SCIENTIFIC SECTION

THE MICROANALYSIS OF MALTED MILK.*

BY C. W. BALLARD.

At first thought the application of microanalytical methods to the examination of malted milks seems rather impracticable and the work was undertaken more in the spirit of curiosity than with any real hope of definite results. Chemical methods, even those involving determinations of diastatic power and amino acids content, had failed to yield dependable information excepting as regards fat, protein, ash and moisture. Diastatic figures, even in authentic samples, were widely different. The fat, protein and ash content of a given sample would certainly establish whether whole or skimmed milk had been used in the preparation; but these results do not tell us if the sample is genuine or not. We are dealing with mixtures of varying composition and although limits of variation can be fixed, chemical methods alone will not give us the information we most desire, besides they are complicated and consume much time. A microanalytical determination of a malted milk sample can be fully completed by an experienced worker in less than one hour. Such a report will tell us what kind of milk and malt has been used, and what is more important, whether the sample is a standard processed article or an imitation mixture.

At the time this investigation was started there were no standards for malted milk. Consequently it would have been difficult to prove that several spurious articles sold under this title were misbranded. Prosecution would have involved the question of proper definition of the term "malted milk" and whether the word "malted" was really used in its adjective sense or merely as a trade name. With the issue of Food Inspection Decision 170 (*Service and Regulatory Announcement* 20, July 2, 1917), the proposition becomes less complicated, for malted milk is defined in this decision. Referring to the text of the decision we find the following:

> "Malted milk is the product made by combining whole milk with the liquid separated from a mash of ground barley malt and wheat flour, with or without the addition of sodium chloride, sodium bicarbonate and potassium bicarbonate in such a manner as to secure the full enzymic action of the malt extract and by removing water. The resulting product contains not less than seven and one-half percent (7.5%) of butter fat and not more than three and one-half percent (3.5%) of moisture."

It will be apparent from this statement, that the term malted milk, as used at present, is not merely a trade name for the product of a few manufacturers. Products now sold under this title must fulfil certain specifications. I might add that very few of the samples examined would comply with the above requirements. Malted milk is not a *mixture* of various malt products and powdered milk, but is a preparation in which the malt and milk have been changed by process of manufacture so that the resultant differs in chemical constitution as well as microscopical characters, from the substances used in its preparation. The term "processed" will be used here to designate the standard malted milk which fulfils the requirements of the above decision.

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* Read before Scientific Section, A. Ph. A., Indianapolis meeting, 1918.

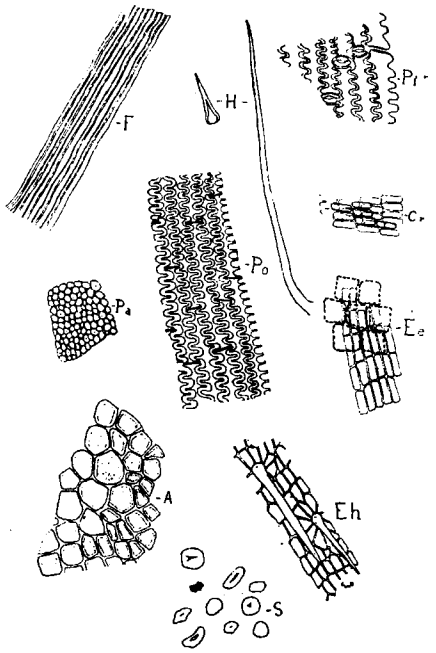

FIG 1

Powdered Malt.

F, Fiber; H, Hairs; Pi, Inner epidermis of palet; Po, Outer epidermis of palet; Pa, Parenchyma; Cr, Cross cells; Ee, Epicarp overlying spermoderm; Eh, Palet tissue with hairs; A, Aleurone cells; S, Starch.

