THE UNIVERSITY OF MINNESOTA

GRADUATE SCHOOL

Report

of

Committee on Thesis

The undersigned, acting as a Committee of the Graduate School, have read the accompanying thesis submitted by Chester Distad Dahle for the degree of Master of Science They approve it as a thesis meeting the requirements of the Graduate School of the University of Minnesota, and recommend that it be accepted in partial fulfillment of the requirements for the degree of Master of Science.

Leroy S Palmer
Chairman

Alice Biester

C. H. Eckles

July 27, 1921

THE UNIVERSITY OF MINNESOTA

GRADUATE SCHOOL

Report

of

Committee on Examination

This is to certify that we the undersigned, as a committee of the Graduate School, have given Chester Distad Dahle final oral examination for the degree of Master of Science. We recommend that the degree of Master of Science be conferred upon the candidate.

Leroy S. Palmer
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July 27, 1921

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SOME PHYSICAL AND CHEMICAL PROPERTIES OF POWDERED MILK

BY

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Introduction

One of the important economic problems in the dairy industry is the stabilization of the supply and demand. This problem is brought about by the great surplus of milk during the spring and summer seasons and by a shortage in the fall and winter. These conditions affect the milk producer, the milk dealer and also the milk consumer. The latter is concerned mostly during the season of shortage, when the price of all dairy products advances. The dealer and producer are affected chiefly during the flush season when the price is lower and the supply greater than the demand. While a large amount of this surplus is made into butter and cheese, this does not offer a complete solution for the problem. The surplus conditions are relieved to a certain extent by this practice but the shortage of fluid milk during the winter months is not solved.

The most recent attempt to solve the fluid milk problem is the preserving of the milk by powdering. This practice had not been carried on to any great extent until the past decade, due to imperfections of the processes of manufacture. The advent of powdered milk will tend to take care of the surplus milk and supply the shortage. Already it is a common practice to standardize whole milk with skim milk powders, and even sell milk made from powdered whole milk and water, and powdered skim milk, sweet butter and water. While this practice is a solution for a shortage, it must be observed from another angle, that of its legality. There are two aspects to this question. The first concerns the advisability of permitting "remade" milk to be sold as natural milk. The second involves the question whether milk can be remade or reconstituted from milk powders and still have all the physical, chemical, and nutritional properties of natural milk. The first aspect of this question is not considered in this thesis.

Milk powders have many advantages and considerable study has been given to their nutritional value, bacterial purity and vitamine content. One question

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which has not been solved is that dealing with the chemical and physical properties of the milks made from the powders. This question is of importance to the user of the product. He should know whether remade milk has the same properties after the powdering prodess and reconstitution as it had before. If a milk has been changed in any manner during the process of powdering so that when it is reconstituted into milk again the properties do not resemble fresh milk, these facts should be ascertained. Such data should be of great value also to those concerned with protecting the consumer against fraud and upholding the pure food laws.

So far there have been no legal standards for "remade" milk. It was not the purpose of this investigation to establish any standards for remade milk, but merely to study some of the physical and chemical properties of the milks as compared to normal milk, to note wherein they differ.

Although a great deal of study has been devoted to powdered milk, the study has been confined principally to other fields. The chemical and physical properties of remade milk have been largely overlooked.

REVIEW OF LITERATURE

Before commencing the review of literature a few definitions should be considered. Several terms will be used from time to time in this thesis, some of which are analogous, such as; powdered milk, dried milk, dehydrated milk and desiccated milk. All these terms apply to fresh cow's milk, either whole, skimmed or partly skimmed, which has been evaporated to dryness. The various products presumably contain no ingredients foreign to cow's milk, so that when water is added to the dried substance in the same proportions that it existed normally in milk, the resulting product will contain the same constituents as fresh milk. There may be some slight change in the state of some of the constituents due to the powdering process, but this defect cannot as yet be altogether controlled.

The terms "remade milk" and "remade cream" according to Redfield[1] (1919) "are the products resulting, when skimmilk powder, or unsweetened condensed milk sterilized or superheated, are mixed with unsalted butter or heavy cream, fresh or frozen, and with water, either by means of some crude device such as a baker's whip or by means of an emulsifier, viscolizer, or homogenizer; or when whole milk powder preferably made from homogenized milk is mixed with water by means of such a device, as a baker's whip, or in a mixing tank equipped with a revolving propeller or paddle."

In this paper the term "remade milk" will refer to any milk resulting from the mixing of powdered whole milk and water in the same proportions as to make the resulting milk resemble as nearly as possible the fresh milk from which it was made.

When the term "synthetic milk" is used in this paper, it will mean a milk made from powdered skimmilk, water and unsalted,sweet-cream butter, in amounts which will make a milk testing 3.5 per cent fat, 87.5 per cent water, and 12.5 per cent solids, the whole being pasteurized at 145°F for 30 minutes, run through a De Laval Emulsifier and cooled immediately at 50°F.

Other terms which will be used are "whole milk powder" and "skimmilk powder". According to the Federal standards the former shall contain at least 26 per cent fat and not more than 5 per cent moisture. Skimmilk powder, according to the same standards must not contain more than 5 per cent moisture, there being no fat standard for this product.

History and Development of Powdered Milk

The origin and history of the powdered milk industry, according to Hunziker(2)(1920), are very closely related to that of the condensed milk industry. The powdered milk industry originated about the middle of the past century. The purpose of condensing and powdering milk is to reduce bulkiness, and to "preserve milk as nearly as possible in its natural condition so as to make possible its economical transportation to all parts of the world."

The essential difference between condensed milk and powdered milk lies in the degree of concentration. Condensed milk is milk whose bulk has been reduced to one-third or one-fourth its volume, while powdered milk is milk from which all or very nearly all of the water has been driven.

Gail Borden in 1856 received a patent on his condensing process from the United States government and Great Britian. Mention is made of a British patent(3) on a powdered milk process as early as 1835, but little is written of any particular process until 1855 when Grimwade secured a patent on a process which has been termed (2) the first commercially usable process. This process was known as the dough-drying process, and foreign ingredients were added to make the casein more soluble. Grimwade's process, while still in use, has given way to other forms which are more simple to operate and which manufacture a product of much higher quality.

Of the earlier powders made many contained added sugar and alkalies. As early as 1893[4], a powder was manufactured on Prince Edward Island, and an analysis showed it to contain a large amount of foreign ingredients, particularly sugar.

In this country the powdered milk industry did not flourish at first. The condensed milk industry offered too much competition, and, since the first powders manufactured were not to be recommended from the stand point of their keeping qualities, it was found difficult to dispose of them. The first powders were also far from being soluble, and it was not until later years, that new inventions and improvements made it possible to manufacture a more uniform product.

In 1899[3] the Swedish inventor Eckenberg patented a process which was a great improvement over previous methods of manufacture. By this method milk was dried on a drum which was heated by steam and which revolved in a vacuum chamber. Also in 1899[5] a machine was invented by W. B. Gere known as the double roll process, but the product made by it was not satisfactory, and for that reason it was not put on the market. Later L. C. Merrell of the same company hit upon the idea of spraying milk into a current of heated air. All other processes previous to this time had involved the drying of milk on heated drums, or in open containers. The spray process gave the quality desired, and immediately the firm of Merrell-Soule began to determine the commercial value of the process. Meanwhile patents had been applied for and the patent office referred the company to a patent granted in 1901 to Robert Stauf, of Posen, Germany, which apparently covered this process. Merrell-Soule then purchased this patent from Stauf for the United States and thirteen other countries.

The wisdom of the purchase of all patents held by the Stauf interests has since been amply demonstrated. In 1915, patent litigations which had been in the courts for three years were decided by the court of appeals in favor of the Merrell-Soule Company, the decision being based on this company's possession not only of its own patents, but also of the basic patents governing the spray process of powdered milk.

It is only during the past decade that the milk powder industry has advanced noticeably in this country. In May 1911[6] there were only ten factories making milk powder. In 1912 there had been over sixty patents issued on powdered milk processes and improvements, and since that date many more have been issued.

The powdered milk industry has also been well established in England and other European countries. For several years remade milk has been a common food for infants in many of the large milk depots in England. A great deal of powdered milk is exported to England yearly from the United States. This practice greatly relieved the milk shortage in that country during the war.

The industry is also becomming well established in newer countries such as New Zealand. American processes are now being installed in that country.[7]

Since the industry has become established in America, the bulk of the powder made has been from skimmilk, although the production of powdered whole milk has shown a substantial increase during the past few years. The following table shows the amounts of various kinds of powders produced in 1918 and 1919.[8]

Kind of Product	Number of Plants Reporting		pounds	pounds
	1918	1919	1918	1919
Skimmilk powder	57	55	26,202,406	34,945,416
Whole milk powder	23	20	4,000,663	9,042,236
Cream powder	10	6	620,886	607,190

The powdered milk industry in the United States is not confined to the eastern part of the country where it originated, but has now spread westward and is including the states where milk can be produced more cheaply, and where there is an abundance of feed, and where the output of milk far exceeds the demand. The indications are that the western part of the United States will produce the bulk of the dried milk in the future while the eastern half of the country and foreign nations will be the users of the product.

Relation of Powdered Milk to the Dairy Industry

Several important questions have arisen in connection with powdered milk, and the influence it will have on the dairy industry in this country. Some have asked the question, "will it under sell fresh milk?" Others ask," can it be sold as cheaply as fresh milk?" While another raises the question as to the nutritive value in comparison with fresh milk.

Some writers(9) believe that the powdered milk industry will act as a stabilizer in several ways. "It will extend summer into winter, by spreading surplus milk produced at a cheap price in the summer months when grass is plentiful, into the winter season. The price could be reduced two to three cents per quart in most cities through the process of drying the milk into a powder, and then "reconstituting" it as milk by the addition of water, skimmilk powder and sweet butter. So perfect is the product resulting from this practice that one large dairy resorted to the use of powdered milk to eke out a scanty supply of fresh milk received from the farms. No complaints were received from any of the customers as to the taste and quality of the milk."

Comparing the cost of fresh and remade milk to the consumer, Dr. Eddy(10) (1920), has shown that remade milk can be sold at a cost of 10.85 cents per quart before bottling. This estimation was made when butter was worth 56 cents per pound, and skimmilk powder 28 cents per pound. At the same time the cost of fresh milk before bottling was 9.5 centers per quart.

At the present market values of butter and skimmilk powder, at 37 cents and 15 cents respectively, the cost of remade milk before bottling would be 6.45 cents per quart.

The heads of dairy departments in ten of the leading states, according to Taylor(11) are not quite agreed as to the effect remade milk will have on the dairy industry. However, the majority of them believe that the powdered milk industry will be a benefit to the dairy industry and to agriculture in general, as stated in the following reasons:

A. "It will help stabilize the market for dairy products.

B. It will increase the use for milk generally, especially in countries where on account of climatic conditions the use of milk has been restricted.

C. It will help solve the problem of surplus milk.

D. It will not compete directly with the market milk industry, but will be used principally for cooking, for bakeries and confectioners.

E. The manufacture of skimmilk powder would be an advantage to dairymen since the prices realized by them in supplying milk for this product are very satisfactory.

F. The more uses milk is put to, the better it is for the dairy farmer."

A few heads of dairy departments take a somewhat pessimistic view of the effect of this industry, for the following reasons:

A. "It will have a tendency to take the place of fresh milk in the newer dairy districts and in districts where as yet dairying has not entered into the scheme of agriculture.

B. It might be a cheaper source of milk than fresh milk, thus putting a price on fresh milk which would discourage production.

C. On account of the lack of local laws or of laxity in enforcing them, it is often mixed with fresh milk and sold as such.

D. It is still doubtful whether remade milk contains all the important growth principles of fresh milk."

From the above statements it would seem that the use of powdered milk in America should not have any detrimental effect on the dairy industry, but would have rather a tendency to stabilize it. At the same time it is apparent that statutes should be provided to prevent the sale of remade milk as fresh milk.

Processes of Manufacture

Powdered milk is manufactured by subjecting fresh milk to certain processes, which, by means of heat, drive off practically all the moisture. There are many processes in use today, but those which may be considered as commercially successful may be classed into three distinct groups, as follows:

1. Dough-drying process.
2. Film or drum-drying process.
3. Spray-drying process.

These processes are mentioned in order of their origin, and will be discussed briefly in the same order.

Dough-drying process.

Hunziker(2) describes the dough-drying process as the earliest and crudest of the processes now in use. The milk is condensed in any manner, with or without agitation, either in vacuum or in open air, by means of heat. The milk is condensed to a dough and the dough spread out on trays and dried to a hard substance in vacuum chambers or in other vaults or drying apparatus provided with a heating device. The dried product is usually ground to a fine powder.

While this process is still in use, it has given way to other forms which are simpler to operate, and which turn out a better product more rapidly.

Film-drying process.

To this group belong many processes which dry the milk with or without previous concentration on the surface of one or more steam-heated revolving drums. In some cases the milk is picked up by the drums, or sprayed onto the drums.

The film of dried milk is then scraped off with each revolution of the drum by means of a mechanical scraper or knife. These drums may operate in the open or under reduced pressure.

One of the most common and successful of the drum methods is the Just-process. It was the first of its type and was patented by John A. Just of Syracuse, New York. It consists of two horizontal steam-heated revolving cylinders which are installed close enough to form a contact at their periphery. The milk is discharged in the center over and between the two cylinders and the dried milk is removed by knives and falls into receptacles.

This patent covers the adding of certain chemicals, (CaO and $CaCl_2$) which are supposed to reduce acidity, and of calcium hypochlorite for the purpose of preserving the fatty acids in the finished product.

The temperature of the heating surface of the drums may reach 212°F. This high temperature tends to reduce the solubility, but the addition of alkalies helps to minimize the solubility destruction.

