# EFFECT OF LECITHIN ON THE RECONSTITUTABILITY OF WHOLE MILK POWDER

by

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A THESIS SUBMITTED IN PARTIAL FULFILLMENT

OF THE REQUIREMENTS FOR THE DEGREE OF

MASTER OF SCIENCE

in the Department

of

FOOD SCIENCE

Faculty of Agricultural Sciences

We accept this thesis as conforming to the required standard.

THE UNIVERSITY OF BRITISH COLUMBIA

May 1971

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ABSTRACT

In the last few decades, considerable emphasis has been placed on the problem of manufacturing very readily soluble whole milk powder. A satisfactory instant whole milk powder is difficult to produce largely because of the presence of fat in the system. In the present study, certain substances were added to the whole milk powder to help the dispersion of its constituents. Attempt has been made to investigate the effect of lecithin from different sources on the instantization of whole milk powder and to find out whether this effect is due to a chemical interaction between the casein and lecithin or due to a change in the physical properties of the powder. It was found that the powder particles agglomerated partially and as a result, the reconstitutability in cold water, 4°C, was improved remarkably. Different degrees of improvement in the sinkability were observed when the powder was treated with 2% lecithin from different sources. There was no evidence for casein lecithin interaction but the results show that alteration in the interfacial tension of butter oil and the surface tension of water by lecithins would play an important role in the sinkability improvement of the powder.

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## ACKNOWLEDGEMENTS

I wish to express my gratitude to Dr. S. Nakai for his constant advise and encouragement. Thanks are also expressed to the members of the Department of Food Science, particularly Dr. W.D. Powrie, Dr. J.F. Richards and Dr. N.R. Bulley.

Suggestions from senior graduate students, Reginald Clarke and Thomas Beveridge are appreciated.

# INTRODUCTION

Dry milk production has become an increasingly important segment of the dairy industry. The production of dry whole milk in North America is roughly about 100 million pounds yearly.

The ultimate aim of the industry is to obtain dry products which recombine with water easily giving very little or no sign of detrimental change compared to the original liquid product. A dried milk, to be easily dispersible and reconstitutable, required certain physical-chemical properties, e.g., good wettability and sinkability, absence of any lumping when placed into water, rapid and complete dispersion of particles, no tendency to form a water-repellent film on the walls of the container or fat clumps on the surface of the reconstituted product and absence of flocculated protein on the walls of the container. Regular milk powder is not however, readily accepted by the housewives for beverage purposes due to the difficulty with which it reconstitutes in cold water and its poor flow characteristics. Ease of reconstitution of dried milk is important also in the ice cream industry where large amounts of milk powder are being used. The drawbacks of regular powder can be overcome by subjecting the powder to the so-called instantizing process by one of the following three general methods:

1. The re-wet method or the agglomeration method in which agglomeration is accomplished by wetting the particles, using steam or water, sufficiently to cause the surface to be tacky

whereby the particles will agglomerate. Re-drying to normal moisture content is usually done by hot air. During the process, a substantial part of the lactose changes to crystalline form, mainly $\alpha$-lactose hydrate.

2. The straight through or single-pass method which gives a powder generally termed semi-instant. This method consists of adjusting the spray drying conditions to give a coarse powder. Such large particles are obtained when the viscosity of the feed to the atomizer is high. High total solids and low feed temperature or a combination of both will give a viscous concentrate. The powder produced in this way will not be as coarse as powder from a re-wet instantizer, and will therefore not possess the same instant properties.

3. Fake-instantization, which is based on the use of additives to give powders with wettability and dispersibility characteristics similar to instantized milk.

Effect of sodium citrate, disodium phosphate, sugar, Span, Tween, Myverol type emulsifiers and lecithin on the reconstitutability of dried milk powder has been studied. Lecithin has been used recently in many patents for instantization of dried whole milk powder.

In the present study attempts have been made to investigate the effect of lecithin from different sources on the instantization of dried whole milk and to determine whether this effect is due to chemical interaction between the casein and lecithin or due to a change in the physical properties of the powder.

Dispersibility and sinkability tests were used as a measure of reconstitutability of the powder. In order to study the mechanism involved in the improvement of the reconstitutability of the powder by lecithin, interfacial tension between water and butter oil containing lecithin; the surface tension of water containing lecithin were measured. Casein lecithin interaction was investigated by gel electrophoresis, ultracentrifugation and the extractability of lecithin from casein lecithin mixture.

## LITERATURE REVIEW

When the agglomeration method was applied to whole milk powder, it was found (18) that the resulting milk powders contained approximately four times as much free fat as the original powder. This substantial increase in free fat content was due to the formation of lactose crystals of the $\alpha$-form. When lactose crystalizes, fat bound to lactose will separate, and further, lactose crystals will rupture fat globule membranes. Flavour deterioration of the powder developed during re-drying and storage due to large amount of free fat. Those results indicate that the re-wet method is unsuitable for whole milk powder. The application of the second method to whole milk powder offers advantages that crystallization of lactose can be avoided but powder produced in this way may not be as coarse as powder from a re-wet instantizer, and will therefore not possess the same instant properties(18).Since the first and second methods are not applicable for the instantization of whole milk powder ,surface active agents such as lecithin have been used in many of the instantization processess for whole milk powder.

McIntire and Loo(20,21) developed two lecithin processess assigned to dairy foods. The principle of McIntire process entails the dry blending with a predetermined amount of lecithin which can be accomplished mechanically by any suitable blending apparatus or by heating the lecithin to an extent sufficient to reduce its viscosity and then spraying or pumping the lecithin through a nozzle upon the starting powder. The resulting mixture is agitated or blended sufficiently to effect a thorough intermixture.

Another lecithin process was developed by Sjollema (39). Spray dried milk powder was heated to 70°C in a steam jacketed rotary mixer. Soy lecithin was heated to 70°C also in a separate vessel and mixed with the milk powder to give a mixture of 2 per cent by weight. The mixture was then cooled under evacuated drum, and the product revealed excellent "instant" property in cold water.

Shields, et al, (36) used lecithin in a particular manner to produce a fat-containing milk product having instant properties. During the final drying operation of whole milk and while the material was still in a moist condition, it was intermixed with lecithin (0.25-1%). These authors claimed that when a quantity of this powder was deposited upon the surface of cold water, the water quickly penetrated the mass and the individual porous aggregates without blocking, whereby the entire mass quickly wetted and sank.

Nova, et al (28) modified the above method by atomization of dispersed lecithin in steam. Nova et al (29) described a method for the production of readily reconstitutable fat containing milk powder. Lecithin was incorporated onto the surface of the whole milk powder by atomizing steam, containing lecithin droplets, onto moistened whole milk powder at a temperature between 54-71°C. This led to an agglomerated powder.

Another process was developed by Tumerman, et al (43) in

1966. Spray dried whole milk powder, 26% fat, was injected by means of high velocity air stream, into an agglomeration chamber. An emulsion of 4% soya bean lecithin in water was introduced into the chamber by an independently operated spray nozzle. The wetted particles, moisture content 15%, were graded for size, and dried to a moisture level of 3%. The final product contained 0.06% lecithin, mainly on the surface of the particles, and was instantly soluble in water.

William et al (44) produced instant fat-containing milk powder, by dissolving lecithin ( 1.1%) in the milk fat at a temperature sufficient to maintain the fat in a liquid state. The milk fat-lecithin mixture was subsequently added to concentrated skim milk (in sufficient quantity to give a fat content of 6-48% and an active lecithin ranging from 0.05-4.0% in the final powder). The mixture was homogenized and spray dried by a process designed to increase the bulk density of the powder. Another method which produced quickly soluble milk powders with a high fat content involved spray drying of a precondensed whole milk to which lecithin was added to the milk fat portion in addition to some suitable thickners like alginate (30). Nonfat dry milk solids of improved dispersibility and solubility were prepared by incorporating 0.033-0.066% active lecithin followed by moistening and agglomerating the powder to increase bulk density (39). The lecithin was mixed with vegetable oil (1:3) at 60°C and then added at a rate of 1 drop/5 sec. to the freshly spray dried milk powder at 37-50°C while mixing in a ribbon blender for 15 minutes. A German patent issued to Shields et al (37) covers the production of dried milk and cream having instant properties. The milk powder particles were treated with 0.1-1% finely dispersed lecithin either during or after agglomeration of the powder.

A readily dispersible or "instant" whole milk powder with good free flowing characteristics has been produced by the New Zealand Dairy Research Institute (34). The milk powder was sprayed with a mixture of anhydrous milk fat and lecithin to give a level of 0.2% lecithin in the final powder which was heat tempered and rapidly cooled. Lecithin has been used also for manufacturing of instant soluble cocoa powder (8) by mixing the cocoa powder with 0.5-5% lecithin in a rotating drum.

Nelson <u>et al</u> (25) observed that Tween 80, Tween 60 or lecithin when added to the whole milk or milk powder precondensate at a level of 1% by weight of the fat, improved significantly the sinkability of powder in water at 25°C. The sinkability increased through a range of 1-4% of the additives. However, at concentrations of 2,3, and 4% partial churning of the fat took place during reconstitution. The greatest increase in sinkability, with almost no occurrance of churning was attained with a mixture of Tween 80 and Atmos 300 (hydrophilic-lipophilic balance of 8.0), added to the precondensate or mixed dry with the powder. However, the effect of commercial surface active agents had been investigated before publication of Nelson's and Winder's work.

Hibbs and Ashworth (13) used surface active agents (Span 62, Tween 60, Myverol 1800, and Myverol 1885) to improve the reconstitutability of whole milk powder. These authors found that the incorporation of the above surface active agents to the precondensate at the rate of 0.05% in a reconstituted bases resulted in churning. Samples stored at 7.2°C churned considerably after one month. This churning could be remarkably reduced by storing at a higher temperature, i.e., 29°C.

Gibson and Raithby (12) observed a marked increase in wettability on adding polyoxyethylene sorbitan monolaurate (Tween 20 and Tween 21) to the precondensate (0.5-1% of the total solids). However, the sorbitan monolaurate (Span 20) was almost ineffective. The authors ascribed the effect of surface active agents either to a lowering of the surface tension of water, thus facilitating its penetration into the interparticle spaces of the powder, or to an orientation of these substances into a layer on the powder particle surface, attracting the water into the powder.

Hollender (14) and Mather and Hollender (19) investigated the effect of surface active agents upon the self-dispersion and churnability of dried whole milk in more detail. Tween 81 at a concentration of 0.1% (added to the precondensate on a fluid milk bases) produced a readily dispersible powder. However, an undesirable churning occurred during reconstitution by mechanical agitation. Both improved self-dispersibility and absence of churning during reconstitution were obtained at a proper hydrophilic-lipophilic balance of surface active agents. This balance was achieved using a combination of two substances, e.g., 0.05% polyoxyethylene sorbitan monostearate (Tween 60) and 0.05%

sorbitan monostearate (Span 62).

