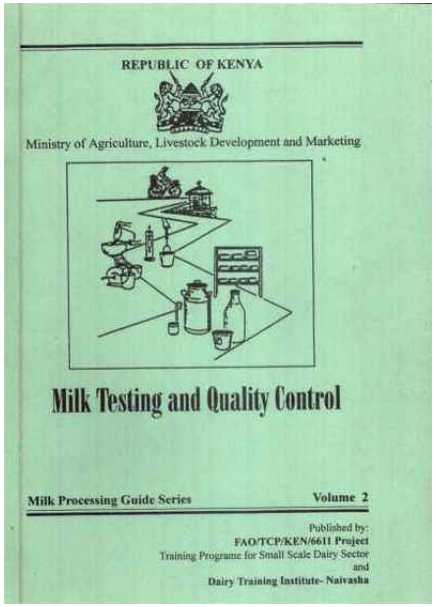
Milk testing and Quality Control



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1. INTRODUCTION

Milk testing and quality control is an essential component of any milk processing industry whether small, medium or large scale. Milk being made up of 87% water is prone to adulteration by unscrupulous middlemen and unfaithful farm workers. Moreover, its high nutritive value makes it an ideal medium for the rapid

multiplication of bacteria, particularly under unhygienic production and storage at ambient temperatures. We know that, in order for any processor to make good dairy products, good quality raw materials are essential. A milk processor or handler will only be assured of the quality of raw milk if certain basic quality tests are carried out at various stages of transportation of milk from the producer to the processor and finally to the consumer.

There are a number of standard manuals and text books on milk quality control. However these may not be easily available to the emerging small scale to medium scale processors in Kenya.

For these reasons, the Training Programme for the Small Scale Dairy Sector under project GOK/FAO/TCP/KEN/6611, has assembled this guide on Milk Testing and Quality Control so that it may be used for training and by the private small scale dairy processors. The methods selected are simple and basic and will suffice the requirements of most milk quality control laboratories of small scale processing units. For the larger plants with bigger laboratories more tests are to be found in the bibliography at the end of this booklet.

2. MILK TESTING AND QUALITY CONTROL

2.1 WHAT IS MILK QUALITY CONTROL?

Milk quality control is the use of approved tests to ensure the application of approved practices, standards and regulations

concerning the milk and milk products. The tests are designed to ensure that milk products meet accepted standards for CHEMICAL COMPOSITION AND PURITY AS WELL AS LEVELS OF DIFFERENT MICRO-ORGANISMS.

2.2 WHY HAVE MILK QUALITY CONTROL?

Testing milk and milk products for quality and monitoring that MILK PRODUCTS, PROCESSORS and MARKETING AGENCIES adhere to accepted codes of practices costs money. There must be good reasons why we have to have a quality control system for the dairy industry in Kenya.

The reasons are:

i)To the Milk Producer.

The milk producer expects a fair price in accordance with the quality of milk she/he produces.

ii) The Milk Processor.

The milk processor who pays the producer must assure himself/herself that the milk received for processing is of normal composition and is suitable for processing into various dairy products.

iii) The Consumer.

The consumer expects to pay a fair price for milk and milk products of acceptable to excellent quality.

iv) The Public and Government Agencies.

These have to ensure that the health and nutritional status of the people is protected from consumption of contaminated and sub-standard foodstuffs and that prices paid are fair to the milk producers, the milk processor and the final consumer.

All the above-is only possible through institution of a workable quality testing and assurance system conforms to national or internationally acceptable standards.

2.3 QUALITY CONTROL IN THE MILK MARKETING CHAIN IN KENYA

i) At the farm

Quality control and assurance must begin at the farm. This is achieved through farmers using approved practices of milk production and handling; and observation of laid down regulations regarding, use of veterinary drugs on lactating animals, regulations against adulterations of milk etc.

ii) At Milk collection Centres

All milk from different farmers or bulked milk from various

collecting centres must be checked for wholesomeness, bacteriological, and chemical quality.

iii) At the Dairy Factories

Milk from individual farmers or bulked milk from various collecting centres

iv) Within the Dairy Factory

Once the dairy factor has accepted the farmer milk it has the responsibility of ensuring that the milk is handled hygienically during processing. It must carry out quality assurance test to ensure that the products produced conform to specified standards as to the adequacy of effect of processes applied and the keeping quality of manufactured products. A good example is the phosphatase test used on pasteurised milk and the acidity development test done on U.H.T milk.

v) During marketing of processed products

Public Health authorities are employed by law to check the quality of food stuffs sold for public consumption and may impound substandard or contaminated foodstuffs including possible prosecution of culprits. This is done in order to protect the interest of the milk consuming public.