James R. Hatmaker of London purchased the Just process for operation in Europe and later modified it. The system is now usually known as the Just-Hatmaker process. There are many other systems of the drum methods, but the so-called Just-Hatmaker process is the more commonly used.

Other systems are, the Eckenberg, the Passburg, the Bufflovak, the Grovers, and the Gratham. The Eckenberg and Bufflovak differ in that the milk is dried under reduced pressure and upon one drum. The powder is scraped off in the usual way and is removed without breaking the vacuum. These processes have the advantage of subjecting the milk to lower temperatures thus tending to preserve some what the solubility of the powder.

The advantages of the drum methods are: (1) better keeping quality of the powders, (2) saving of fuel, and (3) preservation of the vitamines, as has been amply demonstrated by experiments using powder made by the Just-Hatmaker system.

The main disadvantage of the drum method is the low solubility of the powder.

Spray-drying process.

To this group belong several systems where-by the milk is sprayed into an atmosphere of heated air, the small particles of milk surrendering their moisture rapidly and falling to the bottom of the chamber in fine flakes. The moisture laden air escapes to the outside through openings covered with muslin to prevent the escape of the powder. We find many patents and machines making milk powders in this manner.

The Stauf process is the first commercially successful application of drying milk in this manner. The patent claims that the solids of liquids such as blood, milk, etc. may be obtained in the form of a powder by means of bringing a fine spray into a regulated current of air. The Merrell-Soule Company of Syracuse, New York now controll this patent, and also have another patent listed as the Merrell-Merrell-Gere process. This patent while similar to the Stauf patent refers specifically to the drying of previously condensed milk.

The milk is first condensed in a vacuum pan and sprayed under pressure of about 270 atmosphere into a current of dry air, so that all the liquid portion is driven from the milk. The temperature of the air is around 180°F.

During the life of the Stauf patent which expired in 1918, no one could manufacture milk powder by the spray method without paying tribute to the owners of the patent, the Merrell-Soule Company.

Since 1918 we find another spray process known as the Dick Process. It was invented by S. M. Dick of Minneapolis, Minnesota. It differs from the so-called Merrell-Soule process in that the milk enters a tall chamber at the top by gravity and is sprayed and distributed by a revolving disk arrangement, or centrifugal spray. The temperature of the air in the drying chamber ranged from 212 - 260F. The air enters on all sides of the powdering cell through openings placed both near the top and near the bottom.

Other spray processes in use are the Roger's Process, the Mc Lachlan and the Gray. Each differs somewhat from those mentioned above, but they are similar in principle.

The advantage of the spray process lies mainly in the high degree of solubility of the powders. Stocking[12] (1917) holds that the small particles of the milk when sprayed give off moisture so rapidly that they are kept in a cool condition during the drying process. It is claimed that the powders retain all the natural properties of the milk and go back readily to the original state by adding water.

The disadvantage of the spray powders lies in their poor keeping quality as has been stated by Coutts and Porcher.[15] This is believed to be due to the finely devided state of the particles coming into contact with the air. Another disadvantage recently shown is that the spray powders have lost in a large measure the antiscorbutic property of the original milk.

Uses of Powdered Milk

The uses to which powdered milk have been put are numerous. The initial use was as a substitute for fresh milk in the bakery, confectionery and ice cream plants, also for cooking purposes in the home. The number of uses to which milk powder has been put has increased rapidly until now we find practically as many uses for it as we do for fresh milk.

The use of milk powder as fluid milk has not reached any great proportions as yet in this country, although there are instances where it has been used successfully. At the Nitro Plant in West Virginia[13] it was the source of fluid milk during the war. The fresh milk supply was considered unsafe for human consumption, and as a means of obtaining milk for the thousands of employees it was decided to install a plant for reconstituting milk from sweet butter, skimmilk powder and water.

Hospital and other ships of the navy are supplied with fluid milk in the same manner. The ingredients can be stored to a better advantage and over longer periods of time than fresh milk.

Soule [14] is optimistic over the coming use of dried milk as fluid milk, and predicts that the future will see remade milk supplying the bulk of fluid milk in the large cities.

There is no question but what it is the ideal source of fluid milk for campers and travelers, as Coutts [15] points out, in as much as dried milk has portability, keeping properties, and convenience.

In the home it plays an important part in cooking. It may be used in the liquid form or in the dry form. If used dry usually one-fourth of a cup of powder will suffice for a cup of liquid milk.

Milk powder forms the basis for many proprietary infant and invalid foods. It is important in foods of this kind in supplying readily digestible solids.

Larsen and White [16] find that milk powder has a special value for making starter for butter making in locations where a good milk supply is not available. They found that the butter scored higher and showed a lower acidity.

The ice cream maker has profited more than anyone by the use of powdered milk. He has taken advantage of an article which is always constant in supply, so today we find a great deal of the ice cream being made from powdered milk. It is possible for the ice cream maker to have at short notice an unfailing supply of sweet cream of any butter fat standard. By means of an emulsifier or homogenizer, the constituents are put back into the form of cream. This practice will increase the quality of ice cream because it is fresh. A manufacturer can regulate his supply of cream in accordance with the weather condition, and also have a supply equal to the demand, thus eliminating any possible waste due to souring.

Bacteria in Powdered Milk

It is generally understood that powdered milks in general have one distinct advantage over liquid cow's milk. This advantage lies in its bacterial purity. This does not mean that powdered milks are sterile, but means merely that the number of bacteria per gram or per c.c. has been greatly lessened. The reason may be three-fold. First, in order that a powder may have maximum solubility, it must come from milk as free from acid as possible. In maintaining a free acid milk all necessary precautions to insure low acid are taken. In this manner the bacterial count is kept down. Secondly, the heat of pasteurization, condensing, and powdering have a destructive effect on the living bacteria. Thirdly, the moisture content of the finished product falls below 5 per cent which also accounts for the low count.

Some bacteria will always be found in powdered milk. Some of these may get in during the process while others may have existed naturally in the milk and due to some particular characteristic may have survived the drying. According to Jordan [17] (1920), many of the higher forms of bacteria show great resistance to drying. "If the actual body substance is not protected by a gelatinous capsule, the complete removal of moisture speedily destroys the life of the bacterium. Most vegetative forms of bacteria are rather quickly killed by air drying. Of the pathogenic types, the tubercle bacillus is more resistant, while cholera spirillum is one of the more sensitive to drying. The spores of bacteria are much more resistant than vegetative forms."

Hiss and Zinsser[18] (1918) quote Ficher as saying that "the resistance of bacteria to desiccation is influenced by the age of the culture investigated, the rapidity with which the moisture is withdrawn and the temperature."

Bacteria need water or moisture for their development. It has been stated by some that they need a concentration of 30 per cent for proper development.

According to Kendall[19] "bacteria normally contain 80 per cent of moisture in their substance, and they develop typically only in a media containing large amounts of water. They do not vegetate normally in dried media although many forms resist drying for long periods of time."

The above statement would tend to explain the low bacterial counts in powdered milk. While some of the bacteria present may be heat resistant forms others may have entered after the drying process and failed to multiply because of the low moisture content of the powder.

From the standpoint of public health it is the pathogenic forms of bacteria which are of greatest importance. The forms which are not pathogenic do not have a detrimental effect on the remade milk, since as a rule only enough milk is reconstituted at one time to satisfy the immediate needs of the consumer. Usually this milk is used up before the active fermentation caused by the non-pathogenic forms sets in.

Kosswicz[20] claims that drying reduces the bacterial content of powdered milk to some extent, although Bacillus sinapivagus, B. prodigiosus, B. fluorescens liquifaciens are not destroyed. He also claims that it is possible to re-infect the powders again if not kept in closed containers.

Hoffman's[21] results of inoculation experiments with guinea pigs showed that bovine tubercle bacilli are destroyed in manufacturing milk powder by the Just Hatmaker process. This is contrary to statements made by Delipine[22] (1914) who claims to have found a few living tubercle bacilli of the bovine type after going through a drum drying process. However, he found more in a milk which had been powdered by the spray process, and these were capable of producing tuberculosis in guinea pigs. Delipine has probably done more work on the bacteria of powdered milk than any one investigator. He states that in his use of the spray and drum methods he was never able to find a sterile sample. The heat was not sufficient to bring about the death of several saprophytic and some pathogenic forms.

However, he did find some reduction of bacteria due to the death of streptococci staphylococci, sarcinae, B. coli, yeasts, etc.

Vining[23] (1915), states that the average "samples of powdered milk contains 4000-5000 germs per gram weight", and is unwilling to accept the statement that the tubercle bacillus is never present.

Stocking[12] states that out of 2800 samples counted in the Merrill-Soule factory 96 per cent contained below 25000 bacteria per c.c., and had an average of about 2000 per c.c. figured on the liquid basis.

From the foregoing statements it may be said that dried milk should be a much more sanitary food than ordinary fresh cow's milk. If much of the remade milk falls in the neighborhood of 2000 bacteria per c.c. it can readily be seen that it would fall in the same class as certified milk. These milks should not be classed as certified milks, however, unless produced under the same conditions.

It is reasonable to believe that powdered milk would be a safer source of food than ordinary cow's milk, disregarding the source of fresh milk in either case. The reason for this lies in the fact that powdered milk usually passes through two more heating processes than does fresh milk, namely, condensing and drying.

The Vitamines of Powdered Milk

The conception of what constitutes a food has been changed considerably in the past generation. In 1825 it was held that a food consisted of those things of which living beings are composed. This idea was later changed and food was likened to a substance like blood, since it is blood which nourishes a tissue. From 1860 to 1870 energy began to be emphasized and the term calories became prominent.

The next change came with the agriculturalists idea of the balance ration when a proper diet must necessarily contain a definite proportion of protein, carbohydrate and mineral salts. This idea has since been improved upon in that it has been found that a diet which is balanced in this manner may be still lacking in substances of chemical nature now known as vitamines. It has been shown conclusively that these substances are essential to promote growth and reproduction, and to protect the body from certain diseases.

It is not the purpose of the writer to elaborate to any extent on the vitamine question, but to mention merely the connection between vitamines and powdered milk.

The vitamine in which we are particularly interested is the one which is better known as the antiscorbutic vitamine.

While the question of the antiscorbutic properties of powdered milk is directly connected only with the processes employed in its manufacture, it should be born in mind also that other factors apparently influence the content of this vitamine in cow's milk. For example, several investigators, Hart, Steenbock, and Ellis[24] (1920), Hess, Unger, and Supplee[25] (1920), Dutcher and others[26] (1920), have shown that milk produced by cows given a ration poor in antiscorbutic vitamine will be poor in antiscorbutic vitamine, and vice versa.

It was found by Chick, Hume and Skelton[27] (1918) that milk was normally low in antiscorbutic vitamine, and that other antiscorbutic properties were needed in the ration of guinea pigs to protect them from scurvy. These same authors[28] (1918) maintain that the antiscorbutic vitamine which is already deficient in milk is injured by heat and drying and any child given dried or heated milk should receive an additional antiscorbutic in the ration. They recommend orange juice or potato.

Barnes and Hume[29] (1919) found that 100 to 150 c.c. of raw milk protected guinea pigs from scurvy, but the same amounts of dried milk did not protect them. Similar results were obtained with monkeys. They also pointed out that the feed of the cow was an influencing factor in supplying antiscorbutic to milk and milk products.

Drummond[30](1918) is of the belief that the antiscorbutic vitamine of milk might suffer in the drying process.

McCollum and Davis[31](1915) heated a wet skimmilk powder in an autoclave at 15 pounds pressure for one hour and found that it no longer supported growth.

Hart, Steenbock and Smith[32] found that milk sterilized at 120°C for ten minutes, commercial unsweetened condensed milk and a Merrell-Soule milk powder had lost their antiscorbutic properties whan used in quantities equivalent to an amount of raw milk which would prevent scurvy in guinea pigs.

Coutts[15] quotes Naish as stating that there was only one case of scruvy out of 2,730 babies fed on dried milk in England milk depots. This case responded readily to treatment. Dr. Naish has given up the idea of supplying an additional antiscorbutic when feeding dried milk.

Hess and Unger[33](1919) found that Dryco (a brand of powder made by the Just process) possessed antiscorbutic properties. An equivalent of 80 c.c. of dried milk cured cases of scurvy in guinea pigs. This brand of powder was also found to be antiscorbutic by Hart, Steenbock and Ellis[34](1921). The same workers in the same investigation fed a number of other powders, including a Merrell-Soule powdered skimmilk made by the spray method, a skimmilk and a whole milk powder made by the California Central creameries spray process. None of these proved to be antiscorbutic. A skimmilk powder made by the Just process was antiscorbutic.

Winter produced milk dried by the drum method furnished protection if fed in quantities equivalent to 75 to 80 c.c. daily, but offered no protection in amounts to equal 35 to 40 c.c. per day. The authors offer no explanation for the difference in the antiscorbutic potency of the different types of powders. Hoard's Dairyman[35] in commenting on the experiment mentions the fact that the vitamine is not destroyed by heat so much as through oxidation and if milk could be powdered in the absence of oxygen, there would be a greater chance for preserving the vitamine.

This explanation seems to be well grounded since there is usually more heat applied to milk in drum methods than in case of spray. But in both of these processes of manufacture the powders are produced in the presence of air, greater heat being applied to the drum dried powder, which was found to be antiscorbutic.

There is still room for explanation in connection with oxidation and the antiscorbutic potency of powdered milk, especially that made by the spray process.

That oxidation is a bigger factor than heat in destroying the antiscorbutic vitamine has been shown very recently by Anderson, Dutcher and others[35a] (1921). They found that boiled milk was equal to unheated raw milk in antiscorbutic potency, but that milk heated at 145°F for 30 minutes with vigorous mechanical agitation had lost its antiscorbutic power. Oxidation apparently occurred in the milk which was agitated during heating, due to the intimate contact of air with the milk particles. This idea is supported by the fact that when air, oxygen and hydrogen peroxide were forced through milk while being heated at pasteurizing temperatures, the latter (i.e. the oxygen and hydrogen peroxide) caused even more rapid destruction of the vitamine. These also destroyed the vitamine at room temperature, but not nearly so rapidly. Heat accelerated the oxidation greatly. It was also found that orange juice boiled for 30 minutes was antiscorbutic, while hydrogen peroxide at room temperature destroyed this factor.