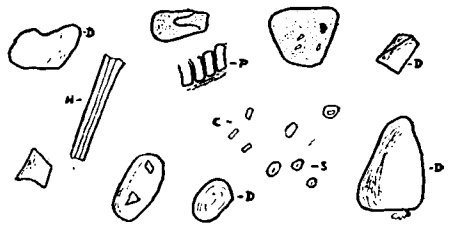

FIG. 2

Dried Malt Extract.

D, Extract or diastase masses; H, Malt hairs; S, Starch; P, Palet tissue of barley.

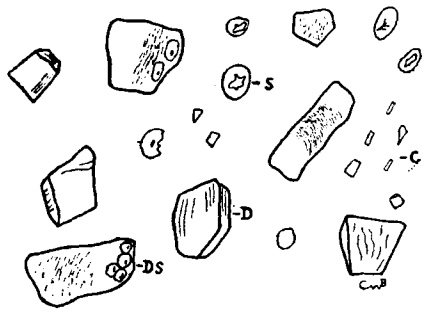

FIG. 3

Diastase.

D, Diastase masses; S, Starch; C, Crystals; DS, Diastase mass with starch.

In the preparation of specimens for examination, temporary mounts are best. Glycerin jelly mounts are very poor as even slight heating alters the appearance of the sample. Glycerin, alcohol and water mixture (1 : 1 : 1) is the best medium if specimens are for immediate use. Permanent slides may be prepared by using glycerin, or better still, petroleum oil as a mounting medium. Oil mounts are necessary in every instance as sugars and water-soluble constituents dissolve and are apt to be overlooked. As a preliminary to the microscopical examination, it is absolutely essential that the analyst be thoroughly familiar with the differences in appearance and structure of the various materials used in the manufacture of malted milk. Milk powders and malts vary widely because of different processes of manufacture. The use of dried malt extracts and various fillers add to the difficulty of the work. The materials found by the writer to enter into the manufacture of malted milks, genuine and otherwise, are powdered skimmed milk, powdered whole milk, powdered malt, dried extract of malt, diastase, bread products, glucose, cane sugar, various starches, gelatin and gums. The microscopical characters of each of these substances will first be described.

SKIMMED MILK POWDER.

The process used in the preparation of skimmed milk powder will have great influence upon its color and microscopical appearance. The milk may be evaporated to dryness in steam kettles, vacuum pans or by various devices which force the

milk in spray form over heated rollers. The color of powdered milk prepared in an open steam kettle is golden yellow to dark orange depending upon the degree of heat employed in the operation. It is rather difficult to reduce this milk to fine powder. Vacuum pan and spray dried milks are nearly white and are very finely subdivided. Manufacturers prefer a milk dried by this method as it is more readily soluble than an open dried milk. The individual particles of a skimmed milk powder prepared by vacuum or spray process, are for the most part spherical, although irregular masses are also found. Each particle is probably an aggregation of fat globules held together by solidified albuminous material. Some of the masses may be likened in appearance to enormous polynuclear leucocytes, the fat globules corresponding to nuclei. Many of the forms show a central portion of irregular outline almost filling the mass and containing numerous fat globules. A few of the particles are without apparent fat globules but appear to be filled with a granular substance. Other forms, especially those of irregular outline, are crossed by radiating or branching striations. Worthy of note is the fact that lactose will begin to crystallize in glycerin or water mounts within two hours and will proceed to such an extent as to fill the entire slide. The constancy of this crystallization is such that it becomes an excellent indication of skimmed milk in many preparations.

WHOLE MILK POWDERS.

