DAIRY PRODUCTS RESEARCH CENTRE
MOOREPARK

END OF PROJECT REPORT 1998: DPRC No. 14

Coffee-Stability of Dried Creamers

Dr. P.M. Kelly

The project set out to investigate the circumstances that cause milk powders and creamers to fail when added to coffee based beverages. As a result, the physical characteristics of Whole Milk powder particles, critical to coffee stability, as well as the key processing parameters, were defined, and a database to assess the potential of novel protein ingredients for use in coffee whiteners was developed.

Teagasc
The Science of Farming and Food

Coffee-Stability of Agglomerated Whole Milk Powder and other Dairy Creamer Emulsions
(Coffee-Stability of Dried Creamers)

Armis No. 4341

Project Team:
P.M. Kelly (Leader)
D.J. Oldfield
C.M. Teehan

The Dairy Products Research Centre
Moorepark, Fermoy, Co. Cork.

Teagasc acknowledges with gratitude grant aid under the Food Sub-Programme (Sub-Measure 3 (iii) - Institutional R&D) of the Operational Programme for Industrial Development. The Programme is managed by the Department of Agriculture and Food supported by EU and national funds.

ISBN: 1 901138 54 2
DPRC No. 14

Teagasc 19 Sandymount Avenue
Ballsbridge Dublin 4

EUROPEAN UNION
European Regional Development Fund

Summary and Conclusions

Coffee as a beverage is usually consumed 'black' or 'white' depending on the taste of the consumer. A variety of milk and non-dairy products in both liquid and powder forms may serve to fulfil a coffee whitening function. Most spray dried products are based either on (a) whole milk powder (b) fat-filled milk powders, or (c) the so-called imitation creamers - the latter are based on stabilised emulsions of vegetable oils which are combined with a variety of bulking agents. Whole milk powders (WMP), also known as Full Cream Milk Powders are normally manufactured to a specified fat content of either 26% or 28%. Fat-filled milk powders are a less defined grouping in fat classification terms compared to WMP. The emphasis is on replacement of milk fat with cheaper sources such as vegetable oils or animal fats during the evaporation and drying process. The resultant products resemble WMP in most physico-chemical characteristics, save whatever effects that may be brought about by the actual process of recombining and emulsification of the replacement fats into a skim milk base. Imitation creamers bear little resemblance to milk and milk powders except where a milk protein source such as sodium caseinate is used as the main fat emulsifier. From a formulation point of view, the emphasis is usually on the use of low cost ingredients. The fat content of such products are typically in the order of 30-35%. In order to satisfy the requirements of coffee stability spray dried products must firstly possess 'Instant' solubility properties i.e. satisfy the dispersability, wettability and solubility criteria normally required when fat-containing powders are added to water. The second requirement is that the creamer, on dissolving in coffee, should not coagulate or give rise to a sludge-like precipitate or sediment. Terms such as "floaters" and "sinkers" are occasionally used to describe the appearance of instability.

Hence, the objectives of this project were: (a) to investigate the circumstances that cause milk powders and creamers to fail when added to coffee based beverages; (b) to evaluate the role of processing variables in relation to their thermostabilising effects on milk during drying of coffee whiteners; and (c) to determine the role of emulsion formation on the stability of imitation creamers.

A unique requirement of this project was to establish in the first instance the technological capability of being able to produce on a pilot scale agglomerated powders with 'instant' characteristics i.e. possessing instant dissolution properties when added to aqueous solutions. Secondly, the accompanying processing steps were required to simulate closely industrial scale manufacture of whole milk powder, so that meaningful translation of the results could be made later. Furthermore, a key research strategy was to differentiate between the effects of process parameters during the two distinct phases - liquid and powder processing.
The main conclusions were as follows:

** An examination of the conditions under which the coffee stability test is carried out proved that it was possible to distinguish between the physical effects of whole milk powder (WMP) and the thermostability of the milk from which it is manufactured. Powder particle structure effects on sediment formation were isolated by (a) prior reconstitution of WMP, (b) the use of increased mechanical action during stirring, and (c) observing the role of ionic and non-ionic surface active agents.*

An adaptation of the coffee stability test indicated that more quantitative information is *provided by measuring sensitivity at different water hardness levels.*

** A simulation test was developed which established relationships between pH and sediment volume for coffee-stable and coffee-unstable WMP, and provided a framework for examining the behaviour of unknown samples. The simulation test also succeeded in focusing attention on the presence of some positive stabilising factors within coffee. It seems likely that the relatively high concentration of potassium in coffee is responsible for this.*

** An integrated pilot scale process for the manufacture of agglomerated whole milk powder which replicated industrial conditions and gave physico-chemical properties similar to commercial powders was set up. With this facility in place, it was possible to isolate and examine individual effects during the manufacture of whole milk powder by conventional approaches as well as exploring novel processing approaches.*

It was possible to interrelate the effects of changes (composition; recombination; preheat treatment; concentration; homogenisation) taking place during the 'wet' and 'dry' processing stages with the physico-chemical characteristics and coffee stability of the resulting powders.