2.4 TECHNIQUES USED IN MILK TESTING AND QUALITY CONTROL

2.4.1 Milk sampling

Accurate sampling is the first pre-requisite for fair and just quality control system. Liquid milk in cans and bulk tanks should be thoroughly mixed to disperse the milk fat before a milk sample is taken for any chemical control tests. Representative samples of packed products must be taken for any investigation on quality. Plungers and dippers me used in sampling milk from milk cans.

2.4.2 Sampling milk for bacteriological testing

Sampling milk for bacteriological tests require a lot of care. Dippers used must have been sterilised in an autoclave or pressure cooker for at least 15mm at 120 C before hand in order not to contaminate the sample. On the spot sterilisation may be employed using 70% Alcohol swab and flaming or scaling in hot steam or boiling water for 1 minute.

Fig. 1: Equipment used for taking milk samples

2.4.3 Preservation of sample

Milk samples for chemical tests.

Milk samples for butterfat testing may be preserved with chemicals like Potassium dichromate(1 Tablet or ? ml 14% solution in a ? litre sample bottle is adequate.) Milk samples that have been kept cooling a refrigerator or ice-box must first be warmed in water bath at 40 ?C, cooled to 20?C, mixed and a sample then taken for butterfat determination. Other preservative chemicals include Sodium azid at the rate of 0.08% and Bronopol (2-bromo-2-nitro-1,3-propanediol) used at the rate of 0.02%.

If the laboratory cannot start work on a sample immediately after sampling, the sample must be cooled to near freezing point quickly and be kept cool till the work can start. If samples are to be taken in the field e.g. at a milk cooling centre, ice boxes with ice pecks are useful.

2.4.4. Labelling and records keeping

Samples must be clearly labelled with name of farmer or code number and records of dates, and places included in standard data sheets. Good records must be kept neat and in a dry place. It is desirable that milk producers should see their milk being tested, and the records should be made available to them if they so require.

2.4.5 Common testing of milk.

2.4.5.1 Organoleptic tests

The organoleptic test permits rapid segregation of poor quality milk at the milk receiving platform. No equipment is required, but the milk grader must have good sense of sight, smell and taste. The result of the test is obtained instantly, and the cost of the test are low. Milk which cannot be adequately judged organoleptically must be subjected to other more sensitive and objective tests.

Procedure:

- ***Open a can of milk.***
- ***Immediately smell the milk.***
- ***Observe the appearance of the milk.***
- ***If still unable to make a clear judgement, taste the milk, but do not swallow it. Spit the milk sample into a bucket provided for that purpose or into a drain basin, flush with water.***
- ***Look at the can lid and the milk can to check cleanliness.***

Judgement:

Abnormal smell and taste may be caused by:

- ***Atmospheric taint (e.g. barny/cowy odour).***
- ***Physiological taints (hormonal imbalance, cows in late lactation- spontaneous rancidity).***
- ***Bacterial taints.***
- ***Chemical taints or discolouring.***
- ***Advanced acidification (pH < 6.4).***

2.4.5.2 Clot on Boiling (C.O.B) Test

The test is quick and simple. It is one of the old tests for too acid milk(pH<5.8) or abnormal milk (e.g. colostral or mastitis milk). If a milk sample fails in the test, the milk must contain many acid or rennet producing microrganisms or the milk has an abnormal high percentage of proteins like colostral milk. Such milk cannot stand the heat treatment in milk processing and must therefore be rejected.

Procedure:

Boil a small amount of milk in a spoon, test tube or other suitable container. If there is clotting, coagulation or precipitation, the milk has failed the test. Heavy contamination in freshly drawn milk cannot be detected, when the acidity is below 0.20-0.26% Lactic acid.

Fig 2. Equipment used in C.O.B. test

2.4.5.3. The Alcohol Test

The test is quick and simple. It is besed on instability of the proteins when the levels of acid and/or rennet are increased and acted upon by the alcohol. Also increased levels of albumen (colostrum milk) and salt concentrates (mastitis) results in a positive test.

