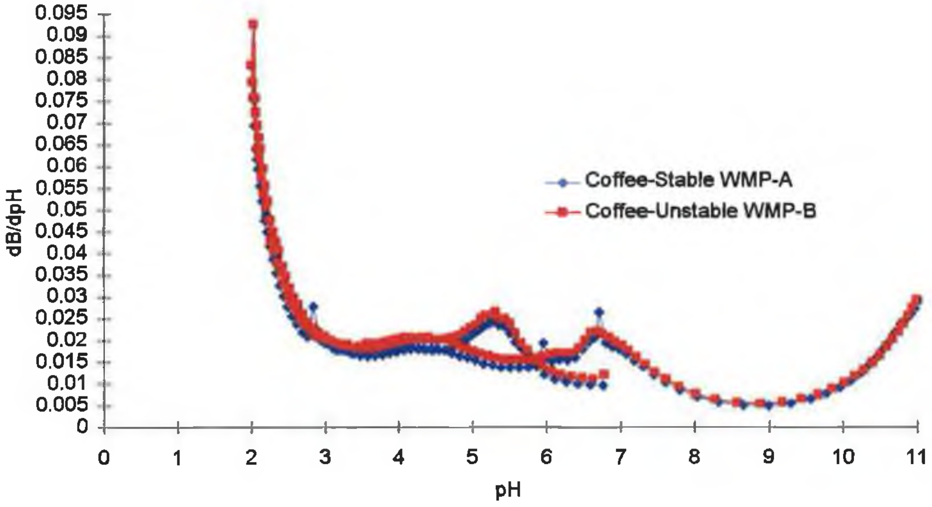


Figure B1. Buffering capacity curves for 10% coffee-stable and coffee-unstable WMP, acidified from initial (pH 6.63) to pH 2.0 with HCl and back titrated to pH 11.0 with NaOH.

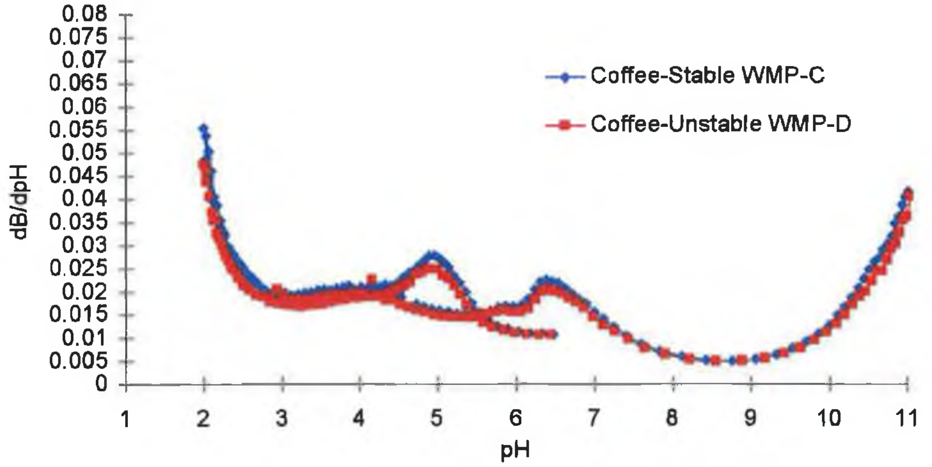


Figure B2. Buffering capacity curves for 10% coffee-stable and coffee-unstable WMP, acidified from initial (pH 6.63) to pH 2.0 with HCl and back titrated to pH 11.0 with NaOH.

Appendix C

Table C-1 Adaptation of the Coffee Test to correct for the water content of Whole Milk Concentrate

% TS	DM WMP* g	Concentrate Equivalent = (DM -WMP)/%TS x 100	Weight of added water = (102-Conc. Equivalent) (g)*
45	1.94	4.31	97.69
44	1.94	4.41	97.59
43	1.94	4.51	97.49
42	1.94	4.62	97.38
41	1.94	4.73	97.27
40	1.94	4.85	97.15

*Assume 3% moisture in 2g powder, as per coffee test.
* 102g =100ml water + 2g WMP

Table C-2 Coffee sediment for concentrate, homogenised concentrate and heated homogenised concentrate prepared from non-preheated milk

% TS	Unhomogenized concentrate		Homogenised Concentrate before heating		Homogenised Concentrate after heating		Homogenization Pressures (Bar)
	(ml)	pH	(ml)	pH	(ml)	pH	
43	0.1	6.25	0.1	6.28	<0.1	6.17	80/20-control
43	0.1	6.27	<0.1	6.30	<0.1	6.22	75/25
44	0.1	6.24	<0.1	6.28	<0.1	5.96*	75/25
45	0.1	6.25	<0.1	6.27	<0.1	5.96*	75/25