Anderson, Dutcher and co-workers conclude that oxidation is a bigger factor than heat in destroying the antiscorbutic vitamine, but that heat hastens or accelerates the oxidation.

The oxidation probably takes place largely at the time of powdering in as much as the spray is so finely divided that a great deal of surface is exposed to the heat and air. It is safe to assume that from the results of experiments, the powders made by the drum process are antiscorbutic, while those made by the spray method are not.

After all, the antiscorbutic potency is not of such great importance in connection with powdered milk feeding since a cheap source is readily found in orange juice, which can be easily administered.

Nutritive Value of Dried Milk

Dried milk in human nutrition.

It is generally held that the only correct food for young mammals is its mother's milk or milk from the same species, because it is adjusted by nature to contain in correct proportions the constituents needed for the young of that species. But there are times when the supply of mother's milk is of insufficient amounts to insure the proper nutrition of the young. Again the mother may die or cease to lactate thus necessitating the obtaining of milk from some other source.

For centuries cow's milk has been the leading substitute for mother's milk or human milk. In some countries goat's milk is commonly used. However, we often find babies who are unable to thrive on cow's milk. Again we find cases where the supply of cow's milk is not to be recommended from the standpoint of its cleanliness. Then sometimes the supply is not constant, as is often the case in the larger cities. These cases are not infrequent and to solve the problem of obtaining a constant supply of good clean milk for infants, dried cow's milk has been substituted for the fluid milk. Dried milk as just mentioned and which will be mentioned often in this paper is, of course, mixed with water in the infant feeding work which will be described.

Many pediatricians and physicians in England have used dried milk in infant feeding with success. Not only has it been successful from the standpoint of nutrition, but it has been found to be a very convenient way of handling the milk in the many milk depots in England. Only enough milk for a feeding needs to be made up at one time, thus eliminating the inconvenience of refrigeration and pasteurization.

As early as 1904(15) dried milk came into prominence in England as a substitute for fluid milk. It was first fed to infants of the poor because it was convenient. Since 1904 its use as an infant food has spread until now there are over 80 districts in England maintaining milk depots for the purpose of supplying dried milk to babies. Convenience is but one of the advantages of dried milk, for Naish(36) holds that there are other advantages, namely: high digestibility, freedom from pathogenic bacteria, and low cost.

High digestibility is an essential in infant feeding because of the delicate structure of the digestive organs of infants. Powdered milk has proved to be highly digestible. Sommerville(37)(1905) concluded from artificial digestive experiments that with respect to fat and protein dried milk is even more digestible than fresh cow's milk. Still(38)(1905) states that he has no doubt the proteins in dried milk are more digestible than those in fresh milk.

Some cases are found where babies are unable to tolerate cow's milk,many becoming violently ill. Vomiting and diarrhoea have been the chief complaints experienced, and the mortality in some cities has been very high, especially in the summer months. It is difficult to produce and refrigerate fresh milk properly in the summer to insure the milk being low in the pathogenic bacteria which are the cause of "summer complaint" and other digestion disturbances. Dried milk has the advantage of not needing refrigeration.

According to Bennett(39)(1918), dried milk is better tolerated by infants than either raw or boiled milk. This is especially true in case of babies who have suffered a "food injury". He recommends dried milk as soon as an infant does not prosper on ordinary milk. He further states that "with a change to dried milk comes a marked improvement. The relief from digestive symptoms is immediate, vomiting has been controlled and intestinal indigestion overcome." Dr. Elmendorf(40) also has found that dried milk is better tolerated than fresh milk and that "Honor Brand" (now called Dryco) gives large heavy stools. Pritchard(41)(1913) holds that dried milk is safer to use than fluid milk as it is freer from pathogenic organisms, but that it must be modified to suit the requirements of each infant.

When some infants are taken off mother's milk and put on cow's milk they frequently fail to gain properly. In many cases the growth curves are practically at a stand-still, but a surprising gain has been noted in most cases when the diet was changed to dried milk. Coutts[15] quotes Winfield as showing the growth curves of infants fed on dried milk to resemble closely the average growth curves of breast fed children. Borland[42] (1920), reports gains with 47 babies fed dried milk. Price[43] (1920) at Boston, fed powdered milk to babies and noted excellent results. He fed groups of children on "Grade A" fresh milk at the same time, and found that the average gain was greater in case of the dried milk. Naish[44] (1908) superintended the feeding of 445 babies at the Sheffield (England) depot with excellent results. He found that "dried milk produced an improvement in weight, bodily vigor and colour." Bennett[38] reports a gain of 7 pounds in 3 months time.

In the feeding of infants on a large scale as was carried out in England, very little scurvy or rickets was noted. The reason probably lies in the fact that the powders fed were largely drum dried, which have been proved recently[34] to be antiscorbutic. Naish[36] states that "scurvy is non-existant in dried milk feeding." Millard[45] (1910) fed over 100 babies with no scurvy or rickets but orange juice was used as an antiscorbutic.

The excellent results obtained from dried milk feeding are due to the fact that the properties of the remade milks resemble mother's milk more closely than fresh milk does. This applies particularly to the protein, especially the casein. The process of manufacture has altered casein in such a manner as to make them coagulate with rennet in a fine, flocculent curd, which is more readily digested than the hard massed curd of fresh cow's milk. The finely divided curds are more commonly found in drum dried powders, due to the more intense heat. This will be shown later in the experimental data. Sommerville[37] (1905) holds that "dried milk in respect to its reaction to rennet, peptic and pancreatic digestion, and in respect to the condition of fat approaches more nearly to human milk. Millard[45] (1910) points out that mothers prefer to use dried milk to nursing their infants after they have noted the results obtained with dried milk feeding.

It is evident that powdered milks have points of distinct advantage over fresh milk, and serve as a very important substitute for both fluid and human milk. It is especially important in case of babies who digest milk poorly, because of its high digestibility and close resemblance in certain physical properties to mother's milk. It is to be recommended highly as a food for young infants. Its nutritive value is equal to fresh cow's milk, although in cases of some powders, where very young children are fed, an antiscorbutic is to be recommended.

Dried Milk in Animal Nutrition

Dried milk plays an important role in animal nutrition as well as in human nutrition, although not such an important role. It has been used in animal feeding with success. When whole milk is sold from the farm and there is no skimmilk for the young calves skimmilk powder is an excellent substitute.

It was found to be successful in calf feeding experiments carried out at the University of Missouri[46]. Many calf feeds prepared by commercial concerns contain skimmilk powder in various amounts. Hayward[47] at Pennsylvania fed skimmilk powder with cocoanut meal, linseed meal, wheat flour and dried blood with good results. Hart and Humphrey[48] even fed powdered milk to dairy cows and found it was as efficient from the standpoint of milk production as dried distillers grain and casein when fed with cornmeal, corn stover and corn silage.

Dried milk is an important constituent in basal rations for experimental and laboratory animals. It has been used extensively by investigators in supplying protein to rations[49,50]

While powdered milk was intended largely for human consumption, it has been found useful in animal feeding. Skimmilk powder has been used more than whole milk powders and may tend to offer a solution to questions of calf feeding or calf raising where whole milk is sold from the farm.

Dried buttermilk, a product not mentioned before in this paper has been used extensively in animal nutrition. It is destined to play a more important role in the nutrition of swine and poultry because of its lower cost than other dried milks.

Physical and Chemical Properties of Powdered Milk

Not a great deal of time has been given to the study of the physical and chemical properties of powdered milk. The majority of attention has been devoted to the study of methods of manufacture and of the nutritional value. Some writers have mentioned the keeping qualities of powdered milk, and these writers are agreed that the whole milk powders go bad or "off flavored" with age, due to some deterioration of the fat. They agree that the drum-dried powders are less liable to this fault than spray-dried powders. They are not agreed as to the type of deterioration.

There seems to be a confusion of the terms applied to "off flavored" powders. The terms applied are rancidity and tallowiness. Some use the terms analogously, but the writer differentiates according to the following definitions: Richmond[51] 1899 holds rancidity of butterfat to be due to hydrolysis of the fat, splitting it up into fatty acids and glycerol, the volatile acids being liberated and the smell of these giving rise to the rancid odor in butterfat. Guthrie[52] 1917 defines rancidity as a butyric acid flavor due to hydrolysis.

Tallowiness in butterfat, according to Hunziker and Hosman[53] (1917) is due to oxidation, giving the butter an odor of old tallow.

Coutts[15] finds old powders are apt to deteriorate, giving rise to dark color, and bad odors, the odor being due to changes taking place in the fat. He considers the change in fat to be the most important element in keeping quality of powdered milk, although he regards packing also as having an important bearing on the keeping quality. Coutts examined a sample which had been kept in a hermitically sealed tin for 3 or 4 years, and found it to be free from rancid odor, but it had a slight stale taste. He further states that milk powders made by the spray process, which are in a finely divided state, do not keep so well as those made by the hot roller process.

Poucher holds, according to Coutts[15] that the change in fat is not rancidity but oxidation, holding that, "rancidity is a microbial phenomenon consisting in a saponification of fatty matter, splitting it into glycerine and fatty acids", and that "it is the latter, especially butyric acid, which gives the odour of rancidity". He believes tallowiness to be due to oxidation of the fat.

The question of the keeping quality of the milk powders will be referred to again later in this thesis as certain of the experimental observations which were made seem to have an important bearing on it.

Next to keeping quality, solubility seems to be the most important factor to be considered. A milk powder to resemble milk closely must have in conjunction with good keeping qualities a high degree of solubility. Hunziker[2] (1920) states that "the solubility of powders will vary with the quality of fluid milk and with the process of manufacture, the powders from the spray process being more soluble than those of the drum method." He further states that "the combination of the heat of desiccation and high acidity of milk, tends to rob the protein and ash constituents of the resulting powder of their natural solubility. The higher the degree of acidity in the fluid milk, the lower will be its solubility."

Hunziker found that "when solutions of film powders were allowed to rest in test tubes there would always be a considerable deposit at the bottom, this being more noticeable in cold water solutions. No such deposit was noticed in the case of spray powders."

Hunziker concludes that "other conditions being the same, the finer the particle the more rapidly will it dissolve, because the smaller the body the larger is its surface in proportion to its cubical contents."

Huyge[54] (1904) stated that a brand of milk powder made at Oostcamp dissolved entirely in water at 70-80 degrees C, while Eckenberg[55] stated that "the milk powder made by evaporating in Vacuo would dissolve into a milk-white like solution with water at 60-70 degrees C., having physical properties similar to milk in that the albuminoids were present in the right physiological condition as a food material."

Others[15] have obtained results which are in accordance with those mentioned above. Monier-Williams in 1918 examined a powder made from whole milk by the Hatmaker process. He made it up with cold, previously boiled distilled water, and noticed that the fat separated out quickly as a yellow layer at the surface. He also found that the enzymes were not active, and that the fat globules were in most cases larger than in normal milk. The powder also contained more undissolved protein than normal milk. The curd when formed by rennet, he found to be flocculent and finely divided, but when formed by acetic acid was similar to that produced from normal milk. The chemical analysis of the milk powder showed the following results:

Moisture	3.7 per cent
Fat	30.0 per cent
Ash	5.29 per cent.

Mendenhall[56] (1918) states, "In the best preparations of dried whole or half-skimmilk the constituents are little altered from their natural state in fresh milk. The butterfat retains the globular form and readily emulsifies when mixed with water, the actual size of the fat globules is apparently reduced by the drying process, the albumen is not coagulated, and the casein is not toughened in drying and is still miscible with water".

Lane-Claypon[57] (1916) mentions a case where the fat globules had changed in form and did not emulsify. The rendered fat rose to the top of the cylinder. She also mentions several analyses of milk powders, which are given below:

Samples	Fat Per Cent	Protein Per Cent	Sugar Per Cent	Water Per cent	Ash Per Cent
I	25	25	38-40	4	
II	22-26	25-26	34-37	-	5-6
III	26-30	24-26	34-40	-	5-7

Marquardt(58) (1920) analyzed some Merrell-Soule powder and found it to contain 25 per cent fat, 7 per cent moisture, 93 per cent dry matter, and .5 per cent insoluble matter. He stated further that the powder was kept at room temperature for over three months without any objectionable changes taking place.

McClellan(59) (1908) found difficulty in extraction of the fat from milk powders "due to the coating of the fat globules during the process of evaporation with an impermeable substance which prevents the solvent from penetrating." It is now known that it is impossible to extract fat from milk or milk powders with the usual fat solvents without first coagulation of the hydrophylic colloids which surround the fat.

Washburn(60) (1919) examined some whole milk powder made by the Dick Process and found the powder to be practically soluble, and having no apparent trace of cooked taste. The cream rose promptly as in fresh milk and was of such a nature that it was possible to whip it. He went so far as to separate the milk making butter from the cream and cottage cheese from the skimmilk, which he claimed to be in a marketable condition, having no peculiar or undesirable flavor. The fat globules showed very little injury.

He further states that the curd produced with rennet was "tender and flocculent at first, which settled into a slightly friable finished curd." The enzymes were active though not as active as in the case of raw fresh milk.

He found the individual powder grains to be very uniform as to size, and spherical in shape. His observation led him to conclude that the powder grains have clusters of butterfat in the center, "or in other words the fat particles are grouped together and then coated about with a layer of colloidal or non-fatty solid portions of milk somewhat like filled apple dumpling." The same powder showed but slight deterioration at the age of 8½ months, according to the same report.

