

# Detection of Moisture, Solid Not Fat and Salt in Butter

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**Abstract:** Milk is a pale liquid produced by the mammary glands of mammals. It is the primary source of nutrition for young mammals before they are able to digest other types of food. Early-lactation milk contains colostrums, which carries the mother's antibodies to its young and can reduce the risk of many diseases. Milk contains many other nutrients and the carbohydrate lactose. An emulsion is a suspension of droplets of one liquid into another liquid. Milk is an emulsion of fat in water. Butter is an emulsion of water in fat. The solute is known as the dispersed phase and the solvent is known as the continuous phase. Other examples of emulsions include margarine, mayonnaise, cream, and salad dressing. A colloidal solution is when matter exists in a state of division in between a true solution, which is sugar in water, and a suspension, which is chalk in water. The characteristics of a colloid are small particle size, electrical charge, and affinity of the particles for water molecules. In milk, the whey proteins are in colloidal solution. This paper detects various types of adulteration present in butter.

**Keywords:** Types of adulterants and adulterations etc.

## I. INTRODUCTION

Milk is very valuable food, readily digested and absorbed. It consists of nutrients, which are needed for proper growth and maintenance of body. Milk and milk products form a significant part of the diet and a substantial amount of our food expenditures goes on milk and other dairy products. In Pakistan, milk is transported from the point of production to consumers and processing plants by middlemen called "Gawalas". They don't maintain proper hygienic conditions during this transport, which leads to increase the total viable bacterial count. They also adulterate milk to increase their profit margin by several chemicals like urea, starch, flour, cane sugar, vegetable oils, detergents etc. Various preservatives like formalin and some antibiotics are also added in milk to increase its shelf life. This addition decreases the nutritive value of milk. These adulterants, preservatives and drugs in milk products cause very serious health related problems.

### *What Is Adulteration?*

Food is the basic necessity of life. One works hard and earns to satisfy our hunger and relax (enjoy) later. But at the end of the day, many of us are not sure of what we eat. We may be eating a dangerous dye, sawdust, soap stone, industrial starch, and aluminum foil and so on! Contaminated foods and drinks

are common sources of infection. Often, we invite diseases rather than good health.

Food adulteration is an act of intentionally debasing the quality of food offered for sale either by the admixture or substitution of inferior substances or by the removal of some valuable ingredient

Food Adulteration takes into account not only the intentional addition or substitution or abstraction of substances which adversely affect nature, substances and quality of foods, but also their incidental contamination during the period of growth.

## II. MATERIALS AND METHODS

### *Preparation of Sample of Butter:*

Warm the sample in an airtight container with the lid screwed down tightly or with the glass stopper in an oven or water bath maintained at  $37 \pm 2^\circ\text{C}$  shake vigorously to obtain a homogeneous fluid emulsion free from unsoftened pieces. In case, the sample does not mix up properly (water separation can be seen) reject the sample. For analysis of butter fat heat a portion of emulsified butter in a beaker to a temperature of  $50 - 60^\circ\text{C}$  until the fat separates. Filter the fat layer through a dried filter paper into a dry vessel. Melt the filtered fat if necessary and refilter to obtain clear fat free from water

### *(1) Determination of Moisture in Butter:*

#### *Apparatus:*

- A. Hot air oven: Maintained at  $100 \pm 1^\circ\text{C}$ .
- B. Flat bottom moisture dish: Dishes of height at least 25 mm and at least 50 mm in diameter, and made of appropriate material (for example stainless steel, nickel or aluminium) not affected by boiling water.
- C. Glass rods with one end flattened and about 9 cm in length.
- D. Desiccator with an efficient desiccant.
- E. Boiling water-bath with rings to take dishes of 50 mm diameter.
- F. Clay pipe triangles.

*Procedure:*

A. Clean the dish and the glass rod and dry them in the hot air oven maintained at  $100 \pm 1^\circ\text{C}$  for at least 1 hour.

B. Allow to cool to the room temperature in a desiccator and weigh the dish. Accurately weigh (to the nearest 0.1 mg) into the dish 3 to 4 g of the prepared butter sample.

C. Place the dish on a boiling water-bath supported on a clay pipe triangle for at least 20 min, stirring at frequent intervals until no moisture can be seen.

Wipe the bottom of the dish and transfer it to the oven maintained at  $100 \pm 1^\circ\text{C}$  and keep it for 90 min. Allow the dish to cool in the desiccator and weigh to the nearest 0.1 mg.

D. Heat the dish again in an oven for 30 min. Repeat the process of heating, cooling and weighing until the differences between two consecutive weights does not exceed 0.1 mg. Record the lowest mass and preserve the residue for the determination of curd.

*Note:* As per IDF (IDF 80-1, 2001) procedure, the weight of butter sample taken for moisture determination is 5 g and drying temperature is  $102 \pm 2^\circ\text{C}$  for 1 h. 12.2.3.