The methods employed in preparing whole or full milk powders are identical with those discussed under skimmed milk and give rise to the same variations in appearance and color. Open dried full milks are deeper in color and even more impossible of reduction to fine powder. Spray dried full milks are more of a cream color than skimmed milks of similar preparation but are as finely subdivided as the latter. Full milks are not as readily mixed with water as the skimmed product, or as it would be put in the trade, they are not as soluble. This fact has been given as a reason why so many manufacturers of imitation articles use skimmed milk powder instead of whole. Incidentally the skimmed product is cheaper. The particles of a spray or vacuum dried full milk are smaller than those of skimmed milk and more regularly spherical in form. Irregular masses are comparatively scarce. The greater number of the masses do not show the fat globules as distinctly as in skimmed milk. For the most part the masses are of granular appearance and pearly lustre. Several masses may be adherent and give the appearance of budding from a main particle. A few show one or two fat globules embedded in the granular substance. Lactose does not crystallize from water or glycerin mounts of whole milk as readily as it does from skimmed milk. Crystallization may be delayed for a day or even longer.

A few preparations are manufactured from open kettle dried milks. This form of milk powder, skimmed or whole, is difficult of description but fairly easy to recognize. The milk particles are in the form of irregular fragments having fat globules scattered over their surface. In full milks dried by this method, the fat globules are very large and almost cover the surface of the fragments. Open dried skimmed milks have smaller and fewer fat globules and many of the fragments of albuminous material are free from adhering globules.

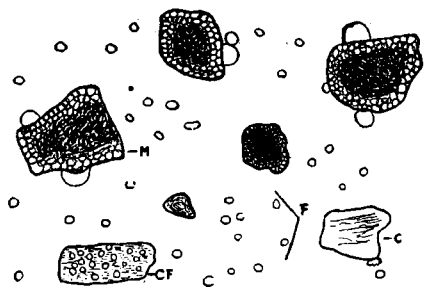

FIG. 4

Dried Milk (Open Heating).
M, Milk masses; F, Free fat globules; C, Coagulated material; FC, Coagulated material with fat globules.

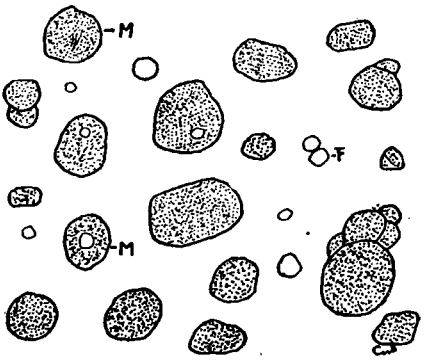

FIG. 6

Whole Milk (Vacuum Dried).
M, Milk masses; F, Fat globules.

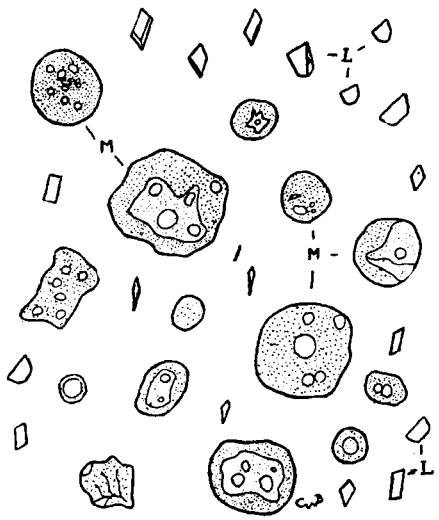

FIG. 5

Skimmed Milk (Vacuum Dried).
M, Milk masses; L, Lactose crystals.

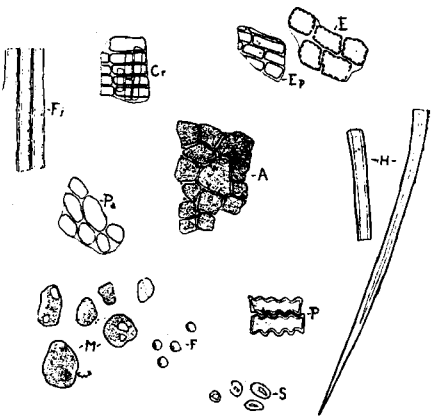

FIG. 7

Standard Malted Milk.
Fi, Fiber from malt; Cr, Cross cells from malt; E, Epicarp cells from malt; Ep, Epidermis of spermoderm from malt; Pa, Parenchyma of malt; A, Aleurone cells of malt; H, Hairs of malt; P, Palet cells of malt; S, Starch; M, Milk masses; F, Fat globules.