Procedure:

The test is done by mixing equal amounts of milk and 68% of ethanol solution in a small bottle or test tube. (68 % Ethanol solution is prepared from 68 mls 96%(absolute) alcohol and 28 mls distilled water). If the tested milk is of good quality, there will be no coagulation, clotting or precipitation, but it is necessary to look for small lumps. The first clotting due to acid development can first be seen at 0.21-0.23% Lactic acid. For routine testing 2 mls milk is mixed with 2 mls 68% alcohol.

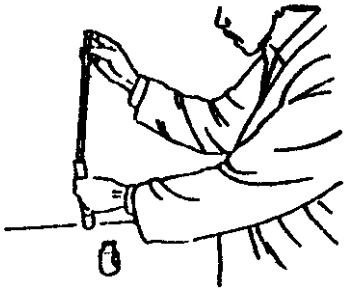

Fig. 3. Equipment used in alcohol test

2.4.5.4. The Alcohol-Alizarin test

The procedure for carrying out the test is the same as for alcohol

test but this test is more informative. Alizarin is a colour indicator changing colour according to the acidity. The Alcohol Alizarin solution can be bought ready made or be prepared by adding 0.4 grammes alizarin powder to 1 litre of 61% alcohol solution.

RESULTS OF THE TEST

Parameter	Normal milk	Slightly acid Milk	Acid milk	Alkaline Milk
PH	6.6 – 6.7	6.4 – 6.6	6.3 or lower	6.8 or higher
Colour	Red brown	Yellowish-brown	Yellowish	Lilac
Appearance of milk	No coagulation no lumps	No coagulation	Coagulation *	No coagulation **

Note:

**** = Sour milk looks yellowish with small lumps or completely coagulated.***

***** = Alkaline milk looks like lilac and it may be mastitis milk. Clots and flakes too, indicate mastitis milk.***

2.4.5.5 Acidity test

Bacteria that normally develop in raw milk produce more or less of lactic acid. In the acidity test the acid is neutralised with 0.1 N Sodium hydroxide and the amount of alkaline is measured. From this, the percentage of lactic acid can be calculated. Fresh milk contains in this test also "natural acidity" which is due to the natural ability to resist pH changes .The natural acidity of milk is 0.16 - 0.18%. Figures higher than this signifies developed acidity due to the action of bacteria on milk sugar.

Apparatus:

- ***A porcelain dish or small conical flask***
- ***10 ml pipette, graduated***
- ***1 ml pipette***
- ***A Burette, 0.1 ml graduations***
- ***A glass rod for stirring the milk in the dish***
- ***A Phenophtalein indicator solution, 0.5%in 50% Alcohol***
- ***N Sodium hydroxide solution.***

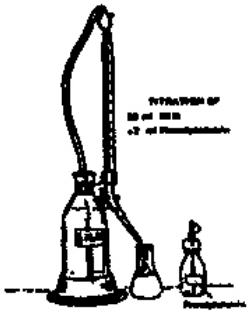

Fig. 4. Apparatus used be acidity test

Procedure:

9 ml of the milk measured into the porcelain dish/conical flask,1 ml Phenopthalein is added and then slowly from the burret, 0.1 N Sodium hydroxide under continuous mixing, until a faint pink colour appears.

The number of mls of Sodium hydroxide solution divided by 10 expresses the percentage of lactic acid.

2.4.5.6 Resazurin test.

Resazurin test is the most widely used test for hygiene and the potential keeping quality of raw milk. Resazurin is a dye indicator. Under specified conditions Resazurin is dissolved in distilled boiled water. The Resazurin solution can later be used to test the microbial activity in a given milk sample.

Resazurin can be carried out as:

i. ***10 min test.***
ii. ***1 hr test.***
iii. ***3 hr test.***

The 10 min Resazurin test is useful and rapid, screening test used at the milk platform.

The 1 hr test and 3 hr tests provide more accurate information about

the milk quality, but after a fairy long time . They are usually carried out in the laboratory.

Apparatus and reagents:

- ***Resazurin tablets***
- ***Test tubes with 10 mls mark***
- ***1 ml pipette or dispenser for Resazurin solution.***
- ***Water bath thermostatically controlled***
- ***Lovibond comparator with Resazurin disc 4/9***

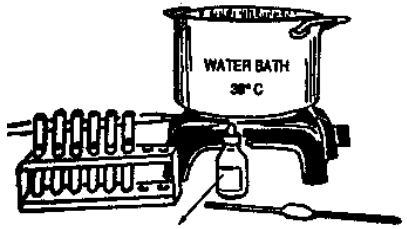

Fig. 5. Apparatus used in 10 min. Resazurin Test

Procedure:

The solution of Resazurin as prepared by adding one tablet to 50 mls of distilled sterile water. Rasazurin solution must not be exposed to sunlight, and it should not be used for more than eight hours because it losses strength.