Novel processing approaches such as (i) fat recombination in the form of cream or butteroil; (ii) protein recombination using commercial ingredients such as milk protein isolate, and (iii) casein enrichment by means of microfiltration retentates enabled additional technological information on the effects of different parameters to be derived.

** Of the milk constituents evaluated, elevated whey protein levels appeared to have the most negative effect on coffee stability. The effect was noticeable when the casein to whey protein ratio was altered by the addition of whey protein concentrate. This observation may help to explain variations in coffee stability throughout the manufacturing season, since seasonal variation in the levels of non-casein nitrogen are known to occur.*

** Concentration of milk solids was identified as a processing parameter which could be manipulated in favour of improved coffee stability. Preheat treatment became an important factor only if the coffee test is conducted under hard water conditions.*

** A greater understanding of the role of ingredient formulation was realised during the course of investigating the stability of imitation coffee whiteners. Different support emulsifiers are needed depending on whether casein or whey protein based formulations are used. The behaviour of casein-based ingredients in their micellar and non-micellar states was established.*

A novel non-milk protein ingredient - soluble wheat protein, proved superior to caseinate in stabilising imitation whiteners for coffee creaming applications, and offers powder *manufacturers a cost effective alternative in their formulations.*

** A database of technological information, produced during the evaluation of imitation coffee whitener stability, has now created an additional Food Application/Formulation Technology evaluation system whereby the potential of novel protein ingredients - produced during the course of other research projects, may be quantified meaningfully in relation to incorporation in coffee whiteners.*

Research and Results

Coffee Test

Most tests are based on the principle of combining creamers with coffee and noting some observations about the appearance and physical state of the resultant mixture. However, there are a number of adaptations depending on the concentrations of ingredients, temperature and stirring conditions. The New Zealand Coffee Stability test has gained widespread acceptance up until now, and forms the basis of a Standard Method that is being adopted by the International Dairy Federation.

Twelve instant whole milk powder (WMP) samples, defined by a dairy processor as being coffee-stable and coffee-unstable, were tested using the New Zealand Coffee-Stability test, which involves the addition of 2g of WMP to 100mL of coffee (8g/L) at 80°C, stirring using a spoon for 6 sec and measuring the sediment volume (mL) after centrifugation at 164g for 5 min. Stable WMP's yielded sediment volumes of 0.5 mL, while unstable powders yielded volumes of 1.0mL.

Initially, it was decided to assess the application of the coffee test in detail by subjecting the above samples to a more extensive investigation in which the following test parameters were broadened considerably:

- *Temperature of the coffee solution - heating before/after reconstitution*
- *pH*
- *Effects of surface active agents - ionic/non-ionic effects*
- *Mechanical effects - stirring action after blending*
- *Particle size classification of the test powders*

In order to 'stretch' some the test parameters, a simulated coffee test was developed using acidic solutions in place of coffee in order to observe performance over a wider pH range.

Nature of Sediment Formation

It is generally recognised that the challenges of temperature and low pH which prevail when '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 for the consumer.

This sediment is composed of casein and denatured whey protein. In the present study it was possible to observe sediment formation by following the progress of protein depletion in the supernatants as sediment volumes increased. 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 whey protein nitrogen index (WPNI) and sediment volume of the industrially-produced powders.

Apart from pH and temperature effects, Ca^{++} has been recognised as a major destabilising factor in the heat stability of milk generally and more specifically in the case of coffee whiteners. In coffee whitening applications, calcium effects are posed by the salts of milk or milk powder and whatever hardness content that is associated with the water used for reconstitution.