* 43% 80/20 and 75/25 did not have as effective whitening power as the 45% and 44%

Table C-3 Coffee sediment for concentrate, homogenised concentrate and heated homogenised concentrate, diluted to 20% TS and prepared from preheated milk

% TS	Unhomogenized concentrate (ml)	pH	Homogenised Concentrate before heating (ml)	pH	Homogenised Concentrate after heating (ml)	pH	Homogenization Pressures (Bar)
43	<0.1	6.20	<0.1	6.19	<0.1	6.30	80/20-control
43	<0.1	6.18	<0.1	6.20	<0.1	6.30	75/25
44	<0.1	-	<0.1	6.17	<0.1	6.29	75/25
45	<0.1	-	<0.1	6.07	<0.1	6.24	75/25

Appendix D

Table D-1 Influence of NaCl /KCl addition on coffee sediments

		Sediment		
Sample	Water Hardness (mg/l $CaCO_3$)	Control	+ 0.2% KCl	+ 0.2% NaCl
A	distilled	3.6	3.3	2.7
A	240	4.0	4.0	4.0

Appendix E

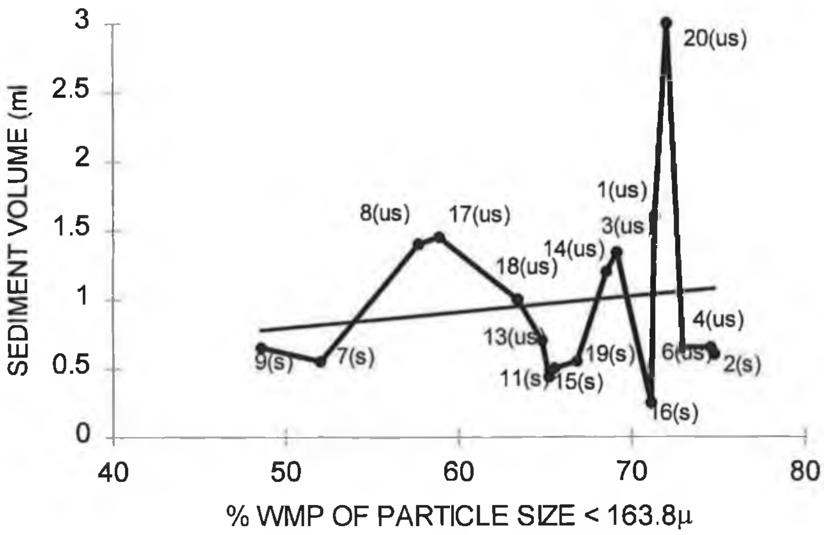


Figure E1. % of particle size WMP < 163.8 μ versus coffee sediment volume (ml) for 20 samples of commercially produced coffee stable (s) and coffee unstable WMP (us).

Table E-1 Malvern Mastersizer Particle Size Analyses and Coffee Test Sediments of some WMP samples

WMP Sample Code	**16**	**20**	**9**	**2**
Coffee-Sediment (ml)	0.3	3.0	0.6	0.6
Particle Size less than	**Proportion of particles (%)**			
53.25μm	13.11	13.41	7.98	18.78
76.32μm	24.32	25.93	14.89	31.57
103.58μm	41.28	42.52	25.97	48.24
163.77μm	71.09	72.06	48.59	74.73
258 95μm	91.93	91.58	71.6	91.92
351.44μm	97.86	97.08	83.46	97.19
555.71μm	100	99.47	94.87	99.83

Appendix F

Table F-1 Effect of preheat treatment on the physico-chemical properties of agglomerated WMP

Heat Treatment	Coffee Sediment (ml)	Bulk Density (kg/m^3 625 taps)	Wettability (seconds)	Dispersibility Scale 1-9	Calcium (mg/l Ca+)		Free Fat % powder
					Ionic	Total	
(a) 75°C x 2 min	0.75	-	>1 min	7/8	72.7	817.75	1.28
(b) 97.5°C x 2 min	1.15	-	>1 min	9	74.0	832.25	2.61
(c) Combination • 85°Cx 2min • 94°C x 10s	0.35	0.43	>1 min	8	65.8	822.50	0.99
(d) Combination • 85°C x 2 min • Cool (10°C) • 94°C x 10s	0.4	0.42	>1 min	9	71.4	802.75	1.16
(e) 94°C x 30s	0.35	0.43	>1 min	7	62.4	831.0	1.22

Table F-2 Effect of variation in powder fat levels on coffee-stability and other physico-chemical characteristics

Fat %	Coffee Sediment (ml)	Bulk Density (kg/m^3 625 taps)	Wettability (seconds)	Dispersibility Scale 1-9	Solubility Index (ml)	Free Fat % powder
24.2	0.2	0.44	> 1 min	8	<0.1	1.29
25.7	0.5	0.45	> 1 min	9	<0.1	1.58
28.1	0.6	0.46	> 1 min	9	<0.1	2.82
30.9	1.25	0.47	> 1 min	9	<0.1	1.24

Table F-3 Effect of variation in Milk Protein content on WMP properties.