Bennett[39] writes that many salts of milks are "rendered inert by boiling and drying. The casein of dried milk is in finely divided particles and does not unite to form large compact curds as does fresh cow's milk."

Pritchard[61] examined some milk powder made by the Just-Hatmaker process and found the particles to be "irregular polygonal plaques of varying dimensions and of striated structures." Those of the spray process were more homogeneous and smaller. The drum-dried powder was more yellow in color, while the spray-dried was white. The cream of the drum-dried powder came to the surface as an oil, and the odor was agreeable.

Although the air-dried powder gave a more normal cream line, it carried with it a disagreeable odor of tallowiness, the "odor of the latter being due to oxidation resulting from the fine state of division in which the fat particles are present in the air."

Merrell[62] claims that milk is not essentially altered from its natural characteristics as a food if properly desiccated. "When dried rapidly at a low temperature the concentration of lactic acid has little effect on the casein." The albumen is not coagulated if partially evaporated milk is sprayed into a current of hot air."

Stevenson and Peck[63] reconstituted some milk from water, butterfat, and skimmilk powder and found it to be a very good product, except that after a time a thin crust of butter formed on the top of the milk. It usually did not act in this manner when kept in a refrigerator for less than 24 hours. The formation of butter, they claim, was due to some change in the ingredients of the skimmilk powder brought about by the drying process, the power to hold fat in emulsion having been partly destroyed.

Knock[64] believes that a milk powder in which the proteins exist in their natural condition is not to be found on the market. He believes "that the preservation of the proteins in their natural condition is an essential requirement of a milk powder, and also that a whole milk powder answering all the requirements cannot be introduced without difficulty even though the danger that the fat may become rancid is obviated."

The writer is of the opinion that the greatest problem facing the powdered milk industry is the one dealing with the keeping quality of the whole milk powders. The skimmilk powders evidently do not offer a great problem since they are known to keep long enough to permit their use universally. Whole milk powders will continue to be manufactured and are being manufactured in large amounts annually, but the fact that they must be bought in small amounts to insure their keeping until used up, necessarily makes them a more expensive source of powdered milk.

GENERAL PLAN OF EXPERIMENT

The plan of this experiment was to make a study of powdered milk from the standpoint of the physical and chemical properties with respect to the process involved in the manufacture of the powders.

Four powders were studied, two whole milk powders, one partly skimmed powder and one skimmilk powder. These powders are all prominent powders and well known to users of powdered milks. They represent powders made by the drum method and the spray method.

In all cases the milk was reconstituted to resemble fresh cow's milk as nearly as possible with regard to the proportion of constituents. The conditions under which the milks were made represent where possible home conditions. Tap water was used except in cases where it hindered certain chemical determinations. The powders were weighed carefully to insure the correct proportions of constituents.

The proportions of powder and water used were according to directions sent out by the company manufacturing the powder. The plan was adhered to at first but it was found advisable to change in some cases, as will be discussed later.

The physical study included a study of the physical structure of the milk powders, the specific gravity, freezing point, electrical conductivity, viscosity, creaming and other physical appearances on standing. The chemical study included the determination of soluble proteins, the rennet coagulability, the buffer value, the keeping quality and enzyme activity. In all experiments a pasteurized or raw milk check was run.

Methods of Remaking the Milk

A. Kind of powders used

The powders used in this experiment were the Dick's whole milk powder made by the International Dried Milk Company of Minneapolis, Minnesota, a Merrell-Soule whole milk powder and a skimmilk powder made by the Merrell-Soule Company Syracuse, New York, and "Dryco" a partly skimmed powder made by the Dry Milk Company, New York City. The Dick and Merrell-Soule powders represent powders made by the Spray process, while "Dryco" represents a powder made by the drum process. These processes have been described briefly in the review of literature.

Fresh powder was obtained at the beginning of the experiment. From time to time fresh samples of Dick's powder were obtained. The stock of Merrell-Soule powder was not replenished during the experiment, but new cans from the original lot were opened from time to time. The same lot of skimmilk powder used in making the synthetic milk was used throughout the experiment. The Dryco powder was renewed once.

B. Equipment for Remaking the Milk

An ordinary egg beater and "Daisy churn" were used in remaking the milk frompowdered whole milk and water. Usually just enough milk was made up at one time for the experiment to be conducted. Tap water was used in nearly all cases at a temperature as hot as the hand could stand. These samples were allowed to cool before using.

A DeLaval Centrifugal Emulsor, No. 2 was used in reconstituting the milk from skimmilk powder, sweet butter and water. This machine has a capacity of 25 gallons per hour.

EXPERIMENTAL

Physical Structure of Milk Powders

Mention has been made in the review of literature concerning the structure of the milk powder particles. Several investigators have observed that a difference exists in the size and shape and general appearance of powders made by the different methods. The powder particles made by the drum method have been described as irregular, polygoneal plaques, while those of the spray process have been described as spherical granules. These descriptions have been confirmed by observations made in this investigation. It was observed also that there was a difference in the size of the particles which were made by the same type of process. However, this feature is largely controllable and need not be discussed at this time.

The purpose of this experiment was to note the appearance of the various particles as to their structure, size, shape, etc. A microscope was used in making the observations. The powders were observed while in a dry state, and later after water had been added.

Some of the dry Dick powder was dusted on a slide, care being taken that no large dlumps were present. When observed under low power, the particles existing singly on the slide appeared to be spherical in shape and fairly uniform as to size. A drop of water was then placed on the granules. This greatly changed the appearance of the field. The granules began to swell rapidly and move about in the water. Some began to disintegrate. The structure could be seen very plainly while in this condition. The particles still appeared spherical, but the fat globules could now be seen plainly existing in their natural form on the surface of the granule, and within, each fat globule being imbedded in the solid matter of the milk serum which had begun to imbibe water.

The most significant feature of the structure of the swollen granule was the fact that within nearly all the granules appeared a large air cell, which was quite undiscernable while the powder grains were dry. This air cell existed in the center of the granule like a core and was surrounded by fat globules and the protein material of milk. The cell was large, and in many comprised about one-half of the total volume of the individual particle.

As a granule disintegrated as many would when in contact with water, the air cell occasionally burst, and sent out showers of fat globules and protein material in a manner resembling closely the bursting of a shell. Some granules disintegrated more rapidly than others. Some disintegrated so rapidly that the air cell or bubble floated about alone for some time before bursting.

Microscopic observation of whole milk powder made by the Gray process showed it to have a structure identical with that of the Dick powder. In the Gray process the milk is sprayed by a centrifugal spray like in the Dick process. This indicates that the method of spraying is a factor responsible in part, at least, for the structure of the samples just described.

The air cell existing within the powder particles is very stable. Ordinary grinding in a mortar has no effect on them. It took several hours grinding in a ball mill to expel the air from part of the powder granules. Prolonged heating at 100°C eliminated most of the air from the Merrell-Soule powder.

The Merrell-Soule powder closely resembled the Dick Powder except that the air cell was smaller and was at times absent entirely. Another difference was that the fat was highly homogenized. These powder particles were smaller and also seemed more soluble in water than the Dick powder.

The powder grains or flakes of Dryco were entirely different in appearance. They were much larger and of no particular shape. When water was placed on the slide containing Dryco, only slight swelling was noted. No air cell was present. The fat globules were observed to be their natural size. The flakes showed little evidence of disintegration, due to their low solubility.

Photomicrographs were made of the various powder grains to show their size and shape under low magnification, and also to show their structure as it is revealed when the individual particles are in a swollen condition. In some cases it was found helpful to stain the fat globules in order to photograph them. This was done with Sudan III in the following manner:

Some of the powder to be photographed was dusted into a solution of 60 per cent acetone and allowed to remain for several minutes. A few drops of N/50 HCl and about three drops of Sudan III in saturated acetone solution were added. This was allowed to stand for several minutes and then washed with 20 per cent acetone. A few of the stained grains were next placed on a slide and photographed. The photographs were made on Wrattin and Wainwrights' panchromatic "M" plates. Ray filters were used to bring out detail or to show contrast in some cases.

More difficulty was experienced in photographing the swollen particles especially the Merrell-Soule powder. The stained particles would not swell in water, so some other liquid had to be used. It was necessary in this case to use a liquid with the ability to swell the particle without permitting the granule to disintegrate rapidly. For the Dick powder it was found that this condition was satisfied by the addition of 2 or 3 drops of 0.1N NaOH to about 1 c.c. of the liquid (20 per cent acetone) containing the stained granules. No swelling was necessary in photographing the Dryco powder. It was found that a higher magnification was sufficient to show the details of this powder.

Extreme difficulty was experienced in photographing the Merrell-Soule powder due to the fact that the particles disintegrated so rapidly in the solutions used. Photographs were made of some unstained grains, which had been heated for several hours at 100°C to destroy their solubility. These were photographed in water, using a No. F "M" filter to give contrast. The resulting photograph showed that the air cell had been expelled by the prolonged heating.

Later some unstained particles were photographed in a solution of formalin, water and 0.1 N NaOH (equal parts). The particles swelled without disintegrating. A No. F. "M" filter was also used in photographing these particles.

PLATE I - DICK'S WHOLE MILK POWDER

Magnified 90 Diameters

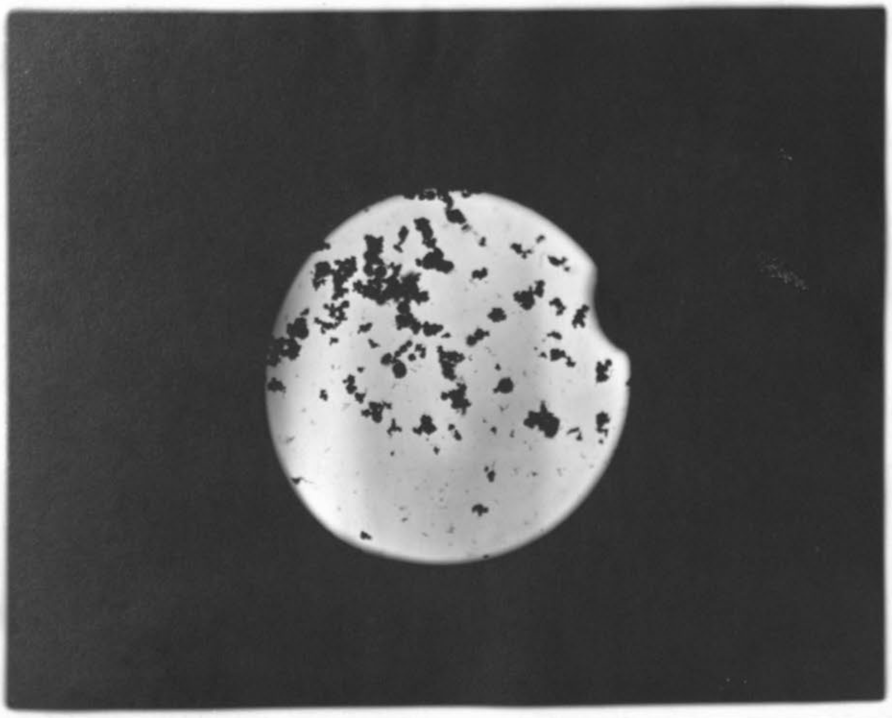


PLATE 2 - MERRELL-SOULE WHOLE MILK

Powder - Magnified 90 diameters

PLATE 3 - DRYCO PARTLY SKIMMED POWDER

Magnified 90 Diameters.

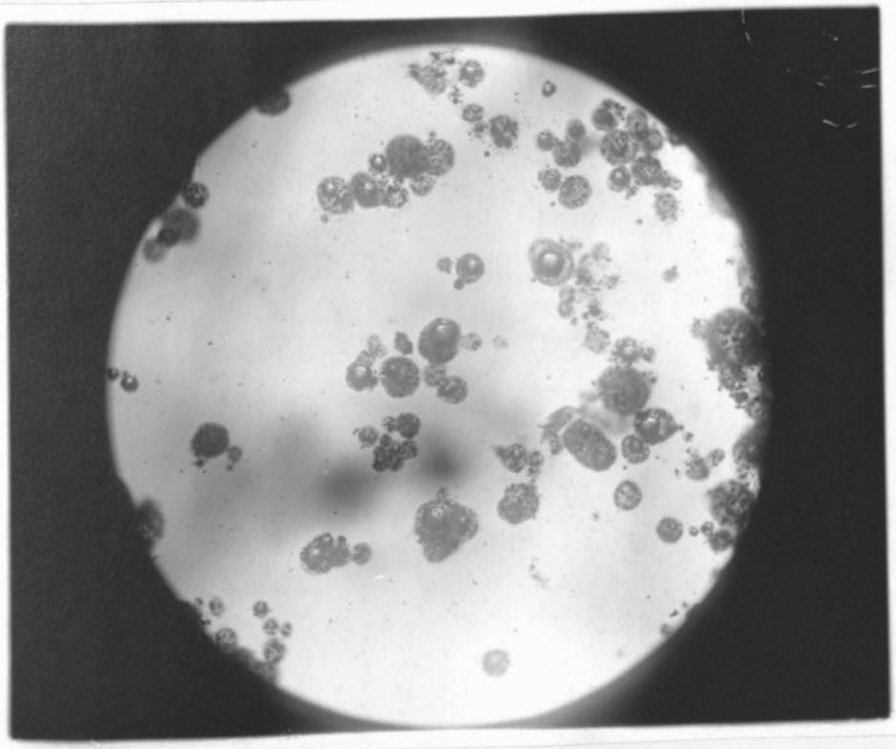


PLATE 4 - DICK'S POWDER SWOLLEN TO

SHOW AIR CELL AND FAT GLOBULES

Magnified 170 Diameters.