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. After addition of the whole milk powder, a strong buffering effect is evident as pH is raised to a typical end-point of 6.2. Thus, the model test allowed an extensive set of curvilinear relationships to be plotted for industrially-produced powders using sediment volumes measured over a wide range of end-point pH values. Clear distinctions (Fig.1) emerged within the end-point pH range of 6.2-7.0 between coffee-stable and coffee-unstable samples where the latter gave rise to

higher sediment volumes.

However, when pH was manipulated by increasing the concentration of coffee it was interesting to note that sedimentation was con-siderably less than at the corresponding pH values in the model system. For example, a coffee stable WMP yielded a sediment value of 0.5 ml in coffee compared to 2.0 ml in acidified water at an end-point pH of 6.2. This raised a question as to why the model test should be more severe than coffee itself. The subsequent line of investigation speculated on a potential contribution by the relatively high ash (9-10%) content of coffee in countering its negative acidic effect. Potassium is reported to range from 3.62 to 5.91% in instant Brazilian coffees. Addition of potassium to the model solutions succeeded in reducing considerably the sediment values of coffee-stable powders, and suggests that this element may be responsible for countering the acidic effect of coffee.

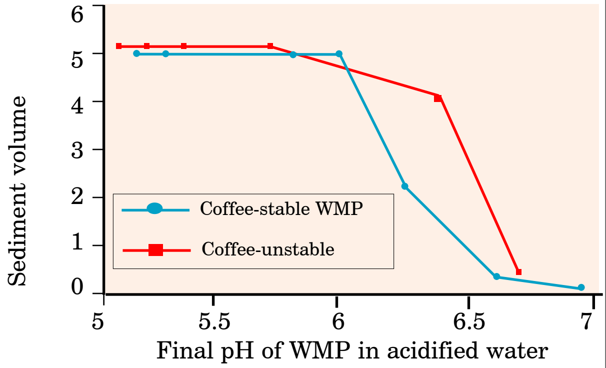


Fig. 1: Effect of final solution pH on sediment formation for coffee-stable and coffee-unstable WMP added to acidified water at 80°C in a simulated coffe test.

The model system was useful also to study the effect of reconstitution temperature on sediment formation. Both coffee-stable and coffee-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 50-90°C, with the coffee-stable samples displaying greater resistance to feathering at the higher temperatures. 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. This observation led to speculation that the rapidly dissolving powder is more exposed to a type of 'acid shock' at the temperatures prevailing in hot coffee and, thereby, results in increased sedimentation

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 thermostability 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 and coffee-unstable samples to < 0.1ml and suggests that the stability of the milk itself was not a factor in observed behaviour of the powders; (b) increasing the mechanical activity by means of controlled stirring of the mixed coffee/milk solution demonstrated a sizeable decrease in sediment formation *(Fig. 2)*, as well as providing comparable results with the rather subjective spoon stirring technique that is a feature of the coffee test; (c) the success of surface active agents (ionic > non-ionic) reduced sedimentation when added to the test water of the coffee/simulated coffee solution. *Surface active agents would be likely to reduce the negative effects of free fat on the surface of powder particles which act as water repellent forces*. Surface active agents feature also in imitation coffee whitener formulations where their stabilising role is attributed to complex formation with protein.

A limited number of commercial and experimentally-produced WMP samples were sent to Institute for Surface Chemistry (Stockholm, Sweden) for analyses of the powder particle surfaces by the ESCA technique. A number of observations of a general nature were noted:

* *the percentage coverage of fat on the surface of coffee-stable powder was lower (by 6.8%) than for the coffee-unstable sample;*
* *there were only slight differences in the surface content of carbohydrate and protein;*
* *it was difficult to distinguish accurately between fat and lecithin; and*
* *the total chemical composition of the surface of the experimentally-produced unlecithinated WMP was 55% fat, 30% protein and 15% lactose.*