Sample	Coffee Sediment (ml)	Bulk Density (kg/m^3 625 taps)	Wettability (seconds)	Dispersibility Scale 1-9	Free Fat % powder	Calcium (mg/l Ca+)	
						Ionic	Total
Control	0.5	0.42	> 1min	9	1.49	61.0	792.3
High Protein	0.35	0.42	> 1 min	8	1.47	61.0	872.3
Low Protein	0.5	0.43	> 1 min	9	1.21	58.7	733.0

Appendix G

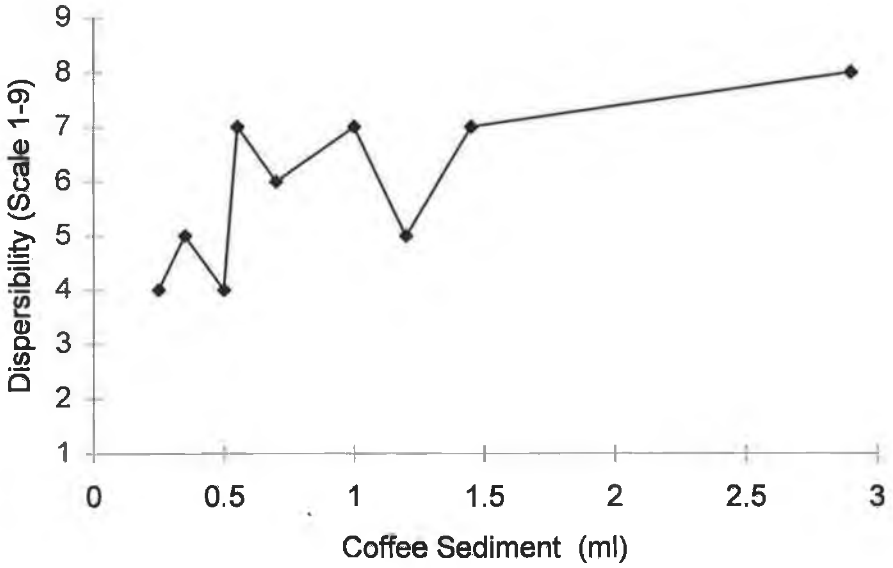


Figure G.1 Figure G.1 Relationship between coffee sediment volumes and dispersibility for industrial samples of instant WMP

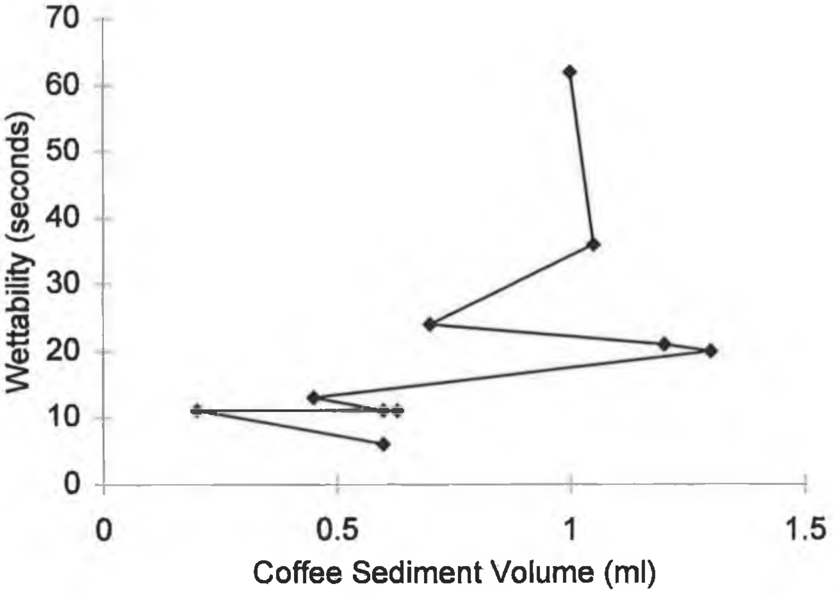


Figure G.2 Relationship between coffee sediment volume and wettability for industrial samples of instant WMP

Table G-1 Physico-chemical properties of fat-filled powders

Sample	Coffee Sediment (ml)	Dispersability Scale (1-9)	Wettability seconds
Control	0.5	8	> 1 min
Cream	0.8	9	> 1 min
Butter Oil	0.8	8	> 1 min

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