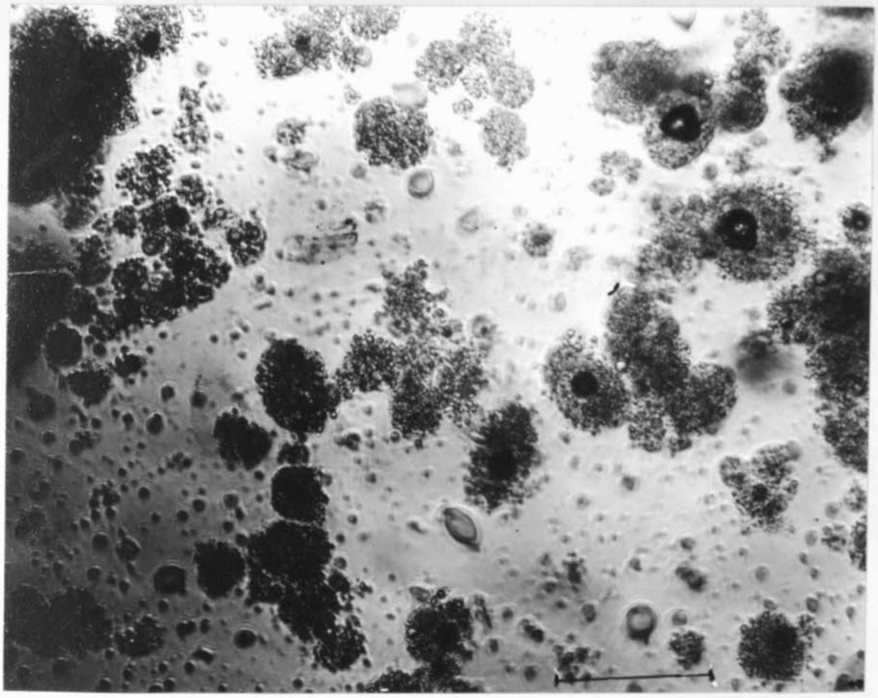


Plate 5 - Merrell Soule Powder

Magnified 330 Diameters to Show
Air Cell and Fat Globules.

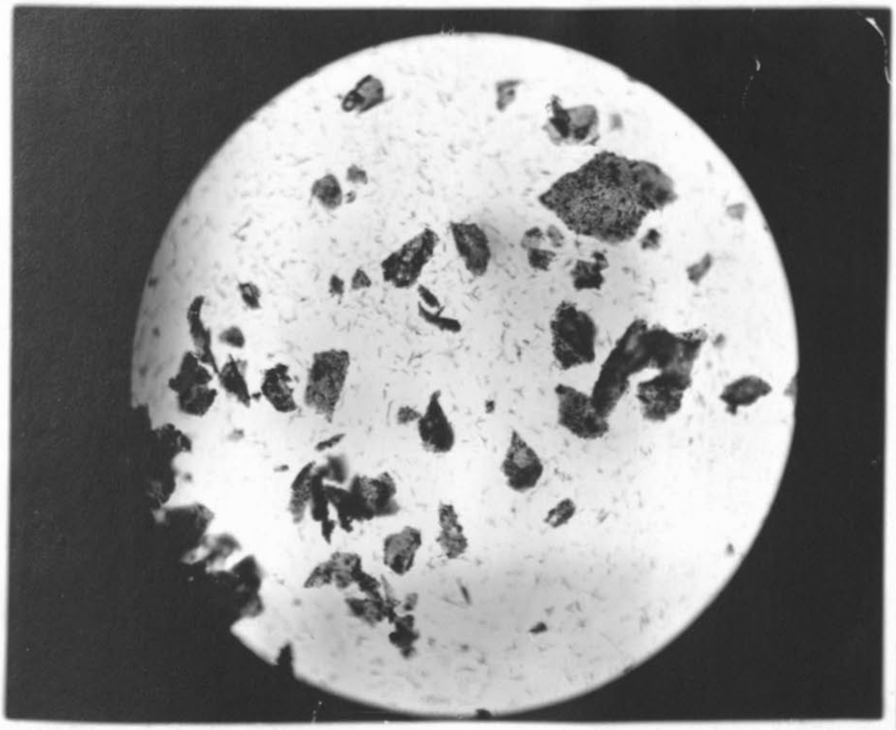


Plate 6 - Dryco Powder Magnified

170 Diameters to Show Fat
Globules - No Air Present.

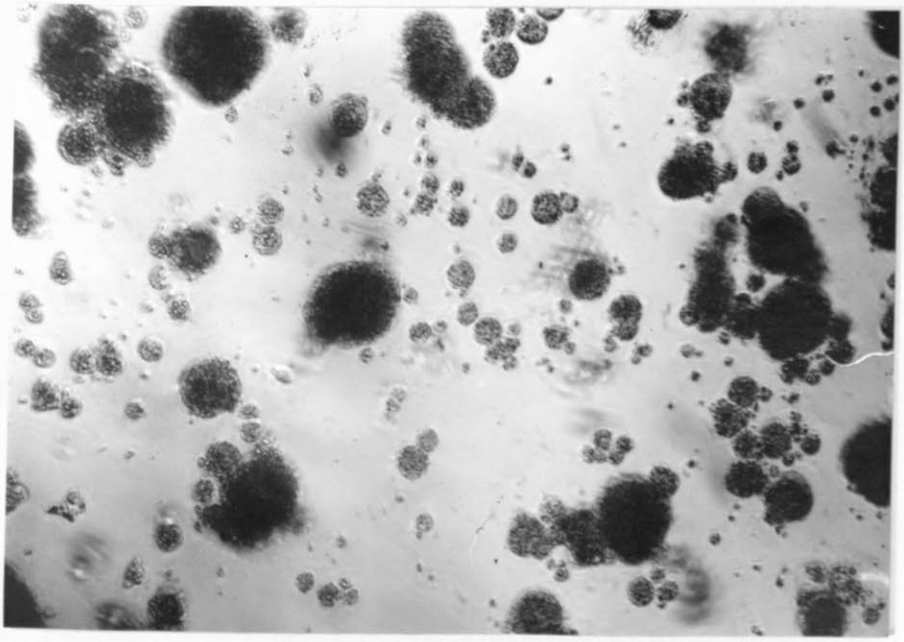


Plate - 7 Merrell-Soule Powder Magnified

170 Diameters to Show Absence of Air
Due to Heating of the Powder.

Plates I, II and III show the size and shape of the particles of Dick, Merrell-Soule and Dryco powders respectively.

Plates IV, V, and VI show the same powder grains in a swollen condition and under higher magnification. A decided difference may be observed in the structure of the different powders. Fat globules may be seen in all powders, and the air cell in the Dick and Merrell-Soule powder.

Plate VII shows the effect of heating on the number of air cells. This powder was heated for several hours at 100°C.

Physical Properties of Remade Milk

Freezing Point of Remade Milk.

The freezing point is one of the least variable of the physical properties of milk. In 1895 Winters(65) pointed out that the freezing point of milk was -.555°C., with very little variation. He also added that any addition of water causes the freezing point to approach nearer to that of water. Monier-Williams(66) (1914) holds that the freezing point of fresh milk is not affected by the removal of fat, or the addition of normal skimmilk, but is raised by the addition of water. Hortvet(67) (1920) lately showed that the freezing point of fresh cow's milk is approximately -.55°C., figuring water as freezing at 0°C. An average of 75 samples showed a freezing point of -.548°C.

The knowledge of the freezing point of milk has added greatly in detecting adulterations with water. It may also aid dairy men who are using reconstituted milk in obtaining a product which resembles fresh cow's milk in the proportions of ingredients.

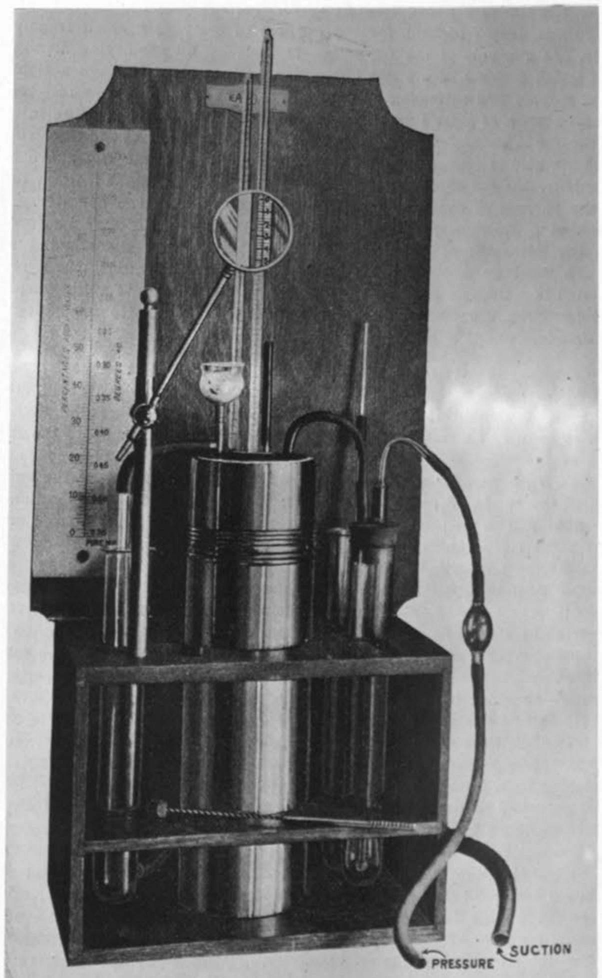
The purpose of this experiment was to determine the freezing point of remade milk to see how closely it resembles the freezing point of fresh milk, when using the proportion of powder and water set forth in the directions by the companies making the powders. In the case of the Dick and Merrell-Soule whole milk powders the proportion was 1 pound of powder to 7 pounds of water, giving approximately 12.5 per cent solids. In case of the Dryco which is partly skimmed, the proportion was 1 to 8, giving approximately 11 per cent solids in the milk. In case of the synthetic milk the following ingredients were used to give 25 pounds of milk testing 3.5 per cent fat, namely: water, 21.67 pounds, sweet cream butter 1.04 pounds, skimmilk powder 2.29 pounds.

A Hortvet Cryoscope was used in making the freezing point determinations. This cryoscope was designed primarily to enable freezing point determinations to be made on milk and other fluids within a short time. The cryoscope is described as follows:

A Dewar flask of one liter capacity is encased in a metal casing, tightly closed by a cork. Through the center of the cork is inserted a tight fitting glass tube. Into a small opening on one side of the cork is inserted a small air inlet tube, while on the opposite side is another tube to permit the escape of vapor. Toward the back of the cork a control thermometer is inserted. The freezing test-tube is inserted into the tight fitting glass tube, enough space being allowed between the two glass tubes to enable a thin film of alcohol to separate them. This freezing test-tube is closed at the top by a rubber stopper through which pass the finely graduated thermometer and a stirrer.

Procedure:-

400 c.c. of ether are measured into the cutlet opening of the Dewar flask. From 30 to 35 c.c. previously boiled distilled water are measured into the freezing test-tube. The water is then cooled by passing dry air through the apparatus, which causes the evaporation of the ether. The air supply is derived from a rubber bulb or a blower attachment. The ether vapor escapes through the outlet opening in the cork. The passage of air is maintained through the apparatus until the mercury on the control thermometer reaches -2.5°C., with occasional stirring of the water. When the mercury column on the finer graduated thermometer reaches approximately 1.2° below that of the probably freezing point of water, the water freezes and the column will rise. The thermometer



HORTVET CRYOSCOPE.

PLATE 8 - HORTVET CRYOSCOPE

USED IN MAKING FREEZING

POINT DETERMINATIONS.

4-21 6M

is then tapped gently and when the mercury column is stationery the reading is made, which will be the freezing point of the water. The water is then removed and a determination made of the freezing point of the milk. When the milk has been super-cooled to approximately 1.2° below that of its probable freezing point a small piece of ice is inserted into the milk test-tube to start the freezing. When the observed freezing point of the milk is determined, the difference between it and the observed freezing point of the water gives the freezing point of the milk.

Table I shows the results of the freezing point determination of samples of milk under investigation in comparison with a determination of the freezing point of normal cow's milk.

Table I

Kind of Milk	Observed freezing Point of Water	Observed freezing Point of Milk	Depression of Freezing Point
	0°C	0°C	0°C
Normal cow's	.045	-.51	-.555
Dick	.045	-.455	-.500
Merrell-Soule	.045	-.500	-.545
Dryco	.045	-.610	-.655
Synthetic	.040	-.520	-.560

It will be seen from Table I that Dryco showed a concentration while the Dick milk showed an excess of water. The other milks gave practically normal freezing points. It is evident that when Dick's milk and Dryco are reconstituted according to directions set forth by the manufacturers, the ingredients are not in the correct proportion to give a normal freezing point.

The next experiment was to determine what amounts of powder and water are required to give a normal freezing point. This was done partly for the purpose of securing the data necessary for remaking the milks for subsequent experiments.

Table II shows the proportions of ingredients required to give a normal freezing point for the four types of remade milk under investigation.

Table II

Kind of Milk	Amount of Ingredients for 100# Milk: Powder	Water	Butter (84% fat)	Observed Freezing point of water	Observed Freezing point of milk	Depression of freezing point Δ
				0°C	0°C	0°C
Dick	12.8	87.2		.04	-.508	-.548
Merrell-Soule	12.5	87.5		.045	-.500	-.545
Dryco	10.6	89.4		.04	-.510	-.550
Synthetic	9.16	86.68	4.16	.04	-.520	-.560

The Specific Gravity of Remade Milk

A normal freezing point as was obtained in the previous experiment is an indication of a correct proportion of water and solids of milk. Excessive water gives a higher freezing point, and also a lower specific gravity.

The purpose of this experiment was to determine the specific gravity of remade milk when using the same proportions of solids and water that were used to give a normal freezing point. The specific gravity of each sample was taken at 60°F two hours after being reconstituted.

Table III gives the specific gravities of the remade milks and a pasteurized milk which was run as a check.

