
UNIT 6 SAMPLING OF MILK AND MILK PRODUCTS

Structure

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6.0 OBJECTIVES

After going through this unit we should be able to:

- 1 identity the different types of sampling equipments;
- 1 distinguish between chemical and microbiological sampling;
- 1 labeling of samples; and
- 1 preservation and transportation of samples.

6.1 INTRODUCTION

Milk is liquid product and sampling from a can or tank or a tanker may not be that difficult. This is common perception, which is wrong. Milk Fat being lighter separates out as cream layer on standing and if cream is not mixed properly the test result of such a sample will definitely be wrong. Vigorous mixing churns fat resulting in an improper sample. In Homogenized milk such problems may not occur. Milk in polypacks from different batches require batchwise sampling. If the milk is unhomogenized during filling from the storage tank variation in fat can be noticed between the initial and last filled packets. To avoid this during filling operation the speed of the agitator (to avoid churning of fat) has to be monitored and also frequent testing of milk for fat% has to be carried out. A calibrated electronic milk tester can be used for monitoring fat%.

In the case of products like milk powder, butter, ghee etc. in packs/containers where large number of units are present, it is better to use statistical methods for sampling.

Sampling can be carried out properly if one understands the product composition. In liquid milk fat being lighter tends to come to the surface on standing. To get a homogenous milk sample, proper mixing has to be done. Without a proper sample, testing is a wasteful exercise.

6.2 SAMPLING

i. Sampling Personnel:

Proper sampling depend mainly on the capabilities of sampling Personnel and they should be:

- 1 Authorized
- 1 Properly equipped
- 1 Properly trained for drawing samples for chemical and bacteriological analysis.
- 1 Free from infectious diseases.
- 1 Physically and mentally fit.

ii. Sample

The term “sample” not only refers to the operation of obtaining representative material but also to the units selected, as well as to the portion analyzed. Of particular importance is the fact that the testing laboratory does not ordinarily become involved in the process until it receives the physical sample(s).

A random sample is one where every unit in a population has an equal chance of being incorporated into the selected sample. Adequate numbers to be drawn as per requirements. Haphazard way of sampling and also sampling at convenient positions/points should be avoided.

iii. Involvement of Laboratory in Sampling

Sampling of food is usually the responsibility of a separate group of personnel in the laboratory. In commercial and public Laboratories the sampling personnel are generally not mixed with the testing personnel. Despite the separation, testing personnel usually can, and should, participate in preparing instruction, particularly with regard to the more scientific aspects of the number of units that should be taken, where they should be taken, how much material should be in each sample unit, the type of containers to be used, and how the samples should be transported to the laboratory.

Despite the availability of instructional manuals, the sample collector must have a basic understanding of the principles of sampling to avoid common mistakes, which could result in an invalid or non-reproducible measurement.

Different values obtained with different samples from the same consignment are unfortunately often ascribed to poor analytical work or to unsatisfactory methods of analysis rather than to the generally more likely cause of either improper sample and/or inherent variability of the lot.

iv. Sampling Equipment Modification

The varied properties of foods, and the physical states and mixture, in which they are found, require that a sampling person carry many types of sampling equipment. Guiding factor for sampling equipment should be taken from National and

International Standards. But in many cases, sampling persons must improvise and use that equipment is available to them. It is up to the testing personnel to review such improvisations to be sure that substitutions do not introduce such substantial sampling errors as to invalidate the analytical results.

v. Sealing and Labeling

- 1 Nature of product
- 1 Date_____ Time_____
- 1 Batch No./Code No.
- 1 Manufacturing place
- 1 Purpose of sampling
- 1 Sample taken from
- 1 Sample Code
- 1 Condition of sample – Storage condition of product at the time of sampling.
- 1 Directions for storage
- 1 Care during transport
- 1 Sampled by – Name & Designation
- 1 Place samples to be sent

vi. Sample Container

Sample containers should be inert and capable of being closed/sealed and should not allow further contamination. Glass containers are good, except that they are fragile and can break if not handled properly. If light may alter a component, opaque containers must be used or transparent ones must be wrapped in an opaque material, such as aluminium foil or brown paper. Plastic bags can also be used in some cases. Solid or semi-solids require wide mouth bottles. For Liquids – narrow mouth bottles can be used.

vii. Preservation of Samples

The most convenient method for preserving samples prior to transportation and storage is with chemical preservatives. On the label of such samples the details of the preservative with name and quantity have to be mentioned.

When applicable, frozen storage (eg., Ice creams) is an excellent method for preserving samples. However, freezing ruptures cells, and enzyme activity thereafter may be substantially accelerated during de-frosting/warming. Samples for microbiological analysis should preferably be transported at less than 7°C.

viii. Microbiological Sampling

Sampling equipment and sample containers should be sterilized. It is done by autoclaving (120° C for 20 mts) or by keeping in hot air oven (100° C for 2

hours). While carrying these material care should be taken to avoid contamination. Immersion of sampling equipment in rectified spirit or ethyl alcohol or ignition in alcohol are also suggested before sampling. Because of these extra precautions it is advisable these samples are not considered for organoleptic evaluation.

ix. Storage and Transportation of Samples

During storage/transportation, three types of changes which most likely to occur are:

1. Changes in composition of samples through evaporation or absorption of moisture, evaporation of other volatile constituents, oxidation, etc.
2. Changes in composition of samples through action of enzymes (especially through activity of hydrolytic enzymes)
3. Changes in composition of samples through action of microorganisms.