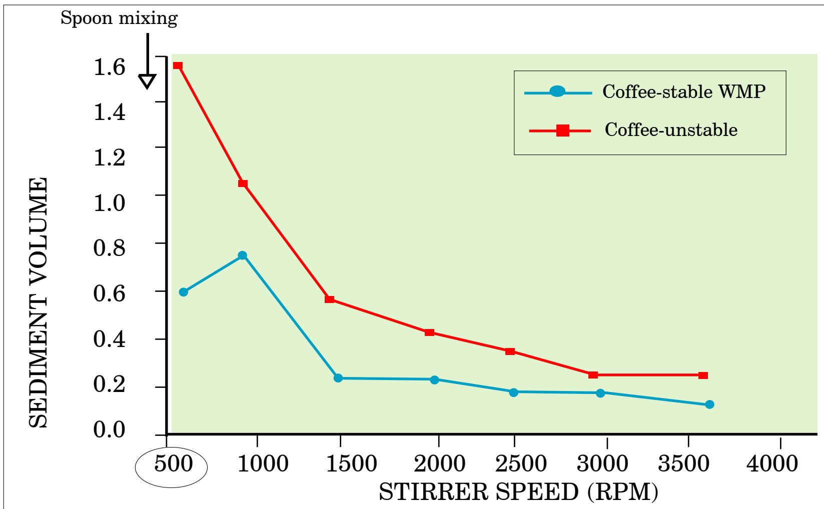


Fig. 2: Effect of stirrer speed on the coffee sediment for coffee-stable and coffee-unstable WMP. Comparison with spoon mixing shown at 500 rpm.

Influence of particle size

Coffee-stable WMP had a greater proportion of powder fines less than 106µ in size. In other words, larger powder particle sizes appear to favour coffee stability and *every effort during agglomeration should be to ensure that the proportion of particles < 106µ should be maintained as low as possible.*

Influence of water hardness on coffee stability

Sedimentation in both coffee-stable and coffee-unstable WMP increased as water hardness was raised in coffee solutions. 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.*

Processing factors

Processing variables involved in the preparation of agglomerated whole milk powders were assessed to determine their effects on coffee stability: fat and protein contents; preheating temperature; concentrate homogenisation; concentrate total solids and lecithination (singly, and in combination); alteration of the ratio of casein to whey protein; and manipulation of milk salts content. An alternative processing approach based on recombination using dairy ingredients was also investigated. The coffee stability test used to evaluate the experimentally-produced powders was extended to monitor the sensitivity of the samples to a range of different water hardness levels.

Fat content

As indicated earlier, whole milk powder is generally manufactured with a standardised 26% fat content. Having adopted a preheat treatment of 94°C x 30s for typical running conditions, the effect of increasing fat-in-powder levels from 24% to 30% during milk standardisation contributed to higher bulk densities and coffee-sediments.

Preheat temperature

A number of individual preheat temperatures and combination heating/cooling/heating regimes were evaluated using the Direct Contact Heating system of the evaporator: (a) 94°C x 30s; (b) 72ºC x 2 min; (c) combination heating involving 85°C x 2 min followed by further heating to 94°C x 1 min; (d) combination heating involving 85°C x 2min/ followed by cooling/followed by reheating to 94°C x 30 sec. In the case of the combination set-up, additional heating was provided in a conventional plate heat exchanger that was not attached to the evaporator. *Arising from this study, preheating at 94°C for 30s produced the lowest coffee sedimentation. Operating at higher preheat temperatures appeared to have a negative influence on the agglomeration characteristics and consequently an indirect effect on coffee stability.*

Later, when the coffee test was adapted to study the sensitivity of the whole milk powders to different water hardness levels, a greater distinction could be seen between the different preheat treatments *(fig. 3)*. In this case, a lower preheating regime (72°C x 30s) resulted in much lower sediment levels (0.25-2.0 mL) for water hardness levels of 0 - 350 mg/L $CaCO_3$ compared with 0.2 - 4.8mg/L $CaCO_3$ in the case of the other two. *However, too low a preheat temperature is also undesirable from a shelf-life point of view as the natural antioxidant stability system within the milk has not been activated under such conditions.* For this reason, subsequent pilot plant studies were conducted by preheating milks to 94ºC for 30s.

Protein content

The protein content of milk was standardised to different levels by the addition of either ultrafiltration (UF) permeate or retentate, which were prepared from skim milk. The fat content was held at the 26% level in powder. *Only marginal differences in sediment values of 0.5, 0.35 and 0.5mL were obtained for powders with protein contents of 28.2% (Control), 30.8% (High protein) and 24.9% (Low protein), respectively.*