Table III

Milks Used	Specific Gravity
Pasteurized	1.0321
Dick	1.0314
Merrell-Soule	1.032
Dryco	1.0336
Synthetic	1.0314

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It will be observed that all the milk fell in range of normal milk, when normal milk is taken between the limits of 1.029 and 1.034. Considering the specific gravity of normal milk as being 1.032 we find that the Merrel-Soule milk is closer to normal than the others. All but the Dryco are under 1.032.

The Viscosity of Remade Milk

Kobler[68] holds that the viscosity of milk is not as heretofore generally regarded, dependent chiefly on the fat content, but is greatly influenced by the casein, in that it falls considerably when the protein is separated from the completely fat free milk. Babcock and Russell[69] pointed out that "the consistency of milk and cream is made up of two factors, one dependent upon the inherent characteristics of the solution (the milk serum) and the other belonging to the matter suspended in this solution (fat, casein, etc.)

The effect of the first factor is termed viscosity in order to discriminate between substances in solution and in suspension. The combined effect of these two factors is what makes the body or consistency of milk and cream."

These writers conclude that the consistency or body of milk and cream is due to:

"1. Viscosity of the serum imparted by solids in solution.

2. The mechanical state of suspended substances (fat, etc.). The casein exerts a greater influence on consistency than fat and in milk is the chief factor."

In their work with pasteurized milk Babcock and Russell found that heat had produced a lowering of the consistency. The consistency did not return altogether when the milk was cooled.

The purpose of this experiment was to determine the viscosity of remade milk as compared to pasteurized milk, to note if the process of powdering had in any way affected the constituents so as to cause a change in viscosity. As has been stated before in the review of literature, the casein is altered in certain of the processes, particularly the drum process, and since viscosity is due partly to the casein a change in viscosity may be expected.

The milks were reconstituted in the usual manner, and the determinations made at a temperature of 25°C. After the determination was made the milks or samples from the same milks were set aside at a temperature ranging from 3°C to 10°C for 24 hours, after which they were warmed to 25°C and the determinations repeated.

A Mc Michael viscosemeter was used but the calibrated disc was rotated as in the Doolittle(70) or Mojonnier method. Four readings were made on each sample. These readings were then calculated in terms of centipoise or absolute viscosity units. The viscosity of the remade milks is given in Table IV.

Table IV

Kind of Milk Used	Temperature	Viscosity	Viscosity after 24 hours
	0°C	Centipoise	Centipoise
Pasteurized	25	1.39	3.23
Dick	25	2.25	4.10
Merrell-Soule	25	1.79	3.64
Dryco	25	7.36	8.91
Synthetic	25	1.94	2.29

It will be observed that the viscosities of all the milks are higher than the viscosity of pasteurized fresh milk. The viscosity in all cases increased on standing at a low temperature for 24 hours.

Electrical conductivity of Remade Milk.

The knowledge of the electrical conductivity of milk has been used in attempting to detect added water in milk. Durand and Stevenson(71) (1918) showed that, while added water decreased the conductivity, the results obtained in their investigations were not uniform enough to warrant the conclusions that added water could be rapidly and accurately detected in this manner. These writers show that the electrical conductivity of whole milk ranges from .00513 to .00560 at 25°C., while milk which has stood in an ice box two days has a conductivity ranging from .00521 to .00755, the increase being due to changes in composition of the milk.

Durand and Stevenson stated that fat and protein of milk decrease the specific conductance by decreasing the number of ions between the electrodes. The skimmilks run showed an increased conductance over that of whole milk.

Jackson and Rothera(72)(1914) showed that high sugar content goes with lower conductivity, and also that there is a diminution of 2.76 per cent conductance for every 1 per cent protein in the milk. These authors quote others as showing the electrical conductivity at 25°C. to range from .0043 to .0056. Peterson is quoted as stating that there is no "proportionality between specific gravity or total solids and electrical resistance."

Coste and Shelbourne(73)(1919) showed that the electrical conductivity of milk increases up to a certain point with the development of acidity. They found no well defined correlation between electrical conductivity and total solids and total ash, but a marked correlation between electrical conductivity and the chlorine content of the milk.

The purpose of this experiment was to note the electrical conductivity of remade milk as compared to a sample of fresh pasteurized milk. The milks were reconstituted in the usual manner. The cell constant was determined for the cell used and found to be .34202. The specific conductivity was calculated fromthe bridge reading and the resistance, which were obtained by balancing the resistance in the cell against the resistance box using a Wheatstone Bridge for the final adjustment.

The formula for determining the specific conductivity is as follows:

$$\text{Specific Conductivity} = \frac{A \times \text{cell constant}}{B \times R}$$

In which
(A = 10000 - B
(B = Bridge reading
(R = Resistance in Ohms.

Table V gives the electrical conductivity of the milks.

Table V

Milks Used	Number of Determinations	Temperature °C	Specific Conductivity
Pasteurized	4	25	.00547
Dick	8	25	.00560
Merrell-Soule	8	25	.005449
Dryco	12	25	.006227
Synthetic	8	25	.005699

With the exception of Dryco the milks show very nearly the same conductivity, and would be classed as normal.

The Creaming Ability of Remade Milk.

Many milk consumers judge the quality of milk by the volume of cream which rises on standing. This is an erroneous method and many milk dealers are unjustly accused of not producting a milk up to the standard fat requirement. It is not at all uncommon to find two samples of milk of the same fat content showing different percentages of cream. These milks may not have received the same processing. It has been shown that the cream line of milk is influenced by various factors, and that the cream line is not an evidence that the milk is not of good quality.

Vanderleck(74)(1917) obtained results which point out that the temperatures to which milk is heated affects the cream line. Clarification also has an effect. Both tend to reduce the volume of cream. Vanderleck found that agitation and heat gave a reduction of the amount of cream.

Kilbourne(75)(1915) summed up the causes which contribute to the loss of cream line on pasteurized milk as follows:

1. "The temperature to which the milk is heated.

2. The length of time which the milk is held at the high temperature.

3. The temperature of the heating medium with which the milk comes in contact during the heating process.

4. The clarification of milk.

5. The type of apparatus used in treating milk.

6. The amount of agitation to which the milk is subjected, especially when hot".

Along with some of the causes of variations in the volume of cream, Hammer(76) (1916) lists pasteurization and homogenization. In general, efficient pasteurization decreases the cream layer in milk, but the decrease need not be serious if pasteurization is properly carried out and the milk properly held afterward. Homogenization does not give a definite cream layer.

Richmond(51) points out that when milk is heated to 70°C the cream line is only about one-half that of fresh milk.

Many of the investigators found that the fat globules in a rapid rising cream line are larger and in many cases appeared in clumps, while those of slow rising cream are smaller.

In the remade milks it would seem that the creaming ability should suffer due to the heating processes through which the milks pass, and the mechanical disturbances experienced in some processes.

The purpose of this experiment was to observe the creaming ability of the remade milks. The remade milks were reconstituted in the usual manner and observed after standing 12 hours in a common refrigerator. The milks were allowed to cream in 100 c.c. graduated cylinders and the cream line was measured as percentage of total volume.

The results are recorded in Table VI.

Table VI

Milks Used	Per Cent Cream Line at 10°C	Remarks
Pasteurized	14.5	
Dick	13.	Sharp,distinct cream line
Merrell-Soule	0	No cream line. Fat homogenized.
Dryco	2.5	From partly skimmed milk powder
Synthetic	3.0	Very rich cream,nearly butter

The Dick milk was the only milk that gave a cream line which resembled that of normal pasteurized milk.

Other Physical Appearance on Standing.

It has been generally observed that spray made powders are more soluble in water than drum dried powders. Not only do spray powders require less agitation in being reconstituted, but after reconstitution the solids-not-fat do not precipitate or settle out from the milk serum as is generally the case of drum made powders. These statements are in keeping with observations made by the writer. Not only did the different types of powders differ in solubility but a difference in solubility was noted between the powders made by the spray process. The Dick powder was somewhat less soluble than the Merrell-Soule powder. After reconstitution there appeared a foam, consisting of insoluble matter, on the surface of both milks derived from spray powders. A large volume of foam was in evidence on the surface of the Dick milk. When a glass of Dick's milk was shaken slightly, small insoluble particles could be seen adhering to the sides of the glass. There appeared to be none in the case of the Merrell-Soule milk and synthetic milk.

There was always a separation of solids-not-fat in the case of Dryco milk. After having stood over night one could observe three distinct layers existing in a sample of milk. There was a layer of casein at the bottom of the container, a layer of water in the center, and a layer of cream at the surface.

When a glass of Dryco milk was shaken slightly, large, insoluble flakes could be seen adhering to the sides of the glass. There was no foam on the surface of the Dryco or the synthetic milk.

Chemical Properties of Remade Milk

Soluble Proteins of Remade Milk.

The albumin and globules of milk are usually regarded as the soluble proteins of milk, although, as a matter of fact recent evidence shows that they, like the casein, exist in colloidal solution. Together the albumin and globulin constitute about .7 per cent of cow's milk, of which albumin forms the bulk. Heat coagulable protein is another term often applied to these proteins. It is true that they are coagulated by heat of 72°C to 76°C., but only partially.

Investigators in many instances have calculated the amount of soluble protein in milk on the basis of the amount coagulated by heat. This method is incorrect, according to Palmer[77] (1919). He showed that only a part of the soluble proteins are precipitated by heat. In many cases only about half of these proteins are precipitated, the amount of heat coagulable protein depending upon the kind of preservative used, acidity of solution, length of time the solution is boiled, and other conditions.

The amount of albumin and globulin in a given amount of reconstituted milk may be an index to the amount of heat which has been applied to the powder going into this milk. In the spray process where the temperature of the drying chamber ranges from 180°F to 240°F we should expect to find a great deal of the heat coagulable proteins coagulated, provided that the fluid milk has attained that temperature. However, in the spray process where the milk enters in a very fine spray and is mixed with the hot air, drying takes place immediately. It is therefore probable that the evaporation of moisture is so rapid in this process that the milk is actually kept cool until a dry state is reached, due to the ab sorption of heat in vaporizing the water. After the milk has been dried there is less danger of injury from heat, as chemical changes produced by heat are much more severe in the presence of moisture.

In case of the milk from drum-dried powders we would expect to find less coagulable protein present, since the mass of liquid milk comes in direct contact with the drum which is heated by steam to 212°F. The fluid milk is in contact with the drum but a few seconds, but this time is probably sufficient to enable the temperature of the milk to reach near the boiling point before the milk is dried. The result would be the coagulation of some of the albumin before the milk had been dried sufficiently to decrease the effect of the heat on the coagulable proteins.

The purpose of this experiment was to measure the amount of "soluble" proteins present in a given amount of remade milk, as compared to the amount present in normal milk. The difference between the amount present in normal milk and the amount present in remade would be the amount coagulated by heat during the processes undergone. The amount of coagulated proteins would be in proportion to the heat to which the liquid milk had been subjected. The milks were remade in the usual manner and the following procedure carried out.

10 c.c. of remade milk were measured into a beaker and diluted with 90 c.c. of distilled water. This was heated to 42°C, and 1½ c.c. of 10 per cent acetic acid was added to coagulate the casein. (The pH was taken at this point and observed to be at the isoletric point of casein.) After stirring and allowing to stand five minutes, the casein was filtered off. This casein was washed with distilled water which was made up to have a pH of 4.8. The filtrate was neutralized to a faint pink with N/10 NaOH, using phenolphthalein as an indicator.

50 c.c. Almen's Tannic acid reagent were added to the filtrate to precipitate the soluble proteins. This was permitted to stand for an hour, after which the precipitate was filtered off, washed, and transferred to Kjeldahl flasks and the Nitrogen determined in the usual manner. The results obtained were in

terms of milligrams of Nitrogen in 10 c.c. of milk. Duplicate samples as well as two blanks were run.

Table VII gives the data regarding the amount of soluble protein in terms of milligrams of nitrogen in the sample in comparison with the average soluble protein nitrogen of fresh milk.

Table VII

Sample	c.c. N/14 HCl	c.c. N/14-NH_4OH	Mg.N in Blank	Mg.N in sample
Fresh Milk				10 to 12.0
$Blank_1$	25	24.2	.8	-
$Blank_2$	25	24.15	.85	-
$Dryco_1$	25	16.95	.83	7.22
$Dryco_2$	25	16.95	.83	7.22
$Dick_1$	25	13.10	.83	11.07
$Dick_2$	25	13.15	.83	11.02
Merrell-$Soule_1$	25	15.50	.83	8.67
Merrell-$Soule_2$	25	15.20	.83	8.97
$Synthetic_1$	25	16.10	.83	8.07
$Synthetic_2$	25	15.90	.83	8.27

From the data given in Table VII it appears that the soluble proteins were effected more by the heat of the drum method than of the spray method, which bears out the statements made in a previous paragraph regarding the temperatures probably attained by the powder in the two types of powdering processes.

Rennet Coagulability of Remade Milk.

Cow's milk coagulates with rennet to form a firm, massed curd. The rapidity of coagulation depends on certain factors, such as acidity, temperature and amount of soluble calcium salts in the milk. If a milk has been heated to pasteurizing temperature it will coagulate more slowly than the fresh milk. It is not definitely settled as to the cause of the slower coagulation, but the common belief has been that the soluble calcium salts are changed to insoluble salts.

In the review of literature it was stated that where remade milks were coagulated with rennet a curd resembling human milk was obtained from certain powders. The curd was much finer and did not form in a hard mass.

The purpose of this experiment was to note if the ability of remade milk to coagulate with rennet had been altered by the process of powdering and reconstituting. The type of curd was noted and compared with the curd formed when raw and pasteurized milk were used. If the curd formed resembled raw or pasteurized milk there would be a possibility of making cheese from powdered milks.