Therefore bacteriological samples are to be stored and transported in cold condition, Dry samples to be stored in less humid conditions. Storage instructions on the label has to be read thoroughly and followed.

Ideally, samples should be analyzed promptly after collection, but rarely it is possible. Hence adequate care should be taken in storing samples at the laboratory before analysis.

x. Milk Sampling Equipment

i) Manual agitation in small vessels

For mixing liquids in small vessels (e.g. in buckets and cans) a stirrer normally known as plunger is suitable. A plunger is disc with holes with a SS rod welded in the middle of the disc as a handle. The length of the rod and the diameter of the disc depend on the size of the container. Normally 1 meter length plunger is used.

ii) Manual mixing in large vessels

Two meters length plunger is used for mixing milk in road/rail tankers.

xi. Dippers

Dipper is a SS tumbler with a handle welded on one side. Dippers of holding capacity ranging from 50 to 200 ml. are available in the market. Depending on the depth of the container suitable dipper can be used.

xii. Borers

Borers of sufficient length to reach the bottom of the product container. The borer shall be made entirely of polished stainless steel. The edge of the borers is sharp and serve as a scraper.

xiii. Butter Triers

Butter triers of sufficient length to pass diagonally to the bottom of the product container, and of dimensions suited for the purpose envisaged.

xiv. Cheese Triers

Cheese triers of shape and size appropriate to the cheese to be sampled.

xv. Spoon, Knief or Spatula Or Ice Scoop

Smooth surface and of sufficient size.

xvi. Cutting Wire of Sufficient Size and Strength.

- 1 Preferably of SS, no crevices.
- 1 Surfaces smooth

xvii. Use of Sampling Equipment

- 1 Agitators for mixing liquids in bulk shall have a surface sufficient to produce adequate mixing of the products. In view of the different shapes and sizes of containers, no specific design of agitators can be recommended for all purposes, but they shall be designed in such a way as to avoid damage of the inner surface of the container during mixing. The extent of mixing depends on the period of time over which the milk has been at rest.
- 1 The product to be mixed in the tank or vessel determines the technical characteristics and construction of built-in agitators.
- 1 Removable agitators are usually provided with a propeller and are introduced into transport, road and rail tanks through the manhole.
- 1 It is recommended that the stirrer be inclined 5° to 20° as this allows vertical mixing of the liquor as well as horizontal movement.
- 1 Thoroughly mix the milk by stirring or plunging (Plunger) in small vessels, milk buckets and cans.
- 1 In milk tanks or vats mechanically agitate the milk for at least 5 min. until sufficient homogeneity is obtained. If the tank is equipped with a periodical, time-programmed agitation system, sampling may be carried out after only a short duration of agitation.
- 1 In those instances where the propeller of the agitator is close to the surface of the milk, do not use the agitator since this is likely to lead to the formation of foam or churning.
- 1 Mixing using a plunger or a removable agitator to be used in road, rail tanks or vessels of similar size shall be performed as follows:

When samples are taken within 30 min. after filling the container, mix the milk for at least 5 min. by plunging or stirring with an agitator, when the milk has been stored in the tanker for a longer period of time, mixing shall be extended to at least 15 min.

- 1 In a large vessel with a bottom discharge outlet or a sampling cock installed at another place, there may be, at the discharge outlet, a small quantity of milk, which is not representative of the whole contents even after mixing.

Accordingly samples shall preferably be taken through the manhole. If samples are taken from the discharge outlet valve or the sampling cock, discharge sufficient milk to ensure that the samples are representative of the whole.

- 1 The efficiency of the method of mixing applied in any particular circumstances shall be demonstrated as being adequate for the purpose of the analysis envisaged; the criterion of mixing efficiency is the repeatability of analytical results from different parts of the whole, or from the outlet of the tank at intervals during discharge. To be precise samples from top, middle, bottom or any part of the container should show the same test results.
- 1 Samples should be proportionate to the quantity in the container

xviii Sampling of Same Products

i) Silo/Storage Tank

Depending on the capacity, agitation is done with the help of mechanical agitator fitted into the silo/tank. Time of agitation varies depending on the capacity. After the agitation is complete, allow the flow of 2 to 3 liters of milk through sampling cock into a bucket. Rinse the sample bottle with fresh flow of milk. Drain the milk into the bucket. Then draw sample. Always draw samples in duplicate. The bottles have to be properly labelled before drawing the samples. Adequate precautions have to be taken before drawing sample for microbiological testing.

ii) Road Tankers

Check the conditions of the seals. Open the manhole lid. Observe for any foreign matter. If it is free from foreign matter, plunger for about 15 minutes in all directions. Avoid mixing of air into milk. Take samples as explained in 'Silo/Storage Tank'.

iii) Milk Powder

0.0 After every 70 bags, select one bag of powder for sampling. Normally one truck load of milk powder contains 15 MT.

iv. Butter

After every 70 cartons, select one carton of butter for sampling. Normally one truck load of butter contains 15 MT.

v. Milk Powders/Butter

For containers upto 25 Kgs :

- 1 Select 4 samples in 100 containers/Packs. Take about 200 grams powder of sample . One is kept for microbiological purpose.
- 1 For composite sample add 50 grams of powder from each sample. Mix well and take 200 grams as sample..
- 1 Always draw sample in duplicate. In case of doubt the second sample can be analysed.