The effect of the interfacial tension of butter oil on the wettability of the powder was studied by Baker *et al*, ( 7 ). Their results showed that alterations in the interfacial tension of the fat component of a milk powder by Span and Tween type emulsifiers did not in itself exert a marked effect on wettability.

The fat content of dried milk and its degree of unsaturation affects the wettability as well as the self-dispersion of the powder. Self-dispersion of dry whole milk was 4.559, 1.448, 0.955 and 0.527g of fat contents of 1.4, 9.8, 27.0 and 34.9% respectively (41).

Reinke *et al*, (33) found that reducing the fat content of milk increased the self-dispersibility of the powder. Ashworth ( 3 ) found that in freshly made fat-containing powders the effect of the fat content on the wettability was only small; the low, medium and high fat contents (12, 25, and 38%) giving similar high values for wettability. At low storage temperature (1.7 - 7.2°C) these powders maintained their high wettability even up to 2 years storage. However, storage at 29.4°C caused considerable deterioration of wettability in medium and high fat powders. The dried skim milk showed a wettability of the same value in both low and high storage temperatures which was not affected by the time of storage.

A temperature-conditioning was developed by Bullock and Winder ( 9 ) which allows the recovery of the initial reconstitutability possessed immediately after drying. This phenomenon was attributed to the physical rearrangement of the glycerides of the surface fat on the powder particles.

Pyne, (31) has measured the contact angles of water droplets on the flat surface of highly compressed tablets of dry milk products. The non-fat dry milk surface was readily wettable as shown by a cosine of 0.86. However, the wettability (cosine Ø) decreased with an increase in the fat content. Pyne also showed that the surfaces of particles containing fat in the liquid state were more wettable (higher cos Ø) than those containing fat in the solid state.

A process was devised for manufacturing instant whole milk powder by coating the powder particles with the fraction of the milk fat which is liquid at a low temperature (16-18°C) (24,27). The coating material may also contain skim milk solids, lecithin and/or a glyceride of vegetable origin.

It was shown by Baker et al (6) that a milk powder containing a low-melting (19-21°C) butter oil fraction had approximately the same wettability and dispersibility at 24°C as did a superior grade of instant skim milk powder. The interfacial tension (oil-water) of this low melting butter oil fraction was lower than that of higher-melting fractions which yielded much less wettable milk powders. However, Stone et al, (41) concluded from their studies on the self-dispersion of milk powders, in which the butter fat was replaced by corn oil, that the superior dispersibility of the powder was due to the fact that the fat was in the liquid state.

Nelson and Winder (25) observed that the highest sinkability in water at 25°C was always associated with the highest liquid fat content of spray dried whole milk.

The temperature of water had little effect on the self-dispersion of spray-dried whole milk between 1.7°C to 32.2°C (41). However, between 35 and 37.5°C near the melting range of milk fat, a steep increase in self-dispersion took place, followed by gradual increase between 37.5 and 60°C. Similar significant increases in wettability of dried whole milk at water temperature of about 35°C was observed by others: Mol and de Vries, (22), Radema and van Dijk, (32) and Favstova (10).

## METHODS AND MATERIALS

All measurements were performed in duplicate.

### Addition of Lecithin and Commercial Emulsifier to the Powder

An appropriate amount of lecithin* was weighed and dissolved in 10 ml. pet. ether, then this solution was added to an appropriate amount of commercial spray dried whole milk powder** to give a final concentration of 2% lecithin (w/w) with continuous stirring for about one minute. The powder was exposed to the air for 3-4 hours in flat container at room temperature for pet. ether evaporation. The same solvent was used for addition of Span type emulsifiers and acetone for Tween type emulsifiers because of their insolubility in pet. ether.

### Dispersibility Test

The method of Sinnamon *et al* (38) was slightly modified as follows: 4 gm. of milk powder was added to 40 ml. water at 4°C in a 250 ml. beaker. The mixture was stirred by hand with a spatula for a predetermined time interval, 10, 20, 30, 40, and 50 seconds, dispersing as much material as possible, and poured through a cloth layer over a 200 mesh funnel with the aid of about 15" vacuum. Five milliters of the filtrate was dried in an oven 93°C for 5 hours. Percent dispersibility was calculated.

### Extractability of Lecithin from a Mixture of Casein and Lecithin

The mixture of acid casein and lecithin of different concentrations was prepared and left overnight at room temperature,

* Distributed by Nutritional Biochemicals Corporation, purity: Veg. Lecithin 95%, Bovine Lecithin 90%, Soybean Lecithin ( oil not removed ).

** KLIM Trade Mark, 28% butterfat, 26.5% protein, 37.2% lactose, 5.8% minerals and 2.5% moisture.

then freeze-dried. The mixture was prepared as follows:

A. Acid casein, 2% was dissolved in an imidazole buffer, pH 7.0.

B. Lecithin 0.2% was dispersed in the same buffer by 1 min. sonication with Biosonik (Bronwill Scientific).

C. Imidazole buffer, pH 7.0.

Appropriate amounts of A,B and C were mixed to make casein-lecithin ratios of 5, 10, 20, 30, 40, 50, 70, and 90 (mg/mg). The extractability of lecithin from the freeze-dried mixture was measured by using three 4 ml portions of hexane-acetone mixture (4:1). The phosphorus content of the extracts was measured according to Allen (1), with a slight modification as follows: The hexane-acetone solvent was transferred into a 10 ml Kjeldahl flask, evaporated with gentle heat, and digested with 2.0 ml of 60% perchloric acid. The digest was then diluted with 2 ml of the amidol reagent and 1 ml of the ammonium molybdate solution and transferred quantitatively to a 25 ml vol. flask with distilled water. The blue color produced was measured after 12 minutes in a Beckman DB Spectrophotometer at 690 mμ. The amount of phosphorus present was read from a calibration curve prepared by the same procedure for aliquots of a standard phosphate solution containing from 0.01 to 0.2 mg P.

Reagents

Perchloric acid, 60% solution sp. gravity 1.54.

Ammonium molybdate solution, 8.3% solution. A small quantity of concentrated ammonium hydroxide was added to facilitate solution.

Amidol reagent, 2 gm. of amidol and 40 gm. of sodium bisulphite dissolved in glass-distilled water and diluted to 200 ml. The reagent was stored in a well-stoppered bottle painted black outside. This solution was used within 10 days.

Standard phosphate solution, a stock solution containing 1 mg. P per ml prepared by dissolving 1.0967 gm $KH_2PO_4$ (dried in an air oven) in distilled water and diluting to 250 ml. and stored at $4^oC$.

## Ultracentrifugation

Schlieren patterns of acid casein with and without lecithin were obtained in a Beckman L2-65B preparative ultracentrifuge at $20^oC$ with Schlieren optics. One percent acid caseins with and without 0.35% lecithin were prepared in 0.08 M imidazole buffer, pH 7.0. Centrifuge speed was 60,000 rpm.

## Polyacrylamide Gel Electrophoresis

Polyacrylamide gel electrophoresis was performed by a modification of Aschaffenburg's method (2). The gel was prepared as follows: 10.5 gm acrylamide and 0.52 gm N, N-methylenebis-acrylamide were dissolved in 0.1 M Tris-glycine buffer (pH 9.1) and made up to a final volume of 150 ml. This solution was filtered through No. 1 filter paper into a vacuum flask. After the deaeration 1.0 ml. TMED (30% N, N, N', N'-tetramethylene ethylene diamine in 95% ethanol) and 1.25 ml of 10% ammonium persulfate were added to the solution, mixed gently, poured immediately into a mold, covered with a perspex plate and weighed down to expel all the air. The gel was allowed to stand for 30 to 60 minutes to permit completion of polymerization. A horizontal electrophoresis apparatus, with double containers at either end of the gel was used. A sodium chloride solution, 0.1M and 0.175M Tris-glycine buffer were placed in the outer and the inner containers, respectively. The containers were connected with eight folds of cheesecloth. The gel plate was placed on the electrophoresis apparatus and equilibrated by running the current through the gel for 20 hours at 4 °C. The amperage was maintained at 20 mA on a power supply.

Preparation of Sample

A mixture of 2% acid casein and 0.5% lecithin was prepared in 0.175M Tris-glycine buffer in addition to the control (2% acid casein in buffer only). Strips of Wahtman 3MM filter paper 1.5 x 10mm, which were soaked in the casein-lecithin solution after 24 hours, or the control sample and the excess blotted, were inserted in the slot on the equilibrated gel plate. The gel plate was covered with Saran film to prevent drying. Electrophoresis was carried out under the same conditions as the equilibration of the gel, for 24 hours in a cold room. The gel removed from the mold was stained in 0.2% amide black 10 B solution containing 45% methanol and 9.8% acetic acid for 5 minutes and then destained by washing in 10% acetic acid until a clear gel background was obtained.

Sinkability test

The sinkability was determined according to Bullock and Winder (9) with a slight modification as follows:

Materials:

A. A 15 by 20 cm. funnel having an outlet of 1 cm. in diameter to which a short rubber tubing with a pinch clamp was attached.

B. A glass bottle 2.5 cm. in diameter and 5 cm. deep. A piece of screen soldered on the open end of the bottle to sprinkle dried milk on water surface.

C. A stop watch.

Method:

Five hundred milligrams of dried milk were weighed into the small glass bottle. Fifty milliliters of distilled water at 25°C were placed in the funnel with a pinch clamp in place. A 500 ml. Erlenmeyer flask was positioned beheath the outlet. The dried milk was sprinkled quickly and evenly onto the quiescent water surface. After a predetermined time interval, usually 15-30 seconds, the pinch clamp was opened and the portion of the dried milk which had sunk was drawn off quickly with the water onto the Erlenmeyer flask, leaving behind that fraction of the sample which had not sunk.

After complete solution of the sample in the flask, a 5 ml. aliquot was removed and total solids were determined by drying at 93°C for 5 hours, to ascertain the fraction of the sample which sank. The results were expressed as percent sinkability and were calculated as follows:

$$\text{percent sinkability} = \frac{\text{weight of solids in 5 ml. aliquot} \times 50 \times 100}{5 \times \text{weight of original sample}}$$

Free-flowability of the Powder

The static measurement of free-flowability for the milk powders has been done according to Sjollema (40):

A funnel with a narrow stem was mounted exactly 2 cm. above a piece of uniform paper lying on a horizontal flat table. Powder having uniform particle size, 40-60 mesh, was moved down with an electrical vibrator feeder in a very fine stream into the funnel from which it fell on the paper, so that a conic heap was formed. When the top of the powder met the end of the funnel stem, the powder stream was stopped, and the base of the powder heap was outlined with a pencil. After removing the powder, the outlined paper circle was cut out and weighed. As weight per unit area of paper is known, the area of the circular base, and thus the radius were calculated. Then the angle of repose was calculated according to the following formula:

$$\tan \alpha = h/(r-\tfrac{1}{2}a)$$

where: $\alpha$ = angle of repose

h = height of stem above base = 2 cm.

r = radius of the base of the powder heap

a = diameter of the funnel stem = 0.3 cm.