Homogenisation

Concentrate homogenisation before

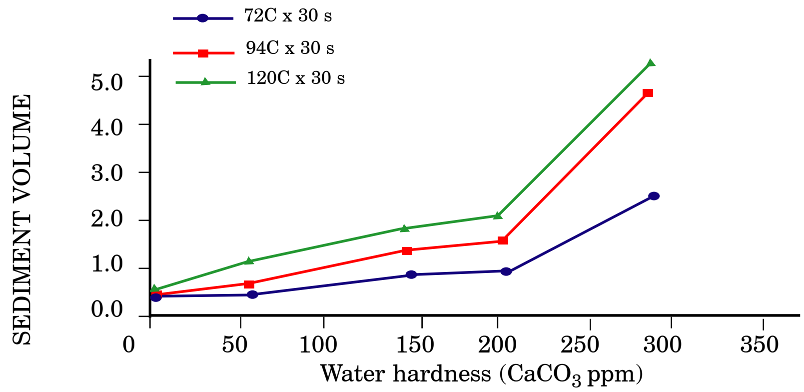


Fig. 3: The effect of preheating temperature on coffee sediment under different water hardness conditions.

drying is essential to reduce free fat levels in the subsequent powder and ensure a stable emulsion upon reconstitution. *Homogenisation pressure did not appear to be significant as sediment volumes were approximately the same for two sets of samples which were subjected to different pressures. However, a dried unhomogenised sample had a sediment volume of 0.2 - 0.4 mL higher.*

Concentrate total solids

At zero water hardness levels, there were relatively small differences (0.4-0.6mL) in sediment formation between WMP's prepared from concentrates dried at 43% and 48% total solids. However, as water hardness levels increased, the powder prepared from the lower solids concentrate was superior in terms of its lower sediment formation (e.g. 3.6 mL v. 4.4 mL at 300 mg/L $CaCO_3$). *Lecithin coating of the powder prepared from the high solids concentrate succeeded in improving coffee sediment values across the entire water hardness range by 0.2-0.5mL.*

Adjustment of the Casein/Whey protein ratio

In order to simulate the extreme changes in the ratio of casein to whey protein that occur in milk over the duration of the milk production season, manipulation of the natural ratio of the 2 protein groups in milk was carried out by addition of whey protein concentrate in one situation, and the addition of a native casein retentate enriched by microfiltration in the other. Due to limited capacity of the MF membrane relative to the size of the drying plant, it was only possible to exaggerate the casein concentration to a limited degree. *Agglomerated whole milk powder with enhanced levels of whey protein possessed poorer coffee stability compared to both the control and casein enriched powder. However, the differences were apparent at > 150 ppm water hardness.*

Soluble milk salts

In an effort to explore the role of soluble salts, 30% by volume of milk was removed by ultrafiltration (UF) in the form of permeate. Edible grade lactose was used to replace that which had permeated through the membrane during UF in an effort to maintain the overall compositional balance of fat, protein and lactose. *There appeared to be no difference between sediment formation upon addition to coffee of the modified whole milk powder when compared with the control sample.* In a variation of this experiment, a reduced-fat (17%) containing modified whole milk powder resulted in substantially lower coffee sediments especially at the higher (> 200 ppm) water hardness levels. *This suggests that the effects of fat content outweigh the more subtle changes that take place in milk salts throughout the manufacturing season.*

Reformulated whole milk powder

In order to determine whether the state of the whole milk emulsion affected coffee stability, whole milk powder was manufactured also using alternative approaches: (i) cream was added to a skim milk concentrate base before drying, and (ii) anhydrous milkfat was added to skim milk concentrate base. In both cases, the preheat treatment step (94ºC for 30s) was confined to the skim milk portion. The emulsions differed in that (ii) above relied totally on the development of a skim milk protein-based 'artificial' emulsion membrane system, while that of (i) contained the naturally-formed lipoprotein/phospholipid rich membrane material that is normally associated with cream. *The results of this study indicated that the 'filled' powders appeared to be slightly less coffee-stable than the conventionally-produced WMP control.*

An alternative approach to the formulation of whole milk concentrate before drying was adopted by reconstituting a commercial source of milk protein concentrate along with edible grade lactose. The resulting mixture of non-fat milk solids provided a base for the incorporation of cream as the source of butterfat in order to standardise composition to the typical specification of 26% fat in powder. Two experimental processing configurations were employed: in one case, reconstitution at the solids concentration (12%) of milk was adopted so that the simulated milk could be processed in the usual manner as for whole milk powder; an alternative method involved reconstitution at higher solids so as to facilitate immediate spray drying without the need of preparative steps such as preheat treatment and evaporation. *Coffee sediments comparable with conventionally-produced whole milk powder were obtained.*

Imitation Coffee Whiteners

A standard coffee whitener formulation consisting of 35% fat, 2-4% protein; emulsifiers; phosphate buffer and dried glucose syrup was used to examine the effects of a wide range of ingredients on the coffee stability of the resulting powders. The study explored the effects of different protein sources (sodium caseinate; milk protein concentrate; whey protein concentrate; soluble wheat starch; milk proteinate); emulsifiers [sucrose polyester (Sisterna); mono-/diglycerides (Dimodan PV); diacetyl tartaric esters of monoglyerides (Panodan 165)] in the presence or absence of phosphate buffer. All trials were conducted by reconstituting to 50% total solids before drying. The coffee stability used for whole milk powder was adapted in order to measure non-sedimenting particles ('floaters') which were more prevalent in the case of whiteners.