Bottles should be sterile and air tight. On the bottles mark clearly whether it is for chemical examination or for microbiological examination

vi. Ghee

In the trade 15 Kgs tins are popular. One tin for every 50 tins may be sampled.

All the containers in a single consignment belonging to the same batch of manufacture shall be sampled together.

Sampling tubes made of stainless steel should be used for large containers.

If the contents are in molten condition gently make the tin upside down 5 to 6 times and then draw a sample.

If the contents are in solid a semisolid condition use the sampling tube as a borer and skillfully pull out the contents from the entire depth of the container. Ensure all the three portions- bottom, middle and top layers are properly represented in the sample.

Take individual samples from each container. Draw about 50 ml or 50 gm sample from each individual sample and put into a sterile dry container. Melt to about 40°C. Mix well and draw two samples. Each sample should be about 200 ml

vii. Products like Ice-cream, Ice Candies, Lollies and other frozen products are always stored under frozen condition(less than minus 18 degree.C). Such samples should preferably be transported in frozen condition to the laboratory for testing. Dry ice can be used.

viii. Dried milk

If the drier is running continuously, draw samples at an interval of 30 minutes and test for

colour, flavour , taste and moisture. More moisture than the dairy standard means less shelf life.

In the case of small packs of one litre or below like milk, flavoured milk, ice cream, table butter, ghee etc. it is always safe to take the entire pack as a sample. Follow batch wise sample. For every 500 units draw one sample.

Check Your Progress – 1

1. Why sampling of milk is difficult?

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2. What are the criteria for sampling personnel?

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3. What is the sample?

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4. Why Laboratory has to be involved in sampling?

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5. What are the conditions for sample container?

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6. What temperatures are used to sterilize sampling equipment for micro biological purpose?

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7. What are the likely changes that occur in samples during storage and transportation?

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8. What are the various types of sampling equipments?

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9. How different milk products are samples?

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6.3 LET US SUM UP

Sampling is an art. Sample should represent the entire product and should be adequate for analysis and repeat analysis. It should be done by trained personnel only.

Testing of each units and confirming its quality is ideal. Remember story of Sabari offering fruit to Lord RAMA! In the Industry this type of testing is not possible and hence a structured system has to be followed.

6.4 KEY WORDS

- Agitation** : Mixing of liquid using suitable sampling equipments like plunger.
- Dipper** : A SS Tumbler with a handle and a spout.
- Plunger** : SS Disc with holes and a long handle welded in the middle of the disc.
- Labeling** : A paper containing all the required details of the sample.

6.5 SOME USEFUL BOOKS

IS 11546:1999/ISO 707:1997 Milk and Milk Products-Guidance on sampling.

6.6 ANSWERS TO CHECK YOUR PROGRESS

Your answer should include the following points

Check Your Progress – 1

1. Milk Fat Separates out and from a cream layer on the top it is not inaccurate mixed thoroughly it create problem of uneven distribution in whole of the milk leading to results. Vigorous mixing on the other hand, result in churning of milk fat again leading to uneven distribution of Fat.
2. He or she must be authorized, properly equipped, Trained for drawing samples for chemical and microbiological analysis, Must be free from infectious diseases and Physically and mentally fit.
3. It is a small but representative of the bulk product. A random sample is that in which every unit of population has an equal chance to of being incorporated.
4. Sampling personal are told to as how, when, how much sample should be taken even then if the test personal in the are involved in sampling only then they can

explain to the sampling personnel the common mistakes which results in a am invalid or non-reproducible measurement.

5. Inert, can be closed properly to avoid further contamination, should be non reactive, must be light in weight, non fragile, opaque to prevent light, wide mouthed for solid or semisolid products.
6. 120° C for 20 min. or 100° C for 2 hours.
7. Separation of different components such as milk fat, if stored at room temperature microbial fermentation may occur leading to developed acidity if frozen the cells may ruptures. Water may evaporate or be absorbed or other volatile constituents may evaporate. Fat, protein, lactose may be hydrolyzed due To enzymatic action.
8. Agitator, plunger, dippers borers, butter triers, cheese triers, spoon, knife, fistula or ice scoop.
9. For milk powder 1 out of 70 bags. For butter I container out of 70. For ghee I tin out of 50. For liquid milk, ice-cream, table butter etc. from the small pack full pack is taken (1 out of 50 or 1 from each batch).