Iodine Value of Lecithin

Iodine value was measured by the official method of A.O.A.C. (5).

Interfacial Tension of Butter Oil (I.T.)

Interfacial tension of butter oil at 40°C was measured according to Baker et al (7) using DuNoy tensiometer. DuNoy tensiometer was used also for measuring the surface tension of water.

# RESULTS

## Effect of Lecithin on the Dispersibility of Dried Whole Milk Powder

Lecithin of different origins (bovine, vegetable, soybean -- refined and unrefined) was added to commercial spray dried whole milk powder, and the dispersibility test was performed.

Figure 1 shows the dispersibility curve for the powder with and without lecithin. All the samples containing different lecithins reached almost the maximum solubility within 1/5 of the time required for the control. The effect of treatment with lecithins on dispersibility of whole milk powder did not significently differ, except in the case of powder treated with refined soy lecithin which showed a slightly increased dispersibility compared to powder treated with other lecithins (Appendix Table 1 and Figure 1).

## Effect of Commercial Emulsifier on the Dispersibility of Dried Whole Milk

In an attempt to find the relationship between the effect of adding lecithin and commercial emulsifiers on the reconstitutability of dried whole milk, Span and Tween type emulsifiers were used on spray dried whole milk in the same manner as in the case of lecithin. Figures 2 and 3 indicate the dispersibility curves of dried whole milk treated with Span and Tween type emulsifiers, respectively. It has been found that Span 85, 80 and 20 improved the dispersibility greatly without any significant difference between emulsifiers. However, Span 40 did not show any improvement

(○——○) instant skim milk powder.

(△——△) whole milk powder containing 2% refined soy-lecithin.

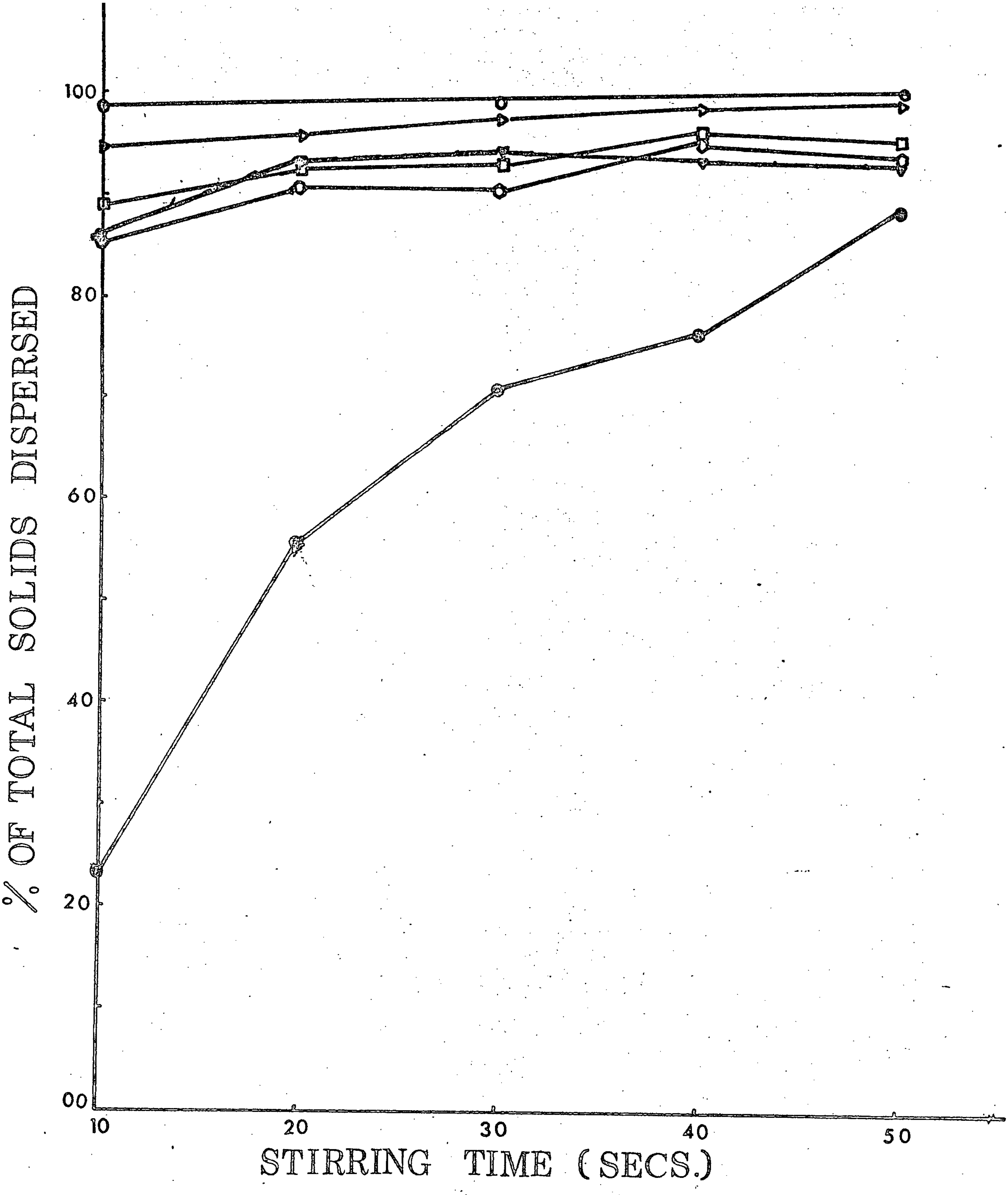
(□——□) whole milk powder containing 2% bovine lecithin.

(▽——▽) whole milk powder containing 2% soy lecithin.

(⬡——⬡) whole milk powder containing 2% vegetable lecithin.

(●——●) whole milk powder without adding lecithin.

Fig. 1. Dispersibility rates for whole milk powder, instant skim milk powder, and whole milk powder containing 2% lecithins of different origins.



100
80
60
40
20
00
% OF TOTAL SOLIDS DISPERSED
10
20
30
40
50
STIRRING TIME (SECS.)

and its treated powder showed no significant difference with the control (Appendix Table II and Figure 2). On the other hand the dispersibility was improved by Tween 20, Tween 80, and Tween 40, almost to the same extent with no significant difference, whereas Tween 60 showed no improvement when compared with the control (Appendix Table III and Figure 3).

Effect of Different Lecithins and Emulsifiers on the Interfacial Tension of Butter Oil and the Surface Tension of Water

In order to study the mechanism involved in the improvement of the reconstitutability of dried whole milk by lecithin, butter oil containing different concentrations of lecithins and emulsifiers was prepared and the interfacila tension (I.T.) between oil and water for butter oil at 40°C was measured (Tables I and II). It is obvious from these tables that different lecithins show different effects on the I.T. of butter oil. The one from bovine decreased remarkably the I.T. The decrease by lecithins from other sources was less than bovine lecithin. the surface tension (S.T.) of water containing lecithin and emulsifiers was also measured at 25°C and is presented in Tables I and II. Bovine lecithin decreased the surface tension of water more than other lecithins did, whereas, different Span and Tween emulsifiers had almost the same effect on the I.T. of butter oil, and the S.T. of water (Table II).

(○—○) Span 80

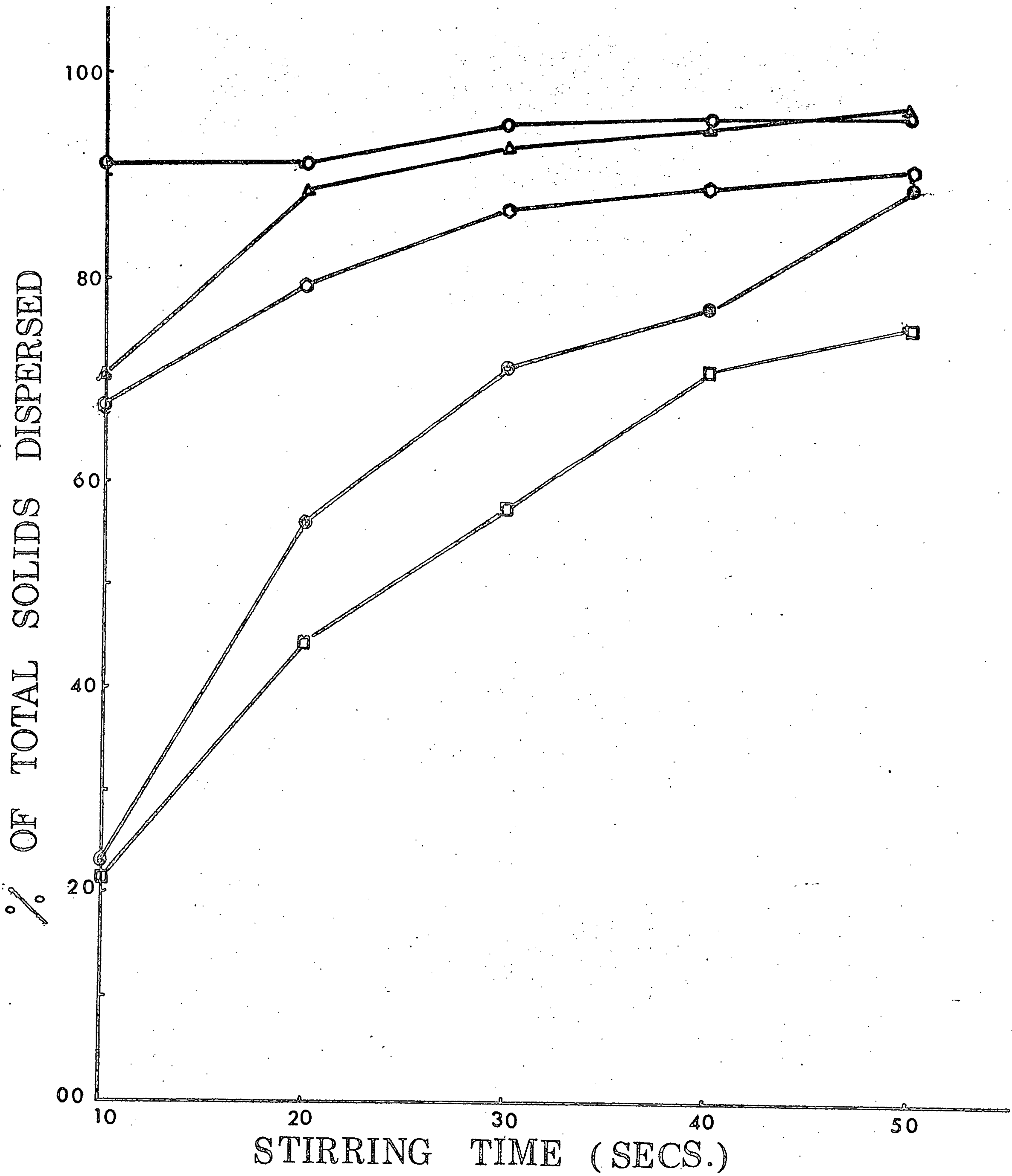
(△—△) Span 85

(⬠—⬠) Span 20

(□—□) Span 40

(●—●) Control

Fig. 2. Dispersibility rates for whole milk powder containing 2% different Span type emulsifiers.