MALT POWDERS AND PREPARATIONS.

It is common practise with many producers of so-called malted milks to use various malt powders or preparations and merely mix these with certain quantities of powdered milk. A preparation of this sort would be more appropriately named "Malt *and* Milk" and is entirely different in composition and microscopical appearance from a standard malted milk which complies with the specifications of Food Inspection Decision 170. In examinations of malted milks one will encounter one or more of the following malt products: powdered malt, powdered dried malt extract and diastase. Descriptions with illustrations of these materials in powdered form are here given.

Malt Powder.—Powdered malt exhibits most of the elements of barley although some of these have undergone changes in the malting process. The elements

present, in the order of their relative amounts are: (1) Starch; (2) hairs; (3) outer epidermis of palet; (4) aleurone cells of endosperm; (5) inner epidermis of palet; (6) cross cells of pericarp; (7) parenchyma of endosperm; (8) fibrous tissue; (9) epicarp tissues; (10) tube cells.

The starch is more or less changed by the action of amylolytic ferments during malting. Many of the grains appear to have been eaten away, the central portion or hilum being the first part to undergo digestion; such grains are hollowed out at this point. Other grains show destruction of the outer portions. These partially digested grains when treated with aqueous iodine solution, give the purple to red reaction of amylodextrins and maltodextrins. Depending upon the degree to which the malting process has been carried, we find more or less unchanged starch. Many of the grains showing no physical change fail to give the blue reaction with iodine.

The hairs are of two kinds; short thick-walled and long thin-walled varieties being present in about equal amount. They are usually broken, but the relative thickness of wall to width of the lumen, serves to distinguish one from the other. These hairs were found in every sample of malt and malt product examined.

The outer epidermis of palet is distinguished by the thick-walled wavy cells common to many plants of the Graminaceae family. Between the wavy cells are stoma with guard-cells tightly fitting and almost occluding the actual opening. We also find in this tissue, very short, thick-walled, warty hairs and hair scars.

Aleurone cells of the endosperm are white, thick-walled, angled and closely packed with dark granular nitrogenous material.

Inner epidermis of palet consists of white, thin-walled, long and more or less angled cells. Short thick-walled hairs and nearly square stoma with small but well-defined apertures occur in this tissue.

Cross cells of pericarp are usually adherent to the spermoderm or coat of the endosperm. They are arranged in two layers, one heavy, the other light, consisting in both cases of rectangular, thin-walled cells with or without intercellular spaces. Both layers extend in the same direction and are at right angles to the underlying spermoderm cells.

Parenchyma of endosperm is thin walled, rounded in outline and may or may not contain starch.

Fibrous elements, if occurring singly, may be mistaken for thin-walled hairs, but all fragments of this tissue were in well defined groups, sometimes with remnants of the vascular tissue attached.

The epicarp cells are similar in structure and appearance to those of the inner epidermis of the palet; hairs are present but stoma appear to be lacking.

Tube cells were not apparent in any sample of malt subjected to examination.

Powdered Dried Malt Extract.—This form of malt is probably used more than any other in the manufacture of mixed products sold under the name of malted milk. A great disadvantage is that it is extremely hygroscopic and preparations containing it must be kept very dry or caking will occur. Upon standing in a moist atmosphere for any length of time, secondary fermentations take place and the product assumes a disagreeable odor. It is fairly soluble and when mixed

with skimmed milk powder and used at the soda fountain, produces a beverage without much sediment, of clean white color and even texture. The drink may even have a better appearance than if a standard malted milk had been used.

The elements of note in dry malt extracts are: (1) diastase masses; (2) starch; (3) crystals; (4) tissue fragments; (5) hairs.