A Marshall rennet can was used to make the speed of coagulation. This can holds nearly a quart of milk. On the inside wall of this cup or can are graduated spaces beginning with zero at the top and extending by half divisions to 10 near the bottom. In the bottom of the cup is a metal tube with a very small bore. This cup is used by cheese makers to indicate when the milk is ready for the addition of rennet. Rennet is added to the milk and when it ceases to flow through the outlet in the bottom of the cup, the number of spaces uncovered is observed.

In this experiment the milk was warmed to 86°F and placed in the cup, while holding the fingers over the outlet. Two c.c. of rennet were diluted to 20 c.c. and added to the milk while stirring. The finger was removed immediately and the number of spaces uncovered at the time of coagulation was noted.

Table VIII gives the number of spaces as measured by the Marshall rennet cup.

Table VIII

Milk Used	Temperature	Spaces Uncovered	Remarks
Normal Raw	86	3.75	Firm clot.
Normal Pasteurized	86.5	5.00	Firm Clot.
Dick's	86	5.25	More flocculent at first Later became more firm.
Merrell-Soule	86	5.50	More flocculent than pasteurized. Not so firm as Dicks.
Dryco	86	all	No coagulation. Very fine state of casein.
Synthetic	86	all	No coagulation. Casein very fine state.

Table VIII shows that Dick's resembled pasteurized more closely than the others, and there were indications that it may be used in cheese making. Dryco failed to coagulate at all, as did the Synthetic.

The Buffer Value of Remade Milk.

According to Van Slyke and Baker (78) "there are many compounds which have the property of effecting the results of the determination of the hydrogen ion concentration. When acid or alkali is added to a solution containing such compounds, the change in hydrogen ion concentration is found to be less than would be expected for the known amount of acid of alkali added. Any substance which tends to prevent change in the original hydrogen ion concentration of its solution, when an acid or base is added, is called a buffer or regulator. Proteins salts, etc. may exercise such an effect. These effects must be determined for individual cases under specific conditions of concentration, temperature, etc. In the case of milk, the compounds acting as buffers are protein, phosphates, citrates and carbonates."

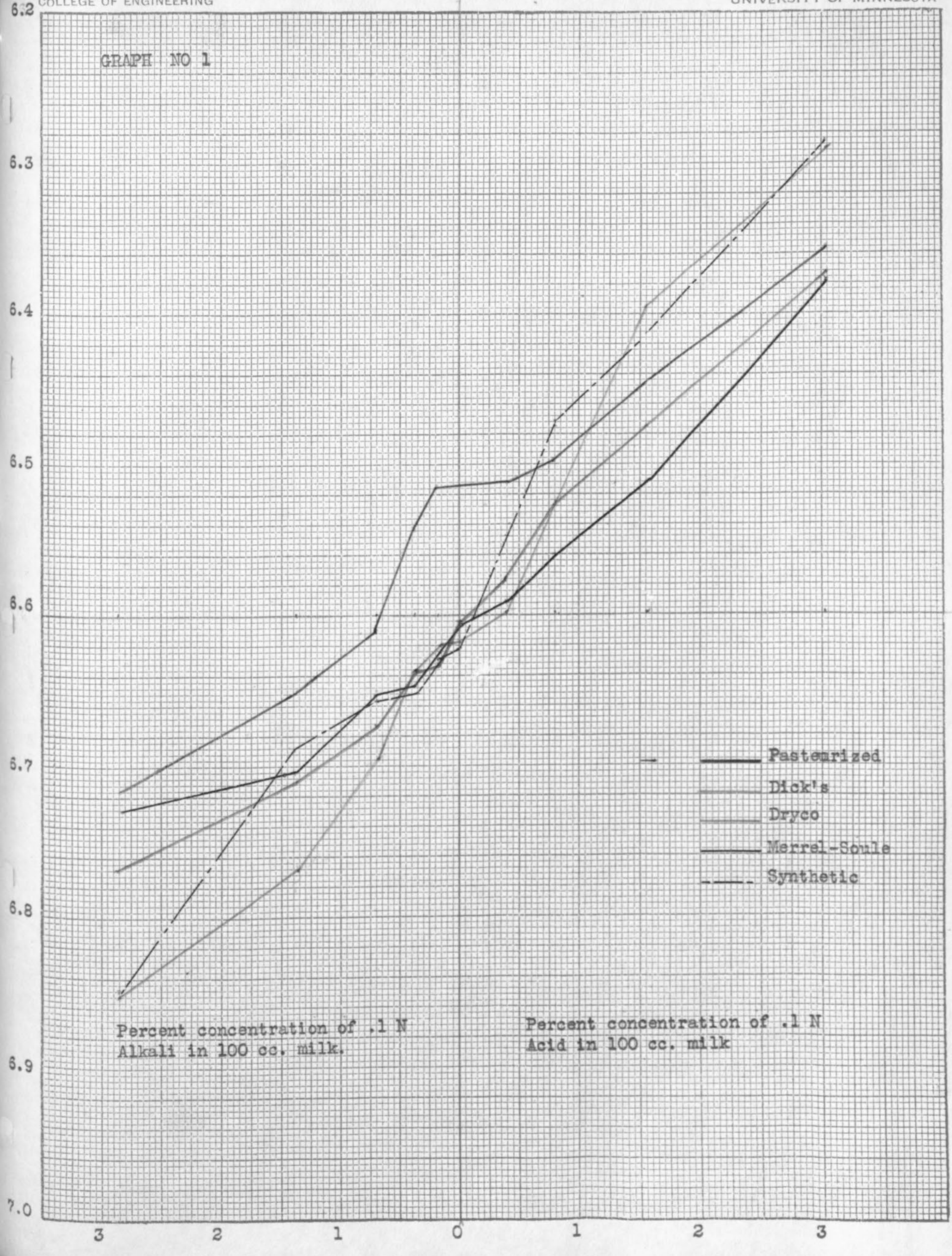
It was the purpose of this experiment to note the buffer value of remade milk when acid and alkali of known strength were added to the milk. It was ex-

Table IX

Amount of acid and alkali added to 25 c.c. Milk	Pasteurized Acid PH	Pasteurized Alk. pH	Dick Acid PH	Dick Alk. pH	Dryco Acid PH	Dryco Alk. pH	Merrell-Soule Acid PH	Merrell-Soule Alk. pH	Synthetic Acid PH	Synthetic Alk. pH
0	6.5141	6.5141	6.6185	6.6185	6.6049	6.6049	6.6066	6.6066	6.621	6.621
.1	6.5124	6.5158	6.5998	6.6202	6.5794	6.6338	6.5964	6.6287	6.549	6.629
.2	6.4988	6.543	6.5260	6.6355	6.5294	6.6372	6.5607	6.644	6.472	6.650
.4	6.4444	6.610	6.3951	6.6950	6.475	6.6780	6.4937	6.6542	6.418	6.657
.8	6.357	6.6508	6.289	6.768	6.3706	6.7120	6.3764	6.7120	6.285	6.690
1.6	6.1472	6.7171	6.0928	6.8521	6.120	6.7654	6.1608	6.7297	6.102	6.850
3.2	5.8109	6.8538	5.8058	6.993	5.785	6.9327	5.7497	6.9514	5.730	6.989
6.4	5.3284	7.1323	5.3233	7.3591	5.2852	7.2707	5.2920	7.2945	5.297	7.380
9.6	4.9234	7.5145	4.850	7.7668	4.9183	7.7430	4.7942	7.8168	4.878	7.940
12.8	4.4664	8.021	4.3892	8.7468	4.3 62	8.4830	4.3858	8.7145	4.494	8.699

The pH values of Table IX are plotted to show more clearly the buffer effects of the remade milk toward acid and alkali. It will be noted that the pH values of all the remade milks are very nearly the same at 0.

The amounts of acid and alkali added are plotted in terms of concentration of tenth normal acid and alkali in 100 c.c. milk. These were calculated from the amounts added to 25 c.c. of milk and are listed in Table X.


pH
6.2
6.3
6.4
6.5
6.6
6.7
6.8
6.9
7.0
GRAPH NO 1
Pasteurized
Dick's
Dryco
Merrel-Soule
Synthetic
Percent concentration of .1 N
Alkali in 100 cc. milk.
Percent concentration of .1 N
Acid in 100 cc. milk
3
2
1
0
1
2
3

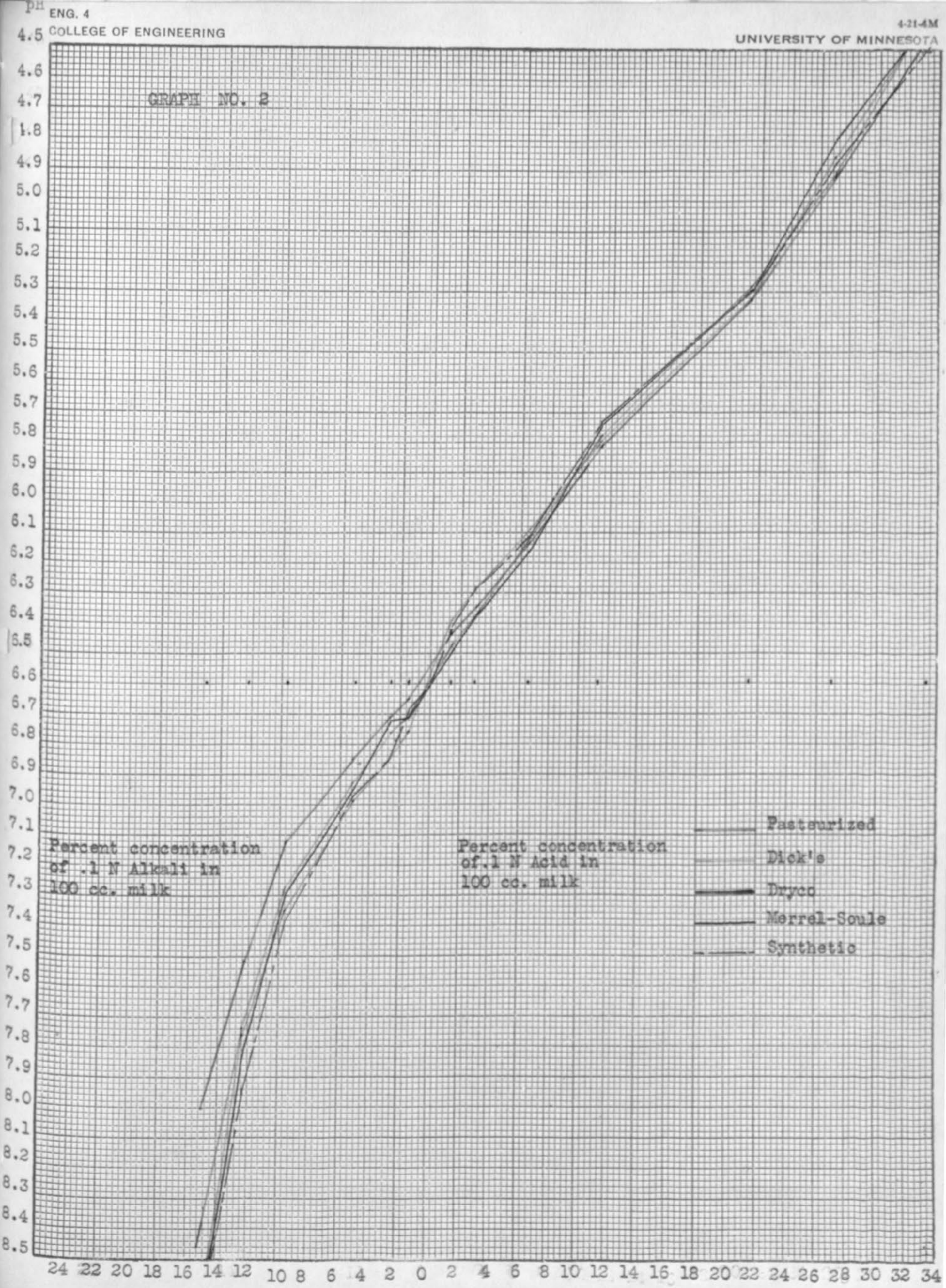

pH
GRAPH NO. 2
4.5
4.6
4.7
4.8
4.9
5.0
5.1
5.2
5.3
5.4
5.5
5.6
5.7
5.8
5.9
6.0
6.1
6.2
6.3
6.4
6.5
6.6
6.7
6.8
6.9
7.0
7.1
7.2
7.3
7.4
7.5
7.6
7.7
7.8
7.9
8.0
8.1
8.2
8.3
8.4
8.5
Percent concentration of .1 N Alkali in 100 cc. milk
Percent concentration of.1 N Acid in 100 cc. milk
Pasteurized
Dick's
Dryco
Merrel-Soule
Synthetic
24 22 20 18 16 14 12 10 8 6 4 2 0 2 4 6 8 10 12 14 16 18 20 22 24 26 28 30 32 34

Table X

c.c. acid added to 25 c.c. milk	c.c. 0.1 N Lactic Acid in 100 c.c. milk	c.c. Lime water added to 25cc milk	c.c. 0.1 N $Ca(OH)_2$ in 100 c.c. milk
0	0	0	0
.1	.398	.1	.167
.2	.793	.2	.352
.4	1.570	.4	.698
.8	3.080	.8	1.370
1.6	6.390	1.6	2.840
3.2	11.330	3.2	5.040
6.4	21.500	6.4	9.560
9.6	27.700	9.6	12.320
12.8	33.800	12.8	15.040

Graph I shows the effect of concentration of acid and alkali up to about 3 per cent plotted on a sufficiently large scale to show the buffer value of these low concentrations of acid and alkali.

Graph II shows the buffer value of the same sample with higher concentrations of acid and alkali. On the acid side there is but slight deviation from the normal curve. On the alkaline side at the maximum concentration the remade milks show less buffer effect than the normal pasteurized milk.