100
80
60
40
20
00
% OF TOTAL SOLIDS DISPERSED
10
20
30
40
50
STIRRING TIME (SECS.)

( ○—○ ) Tween 20

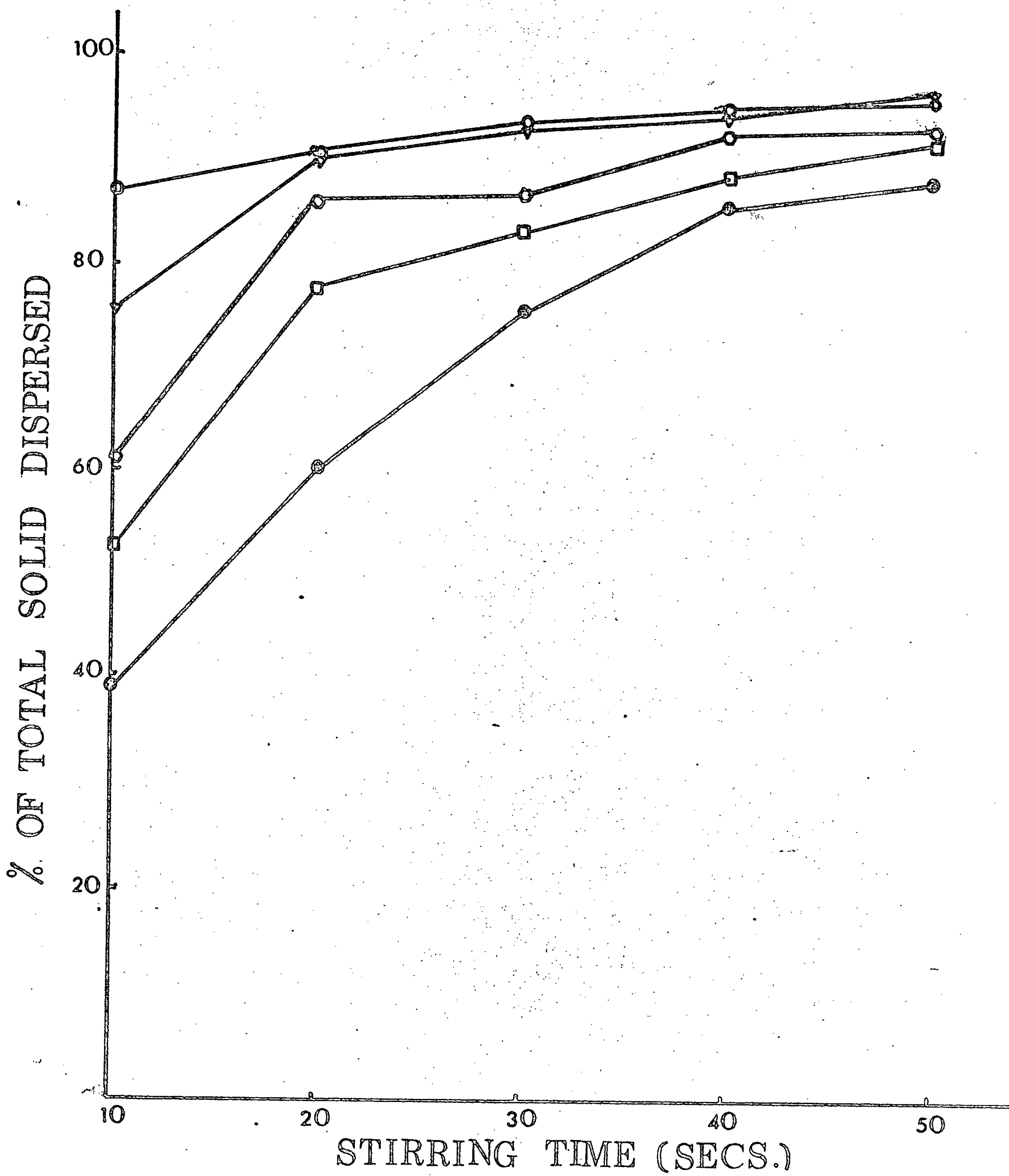
( ▽—▽ ) Tween 80

( ⬡—⬡ ) Tween 40

( □—□ ) Tween 60

( ●—● ) Control

Fig. 3. Dispersibility rates for whole milk powder containing 2% different Tween type emulsifiers.



100
80
60
40
20
% OF TOTAL SOLID DISPERSED
10
20
30
40
50
STIRRING TIME (SECS.)

TABLE I

The iodine value of different lecithins and their effect on the interfacial tension (oil-water) of butter oil, the surface tension of water and the sinkability of dried whole milk

| No. | Type of Lecithin | Iodine Value of Lecithin | Surface Tension of Water Containing 0.050% Lecithin (dyne/cm.) at 25°C | Interfacial Tension (oil-water) dyne/cm at 40°C — Concentration of Lecithin in Butter Oil (%) |  |  |  |  | % Sinkability of Powder Containing 2% Lecithin (30 sec. test time) |
|---|---|---|---|---|---|---|---|---|---|
|  |  |  |  | .0000 | .0312 | .0625 | .1250 | .2500 |  |
| 1 | Bovine Lecithin | 40.9 | 42.8 | 24.5 | 18.8 | 5.3 | 4.1 | 3.8 | 98 |
| 2 | Vegetable Lecithin | 62.5 | 63.3 | 24.5 | 20.4 | 19.3 | 17.2 | 14.6 | 34 |
| 3 | Soybean Lecithin | 85.9 | 65.0 | 24.5 | 21.6 | 20.4 | 17.5 | 16.0 | 26 |
| 4 | Refined soy Lecithin | 67.0 | 68.0 | 24.5 | 21.9 | 20.1 | 16.4 | 15.4 | 16 |
| 5 | Control | ---- | 72.0 | ---- | ---- | ---- | ---- | ---- | 9.5 |

TABLE II

Effect of Span and Tween type emulsifiers on the I.T. of butter oil and the S.T. of water and the dispersibility of powder

| Type of emsulsifier | No. | HLB value[2] | Melting point[2] of emulsifiers C | Surface tension of water containing 0.10% emulsifier dyne/cm. 25°C | Interfacial tension (oil-water) dyne/cm. at 40°C Conc. of emulsifier in butter oil (%) | | | | | % dispersibility of powder containing 2% emulsifier[1] |
|---|---|---|---|---|---|---|---|---|---|---|
| | | | | | 0.000 | 0.031 | 0.062 | 0.125 | 0.250 | |
| Spans | 20 | 8.6 | liquid[3] | 33.2 | 24.8 | 21.3 | 20.0 | 17.5 | 13.4 | 67.8 |
| | 40 | 6.7 | 46 | 43.5 | 24.8 | 21.4 | 18.6 | 17.2 | 12.5 | 21.5 |
| | 80 | 4.3 | liquid | 33.4 | 24.8 | 21.5 | 21.4 | 18.5 | 15.7 | 91.6 |
| | 85 | 1.8 | liquid | 34.5 | 24.8 | 21.0 | 21.0 | 20.5 | 19.3 | 70.4 |
| | | | | | Conc. of emulsifier in butter oil (%) | | | | | |
| | | | | | 0.000 | 0.015 | 0.02 | 0.030 | 0.060 | |
| Tweens | 20 | 16.7 | liquid | 39.5 | 24.8 | 14.9 | 11.5 | 11.9 | 4.6 | 87.3 |
| | 40 | 15.6 | liquid | 39.1 | 24.8 | 15.0 | 11.2 | 8.1 | 4.0 | 61.3 |
| | 60 | 14.9 | 24 | 42.4 | 24.8 | 14.7 | 10.0 | 7.5 | 4.2 | 52.8 |
| | 80 | 15.0 | liquid | 39.7 | 24.8 | 13.2 | 8.0 | 8.6 | 3.8 | 75.5 |

1 % dispersibility as shown on Fig. 3 and 4 after 10 sec. stirring time

2 Reference No. (35)

Iodine Value of Lecithin and its Effect on the Free-flowability of Whole Milk Powder

The angle of repose for whole milk powder treated with different lecithins having uniform particle size was determined as a measure of free-flowability of the powder. Table III shows the angle of repose of the powders treated with different lecithins. It has been found that the sample treated with bovine lecithin yeilded an angle of repose very close to that of instant skim milk powder while lecithins from other origins did not improve the free-flowability of the powder extensively except vegetable lecithin. There was an inverse relationship between the iodine value which reflects the melting point of the lecithin and the free-flowability of the powder (Figure 4).

Hydrophylic-lypophilic Property of Milk Constituents

The hydrophilic property of the protein is very important for the dispersibility of milk powder (16). Attempts were made to check whether there is any interaction between the casein and lecithin which might improve the hydrophilic property of the protein. The interaction was investigated by polyacrylamide gel electrophoresis, ultracentrifugation and the extractability of lecithin from the casein and from the dried skim milk treated with lecithin. However, no indication of interaction between the casein and the lecithin was detected. The mobility on electrophoretogram and the sedimentation coefficient of acid casein did not change after adding lecithin. And no decrease in the extractability of the lecithin was observed for the casein-lecithin mixture and for the dried milk-lecithin mixture (Figure 5).