*Calculation:*

Moisture, % by mass =  $M_1 / M_2 \times 100$

Where,  $M_1$  = mass in g, of the dish with the material before heating to constant weight;  $M_2$  = mass in g, of the dish with the material after heating to constant weight; and  $M$  = mass in g, of the empty dry dish.

Methods of sampling and test for Butter. Bureau of Indian Butter – Determination of moisture, non-fat solids and fat contents – Part 1: Determination of moisture content

*(2) Determination of Fat and Curd (Milk Solids Not Fat) in Butter:*

Fat portion is removed with the help of petroleum ether and residue left behind is dried for determination of curd content. In case of table butter, it is curd and salt content and thus salt content has to be determined separately for calculating curd content.

*Apparatus:*

A. Gooch crucible or sintered funnel - with filter flask and adapter.

B. Glass funnel with folded 12.5 cm Whatman filter paper-1

C. Flat bottom flask: 250 ml capacity

D. Desiccator with efficient desiccant.

E. Asbestos.

F. Hot air oven: Maintained at  $100 \pm 1^\circ\text{C}$ .

G. Conical flask: 250 ml capacity.

H. Glass beads.

*Reagent:*

n-Hexane or, alternatively, light petroleum hydrocarbon solvent (petroleum spirit) with boiling range between 40 to  $60^\circ\text{C}$ . The reagent shall not leave more than 1 mg of residue after evaporation of 100 ml.

*Procedure:*

A. Prepare a celite mat in a Gooch crucible or sintered funnel. Dry it in a hot air oven maintained at  $100 \pm 1^\circ\text{C}$ , cool in the desiccator and weigh. Alternatively, dry, cool and weigh ordinary glass funnel with folded 12.5 cm filter paper.

B. Melt the residue in the moisture dish and add 25 to 50 ml of petroleum solvent and mix well.

C. Fit the crucible to the filter flask or place the funnel with filter paper on a filter stand.

D. Wet the asbestos mat or the filter paper with petroleum solvent and decant the fatty solution from the dish into the asbestos or the filter paper, leaving the sediment in the dish. Macerate the sediment twice with 20 to 25 ml of petroleum solvent and decant again the fatty solution into the asbestos or the filter paper.

E. Filter the solution and collect the filtrate in a clean, dried, tared 250 ml flat bottom flask containing 1 to 2 glass beads.

F. With the aid of a wash-bottle containing petroleum solvent, wash all the fat and sediment from the dish into the crucible or the filter paper.

G. Finally, wash the crucible or the filter paper until free from fat, collecting all the filtrate in the conical flask. Preserve the filtrate for the determination of fat. Dry the crucible or filter paper in the oven maintained at  $100 \pm 1^\circ\text{C}$  for at least 30 min

*Note:* If fat is to be determined only, transfer all the filtrate to a pre-dried and weighed fat flasks containing 2-3 glass beads. Rinse the conical flask with petroleum ether. Evaporate the ether, first on the water-bath and then in the oven at  $102 \pm 2^\circ\text{C}$  for 1 hour or till the time the constant weight is obtained. Calculate the fat content from the residues obtained by using following formula % fat = 100

H. Cool in the desiccator and weigh. Repeat drying, cooling and weighing until the loss of weight between the consecutive weighing does not exceed 0.1 mg. Preserve the residue for the determination of salt.

*Calculation:*

Curd and salt, % by mass (C) =  $M_1 / M_2 \times 100$

Where,  $M_1$  = mass in g, of the filter paper with residue;  $M_2$  = mass in g, of the filter paper alone; and  $M$  = mass in g, of the sample. Percent Fat w/w =  $100 - (M+C)$  Where,  $M$  = Moisture

percent C = Curd & salt percent Curd percent by weight is obtained by subtracting the value of salt percent by weight from the value of C. (Ref:- IS 3507 – 1966 Method of sampling and Test for Butter)

### (3) Determination of Salt Content in Butter

#### Method 1. (Volhard's Method):

In this method, salt present in the butter sample is extracted with hot water MILK AND MILK PRODUCTS 2016 105 from the dried fat-free residue obtained in moisture determination. The chlorides are precipitated by adding excess of silver nitrate. The unused silver nitrate is titrated with potassium thiocyanate using ferric ammonium sulphate indicator.

#### Reaction:

Reagents A. Standard silver nitrate solution: 0.05 N, standardized against standard sodium chloride. Dissolve slightly more than theoretical quantity (8.7 g per 1 L of water) of silver nitrate (equivalent weight 169.89) in halogen-free water and dilute to volume (1 L).