The Keeping Qualities of Milk Powders

Coutts[15] made the observation that milk powders deteriorated with age. This was especially true of the whole milk powders. He also observed that whole milk powders made by the drum method were less liable to deteriorate than those made by the spray method. The common deteriorations noted by Coutts and others were stated to be due to oxidation, although many have used the term "rancidity" to describe this type of deterioration. This is erroneous, since there are two distinct types of deteriorations, one known as rancidity in which hydrolysis of the fat is the cause of the "off flavor", the other known as tallowiness due to oxidation of the fat. The latter type is more commonly found than the former in powdered whole milks.

In this experiment observations were made of the various powders, to note the type of deterioration present. All the spray-made powders want "off flavored" in time, in most cases the cause being oxidation. In one instance a package of Dick's whole milk powder became typically rancid in about three weeks. Another sample of powder of the same make became rancid in about eleven months. This type of decomposition was not noticed however, in any of the other makes of powders.

The samples of Merrell-Soule powders under observation eventually became tallowy. Fresh cans of the original lot (9 months old) were tallowy to some extent but became much more so after being opened.

The drum-made powder, Dryco, did not show any signs of becoming rancid. The powder also failed to show any typical tallowy odor, even after ten months. It had become very slightly stale at this age, but the odor was not nearly so pungent as in the case of the spray process powders.

An attempt was made to test the powders for tallowiness, using the Kreis test. This is a chemical test for tallowiness in fat, and is apparently based upon the reduction of phloroglucin in the presence of HCl, by the oxidation products produced in the tallowy decomposition of fats. The reduction of phloroglucin is manifested by the production of a pink color.

In applying the Kreis test to milk powders it was found necessary to first extract the fat from the powders. This was done by remaking the milk in case of the Dick and Dryco powders, and then shaking by hand until butter had collected. The butter obtained was then melted and the pure fat filtered off. It was impossible to obtain the fat in this manner from the Merrell-Soule powder, as the fat in the dry powder was extracted by ether, after first coagulating the protein with alcohol. The ether was evaporated off leaving the pure fat. The Kreis test is carried out as follows: (79)

10 c.c. of the oil or melted fat are placed in a large test-tube and 10 c.c. of strong HCl (sp. gr. 1.19) added. The tube is closed and shaken vigorously for 30 seconds. 10 c.c. of a 0.1 per cent solution of phloroglucin in ether are added and the tube shaken as before. It is allowed to stand and if the fat is tallowy a red or pink color will appear in the acid layer.

Table XI gives the results of the Kreis test. Tallowy fats and fresh butter were run as controls with the fats from the milk powders.

Table XI

Fats Used	Age of Fat	Results of Test
Old animal fat		Strongly +
Fresh butter		-
Old tallowy butter		Strongly +
Dick	9 months	Strongly +
Dick	6 months	-
Dick	6 months in open container	Strongly +
Merrell-Soule	6 months	Faint - but +
Dryco	9 months	-
Synthetic	Fresh	-

It is noticed that the spray powders gave positive tests at the age of 6 months.

Synthetic milk gave a negative test, as the fat was fresh.

Dryco, drum-dried powder gave a negative test. This finding is in keeping with observations made by other investigators.

Enzyme Activity of Remade Milk.

Milk contains several enzymes which are active unless the milk has been heated above a certain temperature. It has been stated[80] that these enzymes are rendered inactive in milk by heating it above 172°F to 176°F.

In the powdering processes we find that the heat of the air cells and drums is usually above 172°F. If the liquid milk has been actually heated to the temperature of the drying chamber or drum we would expect to find the enzymes of the milk rendered inactive. But in case of the spray process, as previously explained, it is quite evident that the liquid milk does not attain the temperature of the cell. After the milk has become dried the danger of destroying the enzyme in the milk has been greatly lessened, as enzymes offer greater resistance to heat when in a dry state.

In the drum process we would expect to find the enzymes rendered inactive, since the liquid milk comes in actual contact with the drum which has been heated to 212°F. It is true that the liquid milk dries quickly and is on the drum but a few seconds, but it is also evident that the milk in being evaporated in this manner must have nearly reached the temperature of the drum.

There are several tests for heated milk which are based on the activity of these enzymes. The principle enzyme is peroxidase, which in the presence of peroxides such as hydrogen peroxide, sets free oxygen and this oxygen produces marked coloration when certain compounds are present. If milk is heated above 176°F. the peroxidase loses the power of setting free oxygen from a peroxide and therefore no color is produced.

The tests commonly in use for testing the activity of the peroxidase enzyme are the Storch test and Guaiac test.

In this experiment the activity of the peroxidase enzyme was noted, which gave an index to the amount of heat applied to the liquid milk during the process of powdering. A Storch test and Guaiac test were made. The tests may be described as follows:

Storch test:

From 5 to 10 c.c. of milk were measured into a test-tube, then a drop or two of a 0.2 per cent solution of hydrogen peroxide and about two drops of a 2 per cent solution of para-phenylenediamine were added. The mixture was shaken and permitted to stand. If the milk had not been heated above 172° or 176° F. a distinct blue color appeared.

Guaiac test:

From 5 to 10 c.c. of milk were measured into a test-tube, then about five or ten drops of guaiac tincture and the mixture shaken. If the milk had not been heated above 176°F a distinct blue color appeared. It is well to add a few drops of hydrogen peroxide in case a negative result is obtained, because some solutions produce no coloration even in unheated milk.

The results of these tests applied to remade milk are listed in Table XII.

Table XII

Kind of Milk	Storch Test	Guaiac Test
Normal Raw	quickly +	quickly +
Normal Pasteurized	" +	" +
Dick's	" +	" +
Merrell-Soule	slower +	" +
Dryco	-	-
Synthetic	slower +	quickly +

Dryco was the only remade milk which gave a negative enzyme test, which again bears out statements made concerning the temperatures probably attained by the powder in the two types of processes.

Discussion of Results

From the results obtained in this study it is quite evident that some changes have taken place in some of the physical and chemical properties of the milk during the drying process, although the remade milks resembled fresh milk in many instances.

The freezing points of the remade milks were not normal in all cases when the milks were reconstituted according to directions sent out by the companies manufacturing the powders. Dryco showed a higher depression which indicated a concentration. This was undoubtedly due to the fact that Dryco is made from partly skimmed milk. The partial skimming of milk causes a relative increase in the other solid constituents. It is the latter, particularly the dissolved mineral salts, which control the freezing point of milk. Dick milk gave a low depression or showed a slight adulteration which was probably due to one or both of two factors, (1) a high moisture content of the powder, (2) a lower total solid content of the original milk than the standard adopted by the manufacturer. Normal freezing points were obtained in case of both Dryco and Dick powders by varying the amounts of powder and water.

All milk gave a normal specific gravity when reconstituted in the proportions which gave a normal freezing point. The specific gravity of Dick's milk was somewhat lower than 1.032, and Dryco somewhat higher. This is due to the difference in fat content, as Dryco is partly skimmed.

The viscosity of each of the remade milks was higher than normal. This was due to the physical change which had taken place in the casein during the drying. When water is added to the powders the casein swells to a certain degree and causes the liquid to be more gelatinous and of higher consistency than normal milk. The data show that casein of Dryco is changed more by the process of manufacture than was the case with any of the other milks due to the intense heat of the powdering drum.

With the exception of Dryco the milks were normal so far as electrical conductivity was concerned. Conductivity is lowered by fat and protein and raised by salts. Skimmed milk gives a higher specific conductivity than normal milk. Dryco gave a higher specific conductivity due to the partial skimming of the fat, which not only decreased the fat content but relatively increased the mineral content, which are factors influencing conductivity.

The Dick milk was the only milk which gave a cream line of normal volume. The explanation of this lies in the fact that the spraying did not injure the ability of the fat to rise. The fat globules retained their normal size. The reason for the failure of the cream to rise on the Merrell-Soule milk is explained by the fact that the spraying process which exerts a pressure of 4000 pounds per square inch thoroughly homogenizes the fat. The failure of the small globules to rise is due to their surface being increased greatly in proportion to their volume. The resulting increase in tension of the surfaces of the fat globules causes an increased adsorption of the colloidal material of high specific gravity which prevents the rise of the fat globules. The Dryco mi lk being partly skimmed would not be expected to cream. The failure of the Synthetic milk (milk made from butter, skimmilk powder and water) to give a cream line had been observed by others and confirmed in the present study. This is due in part to the inability of the emulsifier to break down the fat into globules having approximately the same size as those of normal milk and also in the fact that there may be some tendency for the extra heating, to which the synthetic milk is subjected to cause the mechanical adhesiveness of the solids not fat to be lessened.

The analysis of the various remade milks for soluble proteins showed that these constituents had been lessened during the process. This shows that the heat in all instances must have reached 72°C. The Dryco milk contained the least soluble protein of any of the milks, which shows that the fluid milk is subjected to a higher heat in the drum method than in the spray method. The

Merrell-Soule powder contained less soluble protein than the Dick which is evidence that this spray process subjects the fluid milk to higher temperatures than the Dick spray process. The higher heat is probably administered either while the milk is superheated in the vacuum pan or while the dry powder is subjected to the continued blast of hot air throughout a "run" before being withdrawn from the powdering cell. The practice of both of these steps in the process was observed by the writer in one plant using the Merrell-Soule system.

The examination of the ability of the remade milks to form a clot with rennet showed that this property of milk had been destroyed in the case of Dryco and Synthetic milks. This is probably to be expected in the case of the Dryco in view of the other data showing the high heat to which the milk is subjected in this process. Apparently the casein has been changed in such a manner that it does not go back into its normal colloidal state on dissolving, so that when rennet is added, the paracaseinate, if it forms at all, does not clot. Small particles do form in the milk and these particles later precipitate, but never formed a clot. The reason for the failure of the Synthetic milk to clot was probably due to the additional heat of 145-150°F for 30 minutes during the process of reconstitution. This point, however, needs further investigation.

The study of the buffer values of the remade milks showed that at low concentrations of acid and alkali there was less buffer effect in remade milks than normal, especially on the alkaline side. The Dick milk showed the least buffer value throughout the complete cycle. On the acid side at low concentrations the Merrell-Soule and Dryco milks showed more buffer effect than normal milk. At high concentrations the buffer values were practically the same on the acid side. On the alkaline side the normal milk showed the greater buffer value at the highest point of concentration. The buffer values differ but slightly. The differences are probably due to the change in casein and salts which act as buffers. This experiment showed, however, that no foreign ingredients had been added to any of the milks.

The observations made on the keeping qualities of the remade milks showed that milk powders made by the spray process deteriorated more rapidly than those made by the drum method. The cause of the deterioration was due in most cases to oxidation of the fat. The reason for such rapid decomposition in case of the spray powders lies in the fact that more of the powder is exposed to air than in the drum powder. In the drum powder the only part exposed to the air is the outside surface of each particle, while in the spray powders both the outside and inside of each particle are exposed to air, because of the presence of air cell within practically all the particles.

The explanation of the cause of the air cell in the spray powders is not clear, although its presence in these types of powders is established. It is of interest to note that the powders made by centrifugal spray methods have much larger air cells than those in which the milk is sprayed into the drying chamber under hydraulic pressure. The cause of the air cell in either case is evidently related to the fact that the milk is dried very rapidly, in a large volume of hot air. It is possible also that someone of the steps in the spraying processes causes an incorporation of considerable air in the milk. The general picture of the dried powders at any rate suggests strongly the drying of very fine foam.

The peroxidase enzyme was present in all milks except Dryco. The presence of the enzyme shows that the liquid milk had not been subjected to a heat in excess of 172°F to 176°F. In case of Dryco the enzyme was absent which is proof that the liquid milk was subjected to a heat of 172°F to 176°F.

Summary and Conclusions

Four remade milks were studied in this investigation. Two were milks made from spray made whole milk powders, one from partly skimmed milk powder made by the drum method, and one made from skim milk powder, sweet butter, and water. These milks were studied from the standpoint of some of their physical and chemical properties. The individual powders were studied from the standpoint of their physical structure and keeping qualities.

I. It was found that the structure of the individual powder grains varied with the process of manufacture. Those made by the spray method were spherical and contained within the center an air cell, while the powder made by the drum method was irregular in shape and contained no air cell. Spray powders made by centrifugal spray contained more air than those spray under pressure.

II. Drum-made powders kept better than spray-made powders. The common decomposition noted was tallowiness of the fat due to oxidation. An occasional case of rancidity was noticed in the Dick spray powder. It is suggested that the poorer keeping quality of spray powder is to be attributed, in part, at least, to the presence of the air cell within the individual grains.

III. Dryco and Dick's remade milk failed to give normal freezing points when made up in the proportions set forth in the directions sent out by the companies manufacturing the powders. By varying the amounts of powder and water normal freezing points were obtained. Merrell-Soule and Synthetic milks gave normal freezing points.

IV. The specific gravity of the remade milk was normal when the milks were reconstituted in the proportions which gave a normal freezing point.

V. All remade milks gave a higher viscosity reading than normal. Dryco, from drum-made powder, showed a greater viscosity than any of the milks made by the spray process.

VI. With the exception of Dryco all milks were normal so far as electrical conductivity was concerned.

VII. The Dick milk was the only milk which gave a cream line of normal volume Merrell-Soule milk showed the least cream line.

VIII. The amount of soluble proteins present was greatest in the Dick milk, and lowest in the Dryco milk.

IX. Dryco and Synthetic milk did not coagulate with rennet. The Dick and Merrell - Soule coagulated but somewhat slower than pasteurized milk, and gave a curd which was somewhat more finely divided.

X. At low concentration of acid and alkali there was less buffer effect in remade milk than in normal, especially on the alkaline side. Dick milk showed the least buffer value throughout the cycle. On the acid side at low concentrations the Merrell-Soule and Dryco showed more buffer effect than normal milk. At high concentration the buffer values were practically the same on the acid side. On the alkaline side the normal milk showed the greater buffer value at the highest concentration of alkali.

XI. The peroxidase enzyme was rendered inactive in the case of drum-made powders, but was active in all the spray-made powders.

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