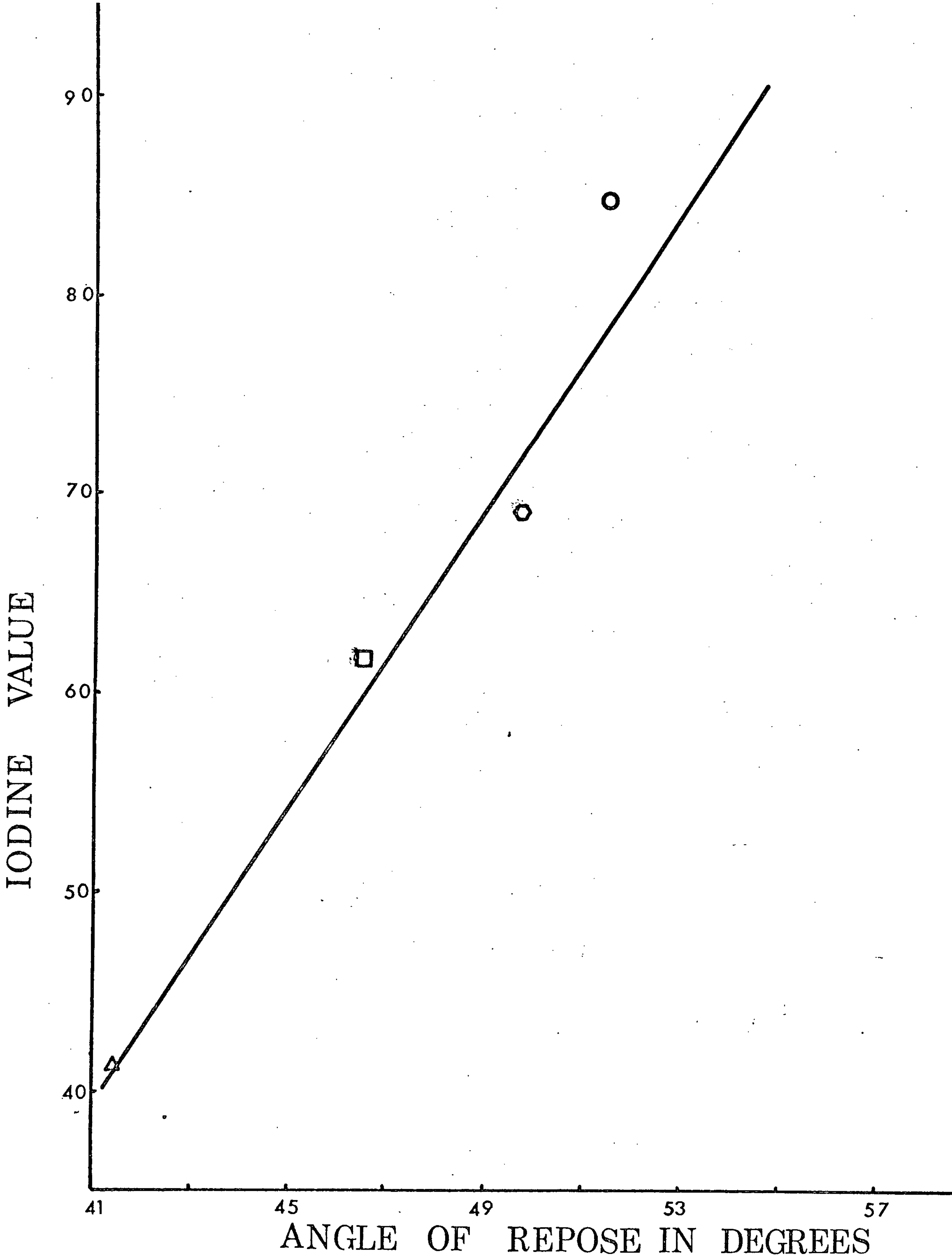
(O) soy lecithin

(O) refined soy lecithin

(□) vegetable lecithin

(△) bovine lecithin

Fig. 4. Iodine value of lecithin and its effect on the free-flowability (angle of repose) of whole milk powder containing 2% lecithin



IODINE VALUE
90
80
70
60
50
40
41
45
49
53
57
ANGLE OF REPOSE IN DEGREES

TABLE III

Angle of repose of whole milk powder treated with 2% different lecithins

| Lecithin Treatment | Angle of Repose (degrees) |
|---|---|
| Control | $51\frac{1}{4}$ [a] |
| Bovine lecithin | $41\frac{3}{4}$ [b] |
| Vegetable lecithin | $46\frac{1}{2}$ [a,b] |
| Soybean lecithin | $51\frac{1}{2}$ [a] |
| Ref. soybean lecithin | $51\frac{1}{4}$ [a] |
| Inst. skim milk | $40\frac{1}{2}$ [b] |

'a' and 'b' are two groups significantly different (Appendix Table V).

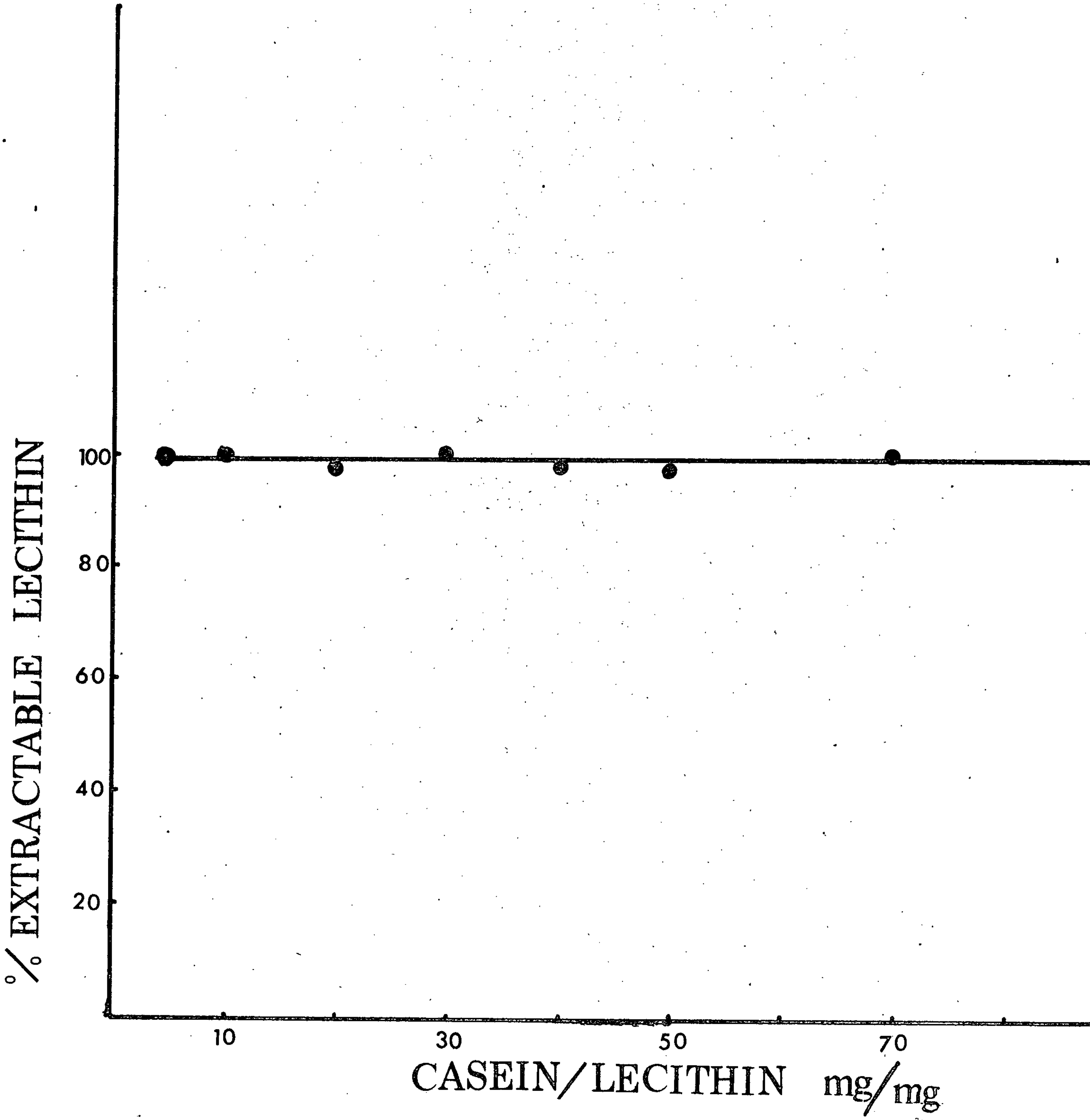
Effect of Particle Size on the Sinkability of the Treated Powder

Powders treated with lecithins or commercial emulsifiers were fractionated into different size particles (130-500 ) by sieve permeametry as it is possible that these additives change the particle size of the powder (Figure 9). The sinkability of the fractionated powders treated with these additives was measured. Figure 6 shows the results of this test. It is noticed that the particle size did not affect the sinkability of the control sample. The additive that did not improve the dispersibility of the powder, Span 40, revealed no effect as well. However, the particle size disclosed a marked effect on the sinkability of powder treated with the additives capable to improve the dispersibility of the powder (Span 20, Tween 20, bovine lecithin). The particle size of powder treated with additives which improved the dispersibility to the lesser extent revealed less effect on the sinkability of the powder (Tween 60).

Concentration of Vegetable Lecithin and the Sinkability and Dispersibility of the Powder