Mix the milk and with a sanitized dipper put 10 mls milk into a sterile test tube.

Add one ml of Resazurin solution, stopper with a sterile stopper, mix gently the dye into the milk and mark the tube before the incubation in a water bath, place the test tube in a Lovibond comparator with Resazurin disk and compare it colourimetrically with a test tube containing 10 ml milk of the same sample, but without the dye (Blank).

READINGS AND RESULTS (10 MINUTE RESAZURIN TEST)

Resazurin disc No.	Colour	Grade of milk	Action
6	Blue	Excellent	Accept
5	Light blue	v. good	Accept
4	Purple	Good	Accept
3	Purple pink	Fair	Separate
2	Light pink	Poor	Separate
1	Pink	Bad	Reject
0	white	Very bad	Reject

2.4.5.7 The Gerber Butterfat test

The fat content of milk and cream is the most important single factor in determining the price to be paid for milk supplied by farmers in many countries.

Also, in order to calculate the correct amount of feed ration for high yielding dairy cows, it is important to know the butterfat percentage as well as well as the yield of the milk produced. Further more the butterfat percentage in the milk of individual animals must be known in many breeding programmes.

Butterfat tests are also done on milk and milk products in order to make accurate adjustments of the butterfat percentage in standardised milk and milk products.

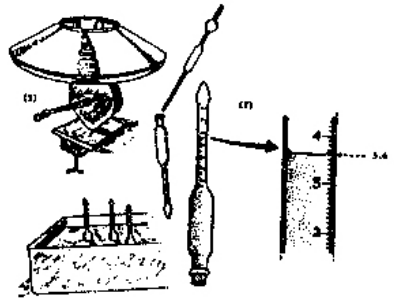

Fig. 6. Equipment used in Gerber Butterfat test

Apparatus for DF test:

- ***Gerber butyrameters, 0-6% or 0-8% BF***
- ***Rubber stoppers for butyrometers***
- ***10.94 or 11 ml pipettes for milk***
- ***10 mls pippetes or dispensers for Gerber Acid***
- ***1 mls pippetes or dispensers for Amyl alcohol***
- ***stands for butyrometers***

Gerber water bath Reagents:

- ***Gerber sulphuric acid,(1.82 g/cc)***
- ***Amyl alcohol***

Treatment of samples.

Fresh milk at approximately 20°C should be mixed well. Samples kept cool for some days should be warmed to 40°C, mixed gently and cooled to 20°C before the testing.

Procedure:

Add 10 mls sulphuric acid to the butyrometer followed by 10.94 or 11 mls of well mixed milk. Avoid wetting of the neck of the butyrometer.

Next add 1 ml of Amyl alcohol, insert stopper and shake the butyrometer carefully until the curd dissolves and no white particles can be seen. Place the butyrometer in the water bath at 65°C and keep it there until a set is ready for centrifuging. The butyrometer must be placed in the centrifuge with the stem (scale) pointing towards the centre of the centrifuge.

Spin for 5 min. at ll00 rpm.

Remove the butyrometers from the centrifuge.

Put the butyrometers in a water bath maintained at 65 C for 3 min. before taking the reading.

(Note: When transferring the butyrometers from the centrifuge into the water bath make sure that the butyrometers are all the time held with the NECK POINTING UP).

The fat column should be read from the lowest point of the meniscus of the interface of the acid-fat to the 0-mark of the scale and read the butterfat percentage.

The butyrometers should be emptied into a special container for the very corrosive liquid of acid-milk, and the butyrometers should be washed in warm water and dried before the next use.

APPEARANCE OF THE TEST

The colour of the fat column should be straw yellow.

The ends of the fat column should be clearly and sharply defined.

The fat column should be free from specks and sediment.

The water just below the fat column should be perfectly clear.

The fat should be within the graduation.