B. Nitric acid: sp. gr. 1.42 approx. 70 % (m/m).

C. Nitric acid: Approximately 5 N.

D. Ferric ammonium sulphate indicator solution: Dissolve 50 g of ferric ammonium sulphate  $[\text{Fe}_2(\text{SO}_4)_3 \cdot (\text{NH}_4)_2\text{SO}_4 \cdot 24\text{H}_2\text{O}]$  in 95 ml of water containing 5 ml of 5 N nitric acid.

E. Standard potassium thiocyanate (KCNS) solution (Approx. 0.05 N): Standardized against standard silver nitrate. Weigh approx. 5.25 g KCNS and dissolve in 1 L water. Allow to stand overnight and filter, if necessary, to get a clear solution, standardize by titration against 0.05 N  $\text{AgNO}_3$  and dilute with requisite volume of water to get exactly 0.05 N KCNS solution.

#### Apparatus:

A. Beakers: 100, 250 ml capacity.

B. Volumetric flask: 100 ml, 1 L capacity.

C. Conical flask: 250 ml capacity.

D. Water-bath: Maintained at 60 to 70°C.

$\text{Ag}^+$  (excess) +  $\text{Cl}^-$  -  $\text{AgCl}$  (solid)

$\text{Ag}^+$  +  $\text{SCN}^-$  -  $\text{AgSCN}$  (solid)

$\text{Fe}^{3+}$  +  $\text{SCN}^-$  -  $[\text{FeSCN}]^{2+}$  (Reddish brown)

#### Procedure:

A. Extract the salt from the residue of curd and salt by repeated washing of the Gooch crucible or filter paper with hot water, or by placing the crucible or filter paper in a beaker of hot water.

B. Collect the rinsing in a 100 ml volumetric flask passing the solution through a filter paper. Allow to cool to room temperature and make up to volume.

C. Take 25 ml water extract into a 250 ml conical flask, and add an excess (normally 25 to 30 ml) of 0.05 N silver nitrate solution.

D. Acidify with nitric acid; add 2 ml of the indicator solution and 1 ml nitrobenzene. Mix and determine the excess of silver nitrate by titration with the potassium thiocyanate solution until the appearance of an orange tint, which persist for 15 s.

E. In the same manner determine the equivalent of 25 ml or the added amount of silver nitrate as thiocyanate using the same volumes of reagents and water.

#### Calculation:

$\text{NaCl}$ , % by mass =  $8 \times N \times \frac{A}{B} \times M$  where N = normality of potassium thiocyanate solution (0.005 N); A = volume in ml, of potassium thiocyanate in blank titration; B = volume in ml, of potassium thiocyanate in the sample titration; and M = mass in g, of the butter sample.

#### Method 2. (Mohr's Method):

In this method, the butter sample is melted in hot water, and the chlorides present in the mixture are titrated with a solution of silver nitrate using potassium chromate as indicator.

#### Reagents:

A. Standard silver nitrate solution (0.1 N): Standardized against standard sodium chloride. Dissolve slightly more than theoretical quantity of silver nitrate (equivalent weight 169.89) in halogen-free water and dilute to volume. Dissolve between 17 g and 19 g of silver nitrate in 1 L of water which is practically free from carbon dioxide. Standardize the silver nitrate solution against standard sodium chloride solution. Store the solution away from direct sunlight.

B. Potassium chromate indicator (5%, w/v): Dissolve 50 g of potassium chromate ( $\text{K}_2\text{CrO}_4$ ) in 1 L of water.

C. Calcium carbonate: Analytical Grade, free from chloride. 12.4.2.2. Apparatus A. Conical flask: 250 ml capacity. B. Burette: 50 ml capacity, graduated to 0.1 ml. C. Pipette: capable of delivering 2.0 ml

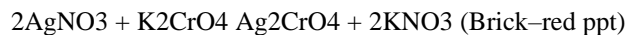
D. Measuring cylinder: 100 ml capacity, graduated.

#### Procedure:

A. Weigh accurately 5 g of butter sample into the 250 ml conical flask. Carefully add 100 ml of boiling distilled water. Mix the contents of the conical flask. Allow to stand with occasional swirling for 5 to 10 min.

B. After cooling to 50 to 55°C (titration temperature), add 2 ml of potassium chromate solution. Mix by swirling. Add about 0.25 g of calcium carbonate and mix by swirling.

C. Titrate at 50 to 55°C with standard silver nitrate solution while swirling continuously, until the brownish colour persists for half a minute.



D. Carry out a blank test with all the reagents in the same quantity except the butter sample. The maximum deviation between duplicate determinations should not exceed 0.02% of sodium chloride.

*Calculation:*

$$\text{NaCl, \% by mass} = \frac{5.8 \times N}{M}$$

Where

N = normality of silver nitrate solution (0.1N);

V1 = volume in ml, of silver nitrate used in the sample titration;

V2 = volume in ml, of silver nitrate used in the blank titration; and

M = mass in g, of the butter sample.

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