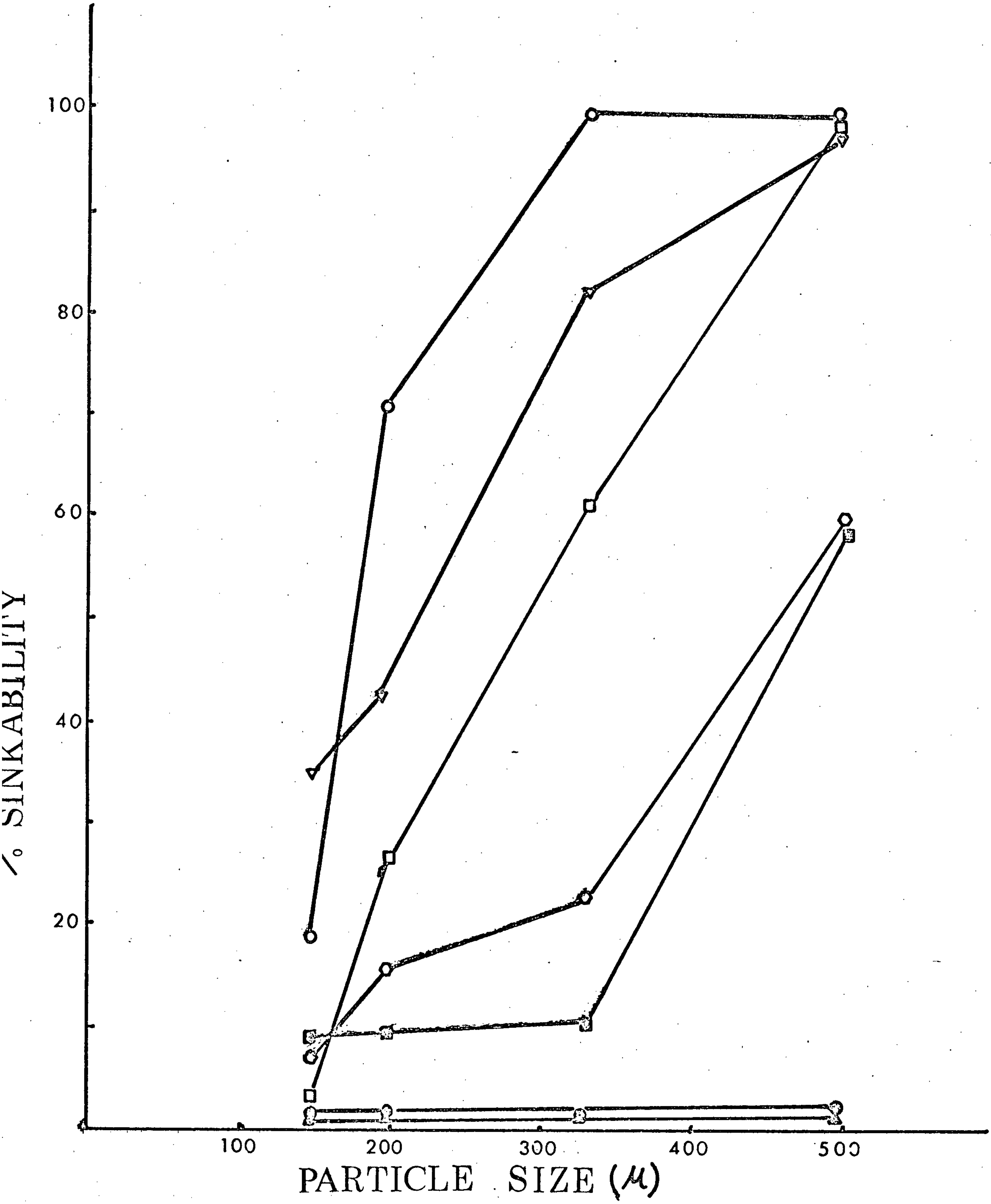
The effect of different concentrations of vegetable lecithin on the sinkability and dispersibility of the powder is seen in Figure 7. The sinkability was effectively improved at concentrations higher than 1%. On the other hand, lecithin has a most pronounced effect upon dispersibility at lower concentrations, e.g., 0.25%, followed by a plateau (Figure 7). The data for the sinkability test fit the equation $y = a + b \log x$ with a

Fig. 5. Extractability of lecithin from casein-lecithin mixtures of different weight ratio(mg/mg).



% EXTRACTABLE LECITHIN
100
80
60
40
20
10
30
50
70
CASEIN/LECITHIN mg/mg

Fig. 6. Effect of particle size on the sinkability (15 secs. test time) of whole milk powder containing 2% Span 20 ( ◎—◎ ), Tween 20 ( ▼—▼ ), bovine lecithin ( ▣—▣ ), Tween 60 ( ⬡—⬡ ), refined soy lecithin ( ■—■ ), Span 40 ( ▲—▲ ), and control ( ●—● ).



% SINKABILITY
100
80
60
40
20
100
200
300
400
500
PARTICLE SIZE (μ)

correlation coefficient of 0.97 (Appendix Table IV).

The lecithin content in particles separated from powder treated with 2% vegetable lecithin increased curvilinearly with particle size from 100 to 300 μ then declined slightly to 500 μ. Lecithin content ranged between 1.27% and 2.4% (Figure 8).

Fig. 7. Effect of concentration of lecithin (vegetable lecithin) on the sinkability (30 secs. test time) and the dispersibility of whole milk powder.

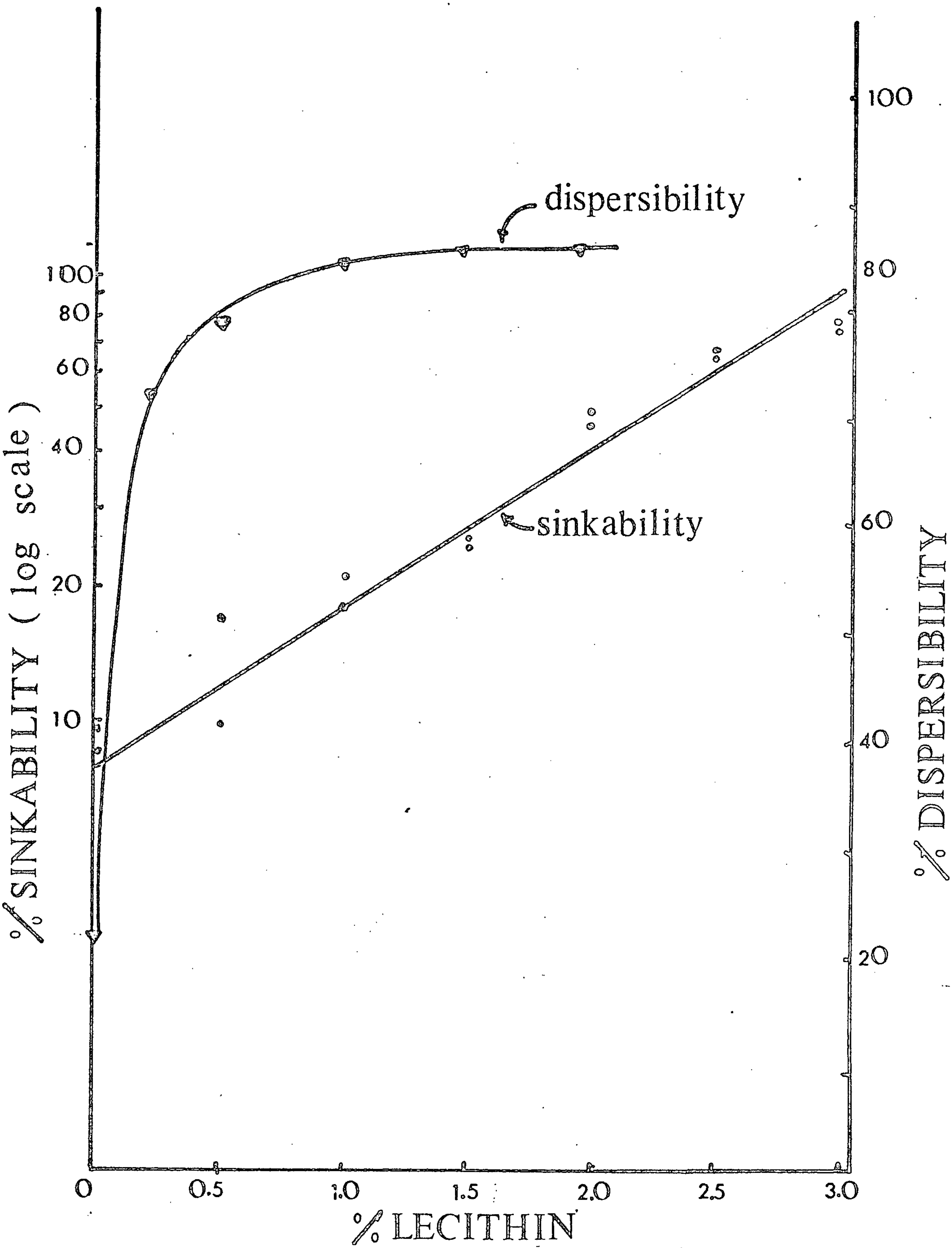
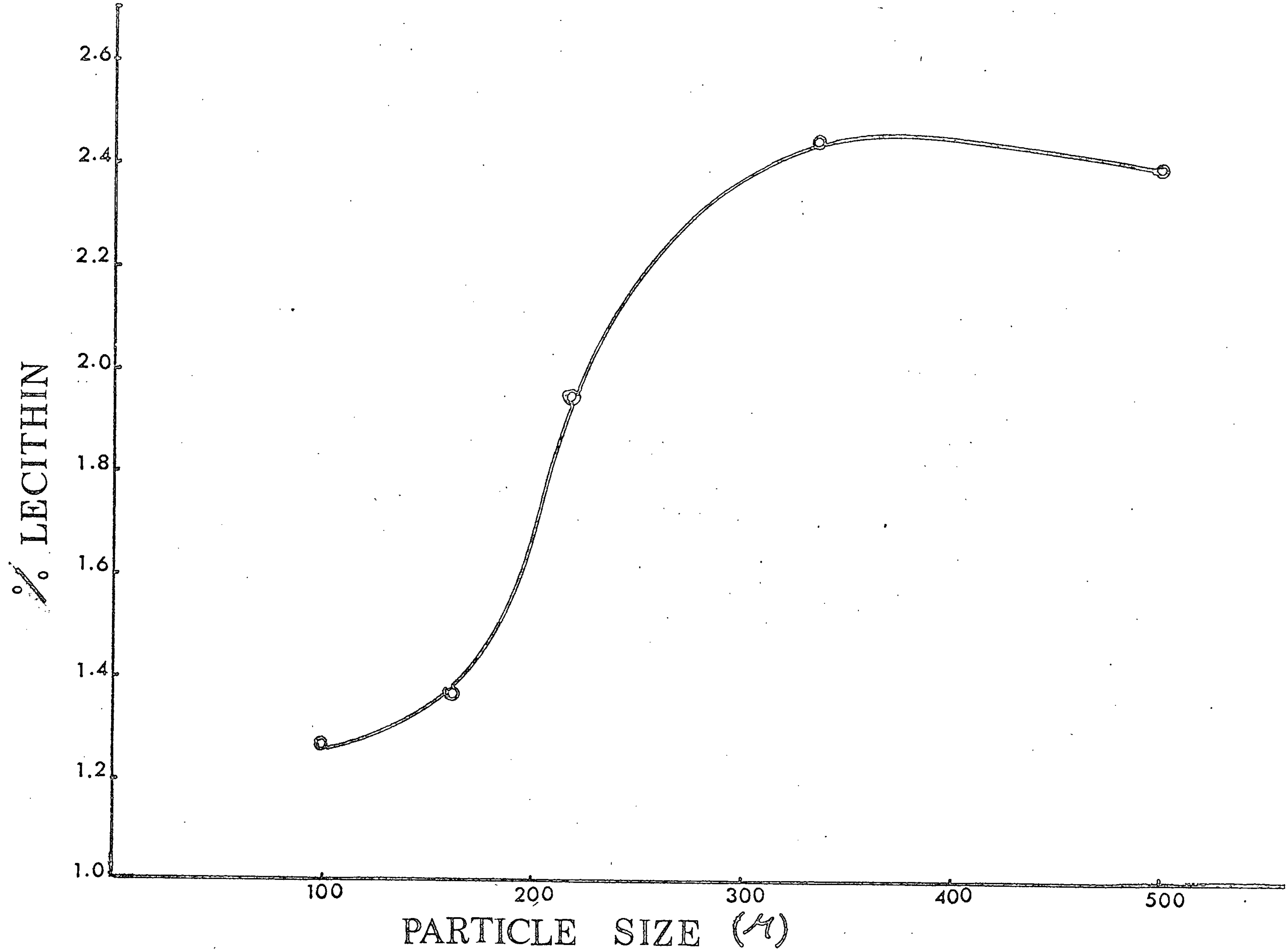

dispersibility
sinkability
% SINKABILITY ( log scale )
100
80
60
40
20
10
% DISPERSIBILITY
100
80
60
40
20
0
0.5
1.0
1.5
2.0
2.5
3.0
% LECITHIN