The diastase masses are irregular, angled fragments of light yellow color, striated and very soluble. The exact character of this material can only be studied in oil mounts.

Starch is present in small amounts and most of the grains show the effects of diastatic action.

Crystals are very small and for the most part are rectangular in shape with a few of rosette form.

Tissue fragments are mainly loose parenchyma and broken walls of aleurone cells.

Hairs are of types characteristic to malt.

MIXED AND PROCESSED MALTED MILKS.

The Food Inspection Decision definition of malted milk requires that enzymic action shall have taken place and shall have modified the constituents of the mixture. In other words the compound shall have been processed. Malted milks prepared by mechanically mixing certain quantities of powdered milk with malt or malt preparations, no enzymic action having taken place, are conveniently referred to as "mixed malted milks" in distinction to the "processed" products just mentioned. Although patent rights have expired, the details involved in the manufacture of the processed products are not by any means public property and are nearly impossible to obtain from successful producers. Sufficient to say that considerable experience and mechanical equipment is necessary in the work. From the appearance of certain samples, even before the definition was published, I had decided that in the process of manufacture enzyme action had produced certain changes in the materials used. The malt starch was almost entirely digested and not over two or three grains were present in a field. The milk was not in spherical masses containing fat globules embedded in albuminous material although free fat globules were numerous and well distributed. It is certain that in processing, the amylolytic ferments of malt convert the starch to sugars and it may be that the small amounts of proteolytic enzymes in malt have an action upon the albuminous materials of the milk causing their disruption or rendering them soluble in some other manner.

Mixed malted milks are readily identified by the presence of unchanged fat masses, yellowish malt extract particles and the relative scarcity of malt tissues. One must search several fields to locate hairs, starch or other malt elements. Dextrose and soluble particles of malt extract will be visible until dissolved. Oil mounts are necessary for prolonged examinations of these soluble materials. One of the samples submitted for examination was found to be a mixture of powdered malt and milk and displayed all the characteristic elements of both materials. Such combinations are rare and because of the large amount of starch present are not readily soluble or miscible.

The characteristic elements in a standard or processed malted milk, in the order of amounts present, are: (1) free fat globules; (2) milk masses; (3) parenchyma; (4) aleurone cells; (5) palet tissue; (6) malt hairs; (7) diastase masses; (8) starch.

Fat globules are very small, are well distributed throughout the field and are not in aggregates or masses.

Milk masses for the most part are irregular and not spherical in form. Embedded fat globules are present in some of the masses but are almost hidden by the granular substance.

Parenchyma occurs as small, much broken fragments of thin-walled cells.

Aleurone cells withstand the malting process and show very little change from those described under malt.

Palet tissue remains unchanged during processing and is described under malt.

Hairs although much broken are otherwise unchanged.

Diastase masses are similar to those described under dry malt extract. They dissolve rapidly in glycerin or water mounts.

Starch is present in very small amount and the grains show but little change from those of malt.

SUBSTITUTE OR SPURIOUS MALTED MILKS.

Although by far the greater number of spurious malted milks upon the market are merely mixtures of powdered milk, dry malt extract and dextrose, other substances may be present. Chief among these added materials are various thickeners. The beverage prepared with a standard malted milk is apt to be somewhat thicker in consistency than that prepared with the imitation. To overcome this defect the producers of substitutes often add baked bread products, corn starch and possibly gelatin and gums. Powdered bread crumbs are most used for this purpose as they are cheap and mix rapidly and well with cold water. Starches, gelatin and gums work best in beverages prepared with hot water. Some of the labels attached to these imitation products show much ingenuity in avoiding direct statements that the article is malted milk. The presence of glucose is often accounted for in labelling by the statement that the material contains a corn product. Bread crumbs have been covered by the general term cereals. In some cases the substitute is sold under various titles framed for no other purpose than evasion of misbranding ordinances.