A newly-developed soluble wheat protein-based ingredient proved to have exceptional stabilising effects compared to all other protein ingredients evaluated. In fact, it was possible to reduce substantially the level of added phosphate from 2.5% to 1.0% as suggested originally by the supplier without compromising performance. Substitution of sodium caseinate with whey protein concentrate reduced coffee stability. However, some synergy was evident by using a 50/50 blend of the two proteins. An alternative emulsifier combination featuring polysorbate, sodium stearoyl-2 lactylate and lecithin when used with a reduced level of whey protein gave comparable performance with caseinate. *The study has produced a useful model for evaluating novel protein ingredients in coffee whitener applications.*

In Summary it may be concluded that:

** The physical nature of whole milk powder particles was shown to play a major role during addition to hot coffee solutions, and if anything, tended to predominate over the stability of the milk itself from which the powder was manufactured. Prior reconstitution of WMP in acidified water before heating resulted in much lower sediment volumes in contrast to direct addition in dried form, and suggested that the physico-chemical characteristics of powders are linked to the coffee stability system.*

** Reductions in sedimentation resulting from the addition of surfactants suggest that water-repellent forces are at play at the interface between powder particles and the solvent (coffee solution) during reconstitution. Perhaps, these factors affect the rate of rehydration to the point that relatively slow transition phases give rise to periods of high concentration in the protein/mineral matrix which proves deleterious for the coffee stabilising properties.*

** Those processing steps (preheating; solids concentration and whey protein content) which* *appear to affect coffee stability most likely do so by influencing the surface characteristics of powder particles.*

** The development of a simulated coffee test based on the use of acidified water proved to be more harsh than coffee. Sediment formation tends to increase as pH is lowered with a dramatic transition effect in the pH range 6.2-6.5. Manipulation of pH by varying the level of coffee addition instead of acid (as used in the simulated coffee test) during testing resulted in lower amounts of sediment formed. This gave rise to speculation that there may be some constituents within coffee which contribute to stabilising the system against the adverse effects of pH. A follow-up study confirmed that potassium supplementation reduces sediment volume in simulated coffee tests - coffee contains relatively high concentrations of potassium.*

** Useful fingerprinting techniques emerged by creating curvilinear relationships between sediment volume v. temperature, and sediment volume v. pH for commercially-produced coffee-stable and coffee-unstable powders. Such relationships provide a useful backdrop when examining the behaviour of unknown samples.*

Publications:

Kelly, P.M. and Teehan, C.M. Thermostability of coffee whiteners. *Proceedings of 3rd Food Ingredients Symposium* 9-10th October 1996

Teehan, C.M. Factors affecting the coffee stability of agglomerated whole milk powder. M.Sc. Thesis presented to *Dublin City University,* February 1997.

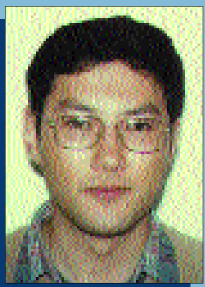
Teehan, C.M., Kelly, P.M., Devery, R. and O'Toole, A. Evaluation of test conditions during the measurement of coffee stability of instant whole milk powder. *International Journal of Dairy Technology.* **50:** 113-121, 1997.

Kelly, P.M., Oldfield, D.J. and O'Kennedy, B.T. The thermostability of imitation coffee creamers. *International Journal of Dairy Technology.* (submitted).

For further information, please contact:
Dr. P.M. Kelly

Fig 2. Effect of stirrer speed on the coffee-stability of WMP

Dr. Phil



Dr. David Oldfield

DAIRY PRODUCTS RESEARCH CENTRE

Moorepark, Fermoy, Co. Cork, Ireland

Tel: +353 (0) 25 42222 - Fax: +353 (0) 42340

E-mail: reception@moorepark.teagasc.ie