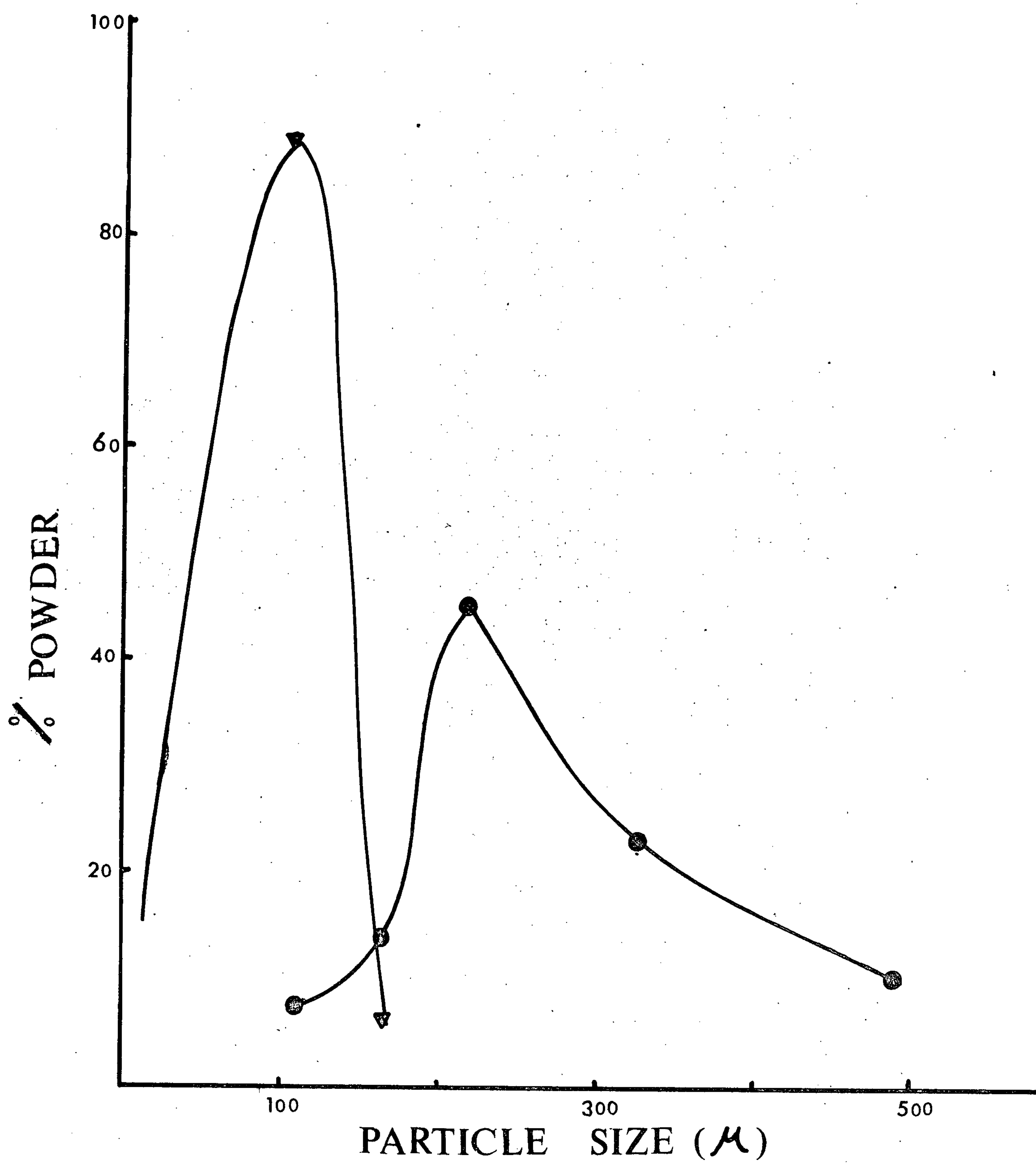
Fig. 8. Distribution of lecithin as related to particle size of dried whole milk powder treated with 2% vegetable lecithin.


% LECITHIN
2.6
2.4
2.2
2.0
1.8
1.6
1.4
1.2
1.0
100
200
300
400
500
PARTICLE SIZE (μ)

Powder treated with 2% vegetable lecithin

Control

Figure 9. Distribution of particle size of the dried whole milk before and after treating with 2% vegetable lecithin.


100
80
60
40
20
% POWDER
100
300
500
PARTICLE SIZE (μ)

# DISCUSSION

Asworth and Bendixen ( 3 ) reported that the physical state of casein is the limiting factor during the dispersion of dried whole milk. Julien and Baker (16) were able to improve the wettability of dried whole milk by limited proteolysis of the whole milk before drying (with Rhozyme P 11). These researchers suggested that this enhancement in wettability was due to a modification of the protein on the micellar surface which resulted in increased hydrophilic character.

The reconstitutability of dried whole milk powder was improved considerably after the addition of lecithin. Lecithin, being an amphiphile, is capable of interacting with milk proteins through hydrogen bonding, electrostatic interaction or hydrophobic interaction. Such interaction should lead to a more hydrophilic casein micelle.

The properties of mixture of various lecithin and dried whole milk powder were investigated to determine the level and type of interaction, if any, and also to evaluate the effect of the various lecithins on the physical properties of the dried whole milk powder.

For measuring the interaction between lecithin and acid casein, different techniques were used, such as the sedimentation analysis, gel electrophoresis and extractability of lecithin from a mixture of acid casein and lecithin. However, there was no difference in the sedimentation coefficient of acid casein and a mixture of acid casein and lecithin. Their mobilities on the polyacrylamide gel electrophoretegram were identical. There was no variation in extractability of lecithin from different casein-lecithin mixtures (Figure 5). From these results it was concluded there was no chemical interaction between lecithin and acid casein. Therefore, the physical characteristics of the powder were investigated.

The dispersibility test failed to show any significant differences among the milk powders treated with different lecithins except in the case of powder treated with refined soy lecithin which showed a slightly increased dispersibility compared to powders treated with other lecithins. Thus the sinkability test was used (Figure 7) which does not require the use of mechanical force in the test. Sinkability is the ease with which the powder particles make contact with water. It is generally accepted that an adequate sinkability is the prerequisite for good dispersibility and sometimes the reconstitutability is measured in terms of sinkability (17).

When lecithins were added to milk powder, a change in the secondary structure* was observed, in which the size distribution of the powder particles was changed and particles having a diameter between 100-500 $\mu$ were produced (Figure 9). However, most of the published data gave an average particle size in the

*According to King(17), dried milk exhibits a well-pronounced dual physical structure, the structure of the individual particle containing the milk solids and small amounts of moisture and air distributed in the state (primary structure), and the structure of the bulk of the dried particles, which represents a typical powder, a system of closely packed solid particles in a gas (secondary structure).

range of 50-110$\mu$ for spray dried whole milk powder (15,17). When the sinkability test for different size particles of lecithin treated powder was performed, the particle size showed a great effect on the sinkability of the powder in that the greater the particle size, the higher was the sinkability (Figure 6). However, the same range of particle size (100-500$\mu$) of the whole milk powder without the addition of lecithin (prepared by moistening the powder to 10% water and re-drying in a vacuum oven at 40°C) did not show any effect on the sinkability of the powder (Figure 6). This was in agreement with the results obtained by Swanson (42) who found no correlation between the average particle size of dried whole milk powder and its reconstitutability. On the other hand, when the amount of lecithin in the different size particles was measured, it was found that the amount of lecithin in these fractions was in the range of 1.27 and 2.35% indicating that the greater the particle size, the higher was its lecithin content (Figure 8). Moreover, higher concentrations of lecithin in the powder increased the sinkability and dispersibility (Figure 7). Thus, it is obvious that there are reasons other than the change in the particle size involved in the improvement of the reconstitutability of the powder by adding lecithin.

The fat content of dried milk may affect the reconstitutability of the powder. Ashworth (4) found that milk powders with low, medium and high fat content (12%, 25% and 34%) showed different ability in maintaining their wettability during storage. A considerable deterioration in wettability was noticed in medium and high fat powders. However, the low fat powder did not show any change in its wettability. Self-dispersion is also affected by the fat content. Stone et al (41) shows that self-dispersion of milk powder containing 1.4% fat was diminished from 4.559 gm to 0.527 gm when the fat content increased to 34.9%. Reink et al (33) also found that reducing the fat content of the powder increased the self-dispersion. With instantized dried whole milk powder, the dispersibility was lowest at 26%, when compared with fat content of 10%, 5%, 2% and 1% (23). As to the effect of the composition of milk fat on the dispersibility of the powder, it appears that the wettability and the dispersibility increased when the melting point was lower. This phenomena was observed by Stone et al (41), Baker et al ( 6), and Nelson et al (25). Bullock and Winder (9 ) found that the sinkability of dried milk powder was greatly affected by the temperature treatment immediately after drying. They devised a temperature conditioning treatment which secured the maximum sinkability. The assumption was that a redistribution of solids and liquid fractions of the milk fat takes place on the surface of the particles. By heating milk powder, surface fat is melted and triglycerides rearrange themselves in a random manner. Rapid chilling then solidifies fat and the solid and liquid triglycerides are fixed in a random distribution. Therefore, the nature of the surface of particles and the kind of compounds present on the surface is considered to be a limiting factor on the reconstitutability of dried whole milk powder.

When different commercial emulsifiers(Span and Tween) having an HLB value in the range of 1.8-16.7were added to milk powder, it was observed that all emulsifiers having a low melting point which existed in a liquid form at 20°C showed greater improvement in the dispersibility of the powder (Table II). However, dispersibility of the powder treated with Span 40, an emulsifier having a melting point of 46°C did not indicate significant difference with the control. Tween 60, which has the highest melting point, 24°C, in the group also did not show a significant difference with the control.