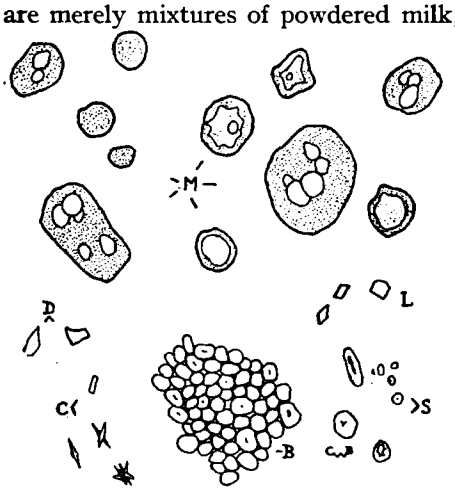

FIG. 8

Substitute Malted Milk.

M, Milk masses (skimmed); D, Diastase or extract masses; L, Lactose crystals; C, Crystals from malt extract; S, Starch; B, Powdered bread.

The following tabulation of microanalyses is sufficient to show the extent to which sophistication is carried in the manufacture and sale of malted milk:

Sample.	Sold as	How prepared.	Form of malt.	Form of milk.	Source.
1	Malted milk	Mixed	Extract	Skimmed	Dairy
2	Malted milk	Mixed	Extract	Skimmed	Manufacturer
3	Malted milk	Mixed	Extract	Skimmed	Manufacturer
4	Malted milk	Mixed	Extract	Skimmed	Manufacturer
5	Malted milk	Processed	Infusion	Whole	Pharmacist
6	Malted skim milk	Processed	Infusion	Skimmed	Manufacturer
7	Malted whole milk	Processed	Infusion	Whole	Manufacturer
8	Malted milk	Mixed	Extract	Skimmed	Manufacturer
9	Malted milk	Mixed	Powdered	Whole	Manufacturer
10	Malted milk	Mixed	Extract	Skimmed	Manufacturer
11	Malted milk	Processed	Infusion	Whole	Confectioner
12	Malted milk	Mixed	Extract	Skimmed	Manufacturer
13	Malted milk	Mixed	Extract	Skimmed	Manufacturer
14	Malted milk	Mixed	Extract	Skimmed	Manufacturer
15	Malted milk	Processed	Infusion	Whole	Confectioner
16	Malted milk	Mixed	Extract	Skimmed	Dairy
17	Malted milk	Processed	Infusion	Whole	Confectioner
18	Malted milk	Processed	Infusion	Whole	Confectioner
19	Malted milk	Processed	Infusion	Whole	Confectioner
20	Malted milk	Mixed	Extract	Skimmed	Confectioner
21	Malted milk	Mixed	Extract	Skimmed	Confectioner
22	Malted milk	Mixed	Extract	Skimmed	Manufacturer
23	Malted skim milk	Mixed	Extract	Skimmed	Manufacturer
24	Substitute[1]	Mixed	Extract	Skimmed	Manufacturer

[1] Contains bread crumbs.

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THE STABILITY OF CANNABIS SATIVA AND ITS EXTRACTS.

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A recent publication by Eckler[1] regarding the deterioration of *Cannabis indica* raises a question which can be answered positively only after a long series of experiments such as Eckler himself conducted.

Over a period of five years, samples of crude drug cannabis were kept under observation, storing it under different conditions. He found that it loses from 1 to 2 percent of its activity monthly, depending apparently on the temperature of the storage room. Five years from now, however, the subject will probably have passed so completely out of general interest that there will be no incentive either to complete the experiments or to make the results public, while at the same time such an apparently authoritative statement going unchallenged may lead to a number of errors. It seems advisable, therefore, to publish some data bearing on this question even if it is not based on systematic experiments. In the course of nearly 20 years' experience in applying the physiological assay process to cannabis preparations, a number of unrelated facts are gradually collected which, taken as a whole, have a value not to be ignored.