These results suggest that the degree of unsaturation in the fatty acid constituent of the lecithin is probably important for its ability to improve the dispersibility of the powder. However, it was found that lecithins having lower iodine values (less unsaturated fatty acid) revealed greater ability to improve the sinkability of the powder (Table I). Since refined soy lecithin showed significantly a greater effect in improving the dispersibility of milk powder than other lecithins (Figure 1), the low sinkability of its treated powder may be due to a cohesion of powders resulting in a poor flow characteristic. The angle of repose of the powder was $50\frac{1}{4}°$ (Table III). The same was true for powder treated with soy lecithin. As soon as cohesive powder is moistened, portions of the powder become coated with the liquid outside, leaving a pocket of trapped air inside which delays its sinkability. Bovine lecithin which has the lowest iodine value, improved significantly the free-flowingness of the powder of which angle of repose was very close to that of instant skim milk powder (Table III). This powder exhibited the best sinkability. It is necessary for surface active agents to locate on the surface of the powder particles in order to improve the dispersibility (14). A method for adding lecithin or commercial emulsifier to milk powder in this study will comply with this requirement.

Gibson and Raithby (12 ) ascribed effects of surfactants either to lowering of the surface tension of water, thus facilitating its penetration into the particle spaces of milk powder, or to an orientation of these substances into a layer on the powder particle surface attracting the water into the powder. Furthermore, the findings of Baker and Samuel ( 7 ) indicate that the lower melting fat fraction has lower interfacial tension towards water than the higher ones suggesting a possible relationship between the interfacial tension of fat and the wettability of milk powder. In a more detailed investigation, they came to the conclusion that rather the combination of two factors, the physical state and the interfacial tension of the fat, are of importance. Being aware of these assumptions the experiments were conducted to investigate the effect of lecithins and commercial emulsifiers on the surface tension of water and the interfacial tension of butter oil, and the relationship with the sinkability or dispersibility of milk powder. It was found that the lecithin having a greater ability to improve the sinkability of milk powder alters considerably the interfacial tension of butter fat and the surface tension of water (Table I). When the same test was done with commercial emulsifiers (Tween and Span) the ability of these emulsifiers to alter the interfacial tension of the butter oil and the surface tension of water was not significantly correlated with their improving ability in the dispersibility of milk powder. It was also noticed that the alteration in the interfacial tension was dependent on the concentration. Generally the Span type was less effective than the Tween type emulsifiers. At a concentration of 0.06% Tween 20, 40, 60, and 80 decreased the interfacial tension of butter oil from 24.8 to 4.6, 4.0, 4.2 and 3.8 dyne/cm respectively (Table II). Meanwhile a concentration of 0.25% was required for Span 20, 40, 80 and 85 to decrease the interfacial tension of butter oil from 24.8 to 13.4, 12.5, 15.7 and 19.3 dyne/cm, respectively. However, there was almost no correlation observed between the effect of commercial emulsifiers on the surface tension of water and their ability to improve the dispersibility of milk powder. All the Tween and the Span type emulsifiers decreased the surface tension of water to almost the same extent at a concentration of 0.1% (Table II). On the other hand, the lecithins that were more effective in decreasing the surface tension of water showed greater improvement in the sinkability of milk powder (Table I).

As lecithins dissolved in an organic solvent was added to milk powder it is possible that the lecithin was distributed on the surface of powder particles enhancing the surface cohesiveness of powder upon evaporation of the solvent. The particles cohered forming porous aggregates which contain more lecithin than the unaggregated particles. During the reconstitution of this powder the lecithin orienting on the particle surface attracts the water into the powder, and alters the S.T. of water and I.T. of butter oil. Other factors might be involved in this improvement such as the degree of unsaturation of the fatty acid content of the surface active agents which seems to be the predominant factor in improving the dispersibility of the powder treated with Tween and Span type emulsifiers.

## SUMMARY

Studies were conducted to observe the changes in the reconstitutability of dried whole milk when different lecithins or commercial Span and Tween type emulsifiers were added to the powder at 2% concentration. Dispersibility and sinkability tests were used as a measure of reconstitutability. In the dispersibility test the powder containing different lecithins reached almost the maximum solubility within 1/5 the time required for the control. Different Span or Tween type emulsifiers revealed different effects on the dispersibility of the powder. However, a different degree of improvement in the sinkability was observed when the powder was treated with 2% lecithin from different sources, or with 2% Span and Tween type emulsifiers. No evidence for casein lecithin interaction was observed. However, it was found that the powder particles were agglomerated partially and a change in the particle size distribution was observed. Moreover, the particle size itself showed no effect on the sinkability of the control powder (without adding lecithin). It was found that those lecithins which improve the sinkability of the powder remarkably decrease the interfacial tension of butter oil and surface tension of water more than do those which show very little improvement in the sinkability. Several Span and Tween type emulsifiers revealed almost the same effect on the interfacial tension of butter oil, and the

surface tension of water.

It is concluded that the state of emulsifiers, whether it is liquid or solid, is the controlling factor in improving the dispersibility of the powder. But the alteration in the interfacial tension of butter oil and the surface tension of water by lecithins would play an important role in the sinkability of the powder.

# APPENDIX

## TABLE I

Analysis of Variance in Dispersibility of Powder Treated with Lecithins

| Source of variation | Degree of freedom | Mean square | F-ratio |
|---|---|---|---|
| Lecithins | 4 | 1945.7 | 627.19** |
| Stirring time | 4 | 570.45 | 183.79** |
| V x T | 16 | 209.23 | 67.41** |
| Error | 25 | 3.1038 | -- |
| Total | 49 | | |

** Significant at probability 0.01

Duncan New Multiple Range Test:-

Dispersibility of powder treated with bovine lecithin, vegetable lecithin and soy lecithin were homogeneous (did not differ by more than the shortest significant range).

| Lecithin treatment | Control | ref. soy. lec. | soy. lec. | veg. lec. | bov. lec. |
|---|---|---|---|---|---|
| Mean value | 63.19 | 97.81 | 97.67 | 91.58 | 93.70 |

| Stirring time(secs) | 10 | 20 | 30 | 40 | 50 |
|---|---|---|---|---|---|
| Mean value | 75.67 | 85.99 | 89.72 | 92.68 | 94.89 |

TABLE II

Analysis of Variance in Dispersibility of Powder Treated with Span Type Emulsifiers

| Source of variation | Degree of freedom | Mean square | F-ratio |
|---|---|---|---|
| Span emulsifiers | 4 | 1455.500 | 15.85** |
| Stirring time | 4 | 960.970 | 9.80** |
| Error | 16 | 98.022 | -- |
| TOTAL | 24 | | |

** Significant at probability 0.01

Duncan New Multiple Range Test:-

Two homogeneous sets were shown:

1. Dispersibility of powder treated with Span 20, 80, and 85.
2. Dispersibility of powder treated with Span 40 and control.

| Span treatment | Control | 20 | 40 | 80 | 85 |
|---|---|---|---|---|---|
| Mean value | 63.48 | 82.78 | 54.12 | 94.00 | 88.68 |

| Stirring time (secs) | 10 | 20 | 30 | 40 | 50 |
|---|---|---|---|---|---|
| Mean value | 54.78 | 72.04 | 80.80 | 85.86 | 89.58 |

# TABLE III

## Analysis of Variance in Dispersibility of Powder Treated with Tween Type Emulsifiers

| Source of variation | Degree of freedom | Mean square | F-ratio |
|---|---|---|---|
| Tween emulsifiers | 4 | 397.92 | 8.24** |
| Stirring time | 4 | 715.78 | 14.82** |
| Error | 16 | 48.30 | -- |
| TOTAL | 24 | | |

** Significant at probability 0.01

Duncan New Multiple Range Test:-

Two homogeneous sets were shown:

1. Dispersibility of powder treated with Tween 20, 40, and 80.
2. Dispersibility of powder treated with Tween 60 and control.

| Tween treatment | Control | 20 | 40 | 60 | 80 |
|---|---|---|---|---|---|
| Mean value | 70.04 | 92.46 | 84.14 | 79.16 | 89.70 |

| Stirring time (secs) | 10 | 20 | 30 | 40 | 50 |
|---|---|---|---|---|---|
| Mean value | 63.26 | 81.24 | 86.92 | 91.30 | 92.78 |

TABLE IV

Curve Fitting by Computing UBC TRIP Program (Triangular Regression Package)

Effect of Lecithin Concentration on the Sinkability of Dried Whole Milk

| Type of Regression | Regression Equation | Correlation Coefficient |
|---|---|---|
| Polynominal Linear Equation | $y = 0.109+0.0383x$ | 0.93 |
| Exponential Linear Equation | $y = -2.563+2.834 \log x$* | 0.97 |
| Recriprocal Equation | $y = 2.778-25.6(1/x)$ | 0.89 |
| Polynominal Quadratic Equation | $y = -0.276+0.0686x-0.000365x^2$ | 0.85 |
| Polynominal Cubic Equation | $y = -1.122+0.1705x-0.00332x^2 +0.0000237x^3$ | 0.78 |

*The equation is only valid over the range of the data, where $x$ = % sinkability and $y$ = % vegetable lecithin.

TABLE V

Analysis of variance in angle of repose of powder treated with lecithin

| Source of variance | Degree of freedom | Mean square | F-ratio |
|---|---|---|---|
| Lecithin treatment | 5 | 50.538 | 33.249** |
| Rep. | 1 | 1.023 | 0.673 |
| T x R | 5 | 1.52 | |
| Total | 11 | | |

** Significant at probability 0.01

Duncan New Multiple Range Test:

Angle of repose for the control,powder treated with refined soy lecithin and soybean lecithin were homogeneous.

Angle of repose for powder treated with Bovine lecithin, vegetable lecithin and instant skimmilk powder were homogeneous.

| | Angle of repose | SSR | LSR | Soy lec. | Refined soy.lec. | Control |
|---|---|---|---|---|---|---|
| Instant skimmilk | 40 1/2 | 6.25 | 8.85 | 11.0 | 10.75 | 10.75 |
| Bovine lecithin | 41 3/4 | 6.18 | 8.74 | 9.75 | 9.5 | 9.5 |
| Vegetable lecithin | 46 1/2 | 6.11 | 8.64 | NS | NS | NS |
| Control (spray dried whole milk | 51 1/4 | 5.96 | 8.42 | | | |
| Refined soy lecithin | 51 1/4 | 5.70 | 8.06 | | | |
| Soy lecithin | 51 1/2 | | | | | |

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