PROBLEMS IN TEST RESULTS

Curdy tests:

- ***Too lightly coloured or curdy fat column can be due to:***
- ***Temperature at milk or acid or both too low.***
- ***Acid too weak.***
- ***Insufficient acid.***
- ***Milk and acid not mixed thoroughly.***

Charred tests:

- ***Darkened fat column containing black speck at the base is due to:***
- ***Temperature of milk-acid mixture too high.***
- ***Acid too strong.***
- ***Milk and acid mixed too slowly.***
- ***Too much acid used.***
- ***Acid dropped through the milk.***

2.4.5.8 The Lactometer test

Addition of water to milk can be a big problem where we have unfaithful farm workers, milk transporters and greedy milk hawkers. A few farmers may also fall victim of this illegal practice. Any buyer of milk should therefore assure himself/herself that the milk he/she purchases is wholesome and has not been adulterated. Milk has a specific gravity. When its adultered with water or other materials are added or both misdeeds are committed, the density of milk

change from its normal value to abnormal. The lactometer test is designed to detect the change in density of such adulterated milk. Carried out together with the Gerber butterfat test, it enables the milk processor to calculate the milk total solids (% TS) and solids not fat (SNF). In normal milk SNF should not be below 8.5% according to Kenya Standards(KBS No 05-l0:-1976).

Procedure:

Mix the milk sample gently and pour it gently into a measuring cylinder (300-500). Let the Lactometer sink slowly into the milk. Read and record the last Lactometer degree (°L) just above the surface of the milk. If the temperature of the milk is different from the calibration temperature (Calibration temperature may be=20 0C) of the lactometer, calculate the temperature correction. For each °C above the calibration temperature add 0.2°L; for each °C below calibration temperature subtract 0.2 °L from the recorded lactometer reading.

EXAMPLE: Calibration temperature of lactometer 20°C.

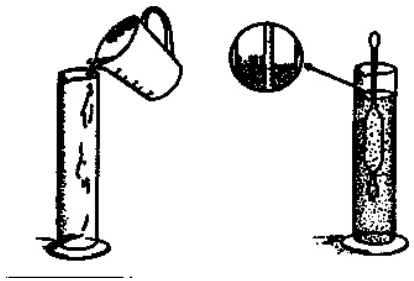

Fig 7. Equipment used for determination of milk density

Sample	Milk temperature	Lactometer reading	Correction	True reading
No.1	17 °C	30.6 °L	- 0.6 °L	30.0 °L
No.2	20 °C	30.0 °L	Nil	30.0 °L
No.3	23 °C	29.4 °L	+ 0.6 °L	30.0 °L

For the calculations, use lactometer degrees, and for the conversion to density write 1.0 in front of the true lactometer reading ,i.e. 1.030 g/ml. Clever people may try to adulterate milk in such a way that the lactometer cannot show the adulteration. But look to see if there is an unusual sediment from the milk at the bottom of the milk can and taste to find out if the milk is too sweet or salty to be normal. Samples of milk from individual cows often have lactometer reading outside the range of average milk, while samples of milk from herds should have readings hear the average milk, but wrong feeding, may result in low readings. Kenyan standards expects milk to have specific gravity of 1.026 -1.032 g/ml which implies a Lactometer reading range of 26.0 -32.0 °L. If the reading is consistently lower than expected and the milk supplier disputes any wrong doing arrange to take a genuine sample from the supplier (i.e. inspect milk right from source).

2.4.5.9 Freezing Point Determination

The freezing point of milk is regarded to be the most constant of all measurable properties of milk. A small adulteration of milk with water will cause a detectable elevation of the freezing point of milk from its normal values of -0.54 C. Since the test is accurate and sensitive to added water in milk, it is used to detect whether milk is of normal composition and adulterated.

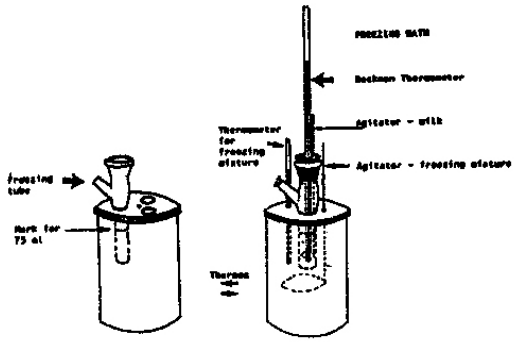

Fig. 8. A Cryoscope is used for determination of freezing point of milk.

2.4.5.10 Inhibitor test.

Milk collected from producers may contain drugs and/or pesticides residues. These when present in significant amounts in milk may inhibit the growth of lactic acid bacteria used in the manufacture of fermented milk such as Mala, cheese and Yoghurt, besides being a health hazard.

Principle of the method: The suspected milk sample is subjected to a fermentation test with starter culture and the acidity checked after

three (3) hours. The values of the titratable acidity obtained is compared with titratable acidity of a similarly treated sample which is free from any inhibitory substances.

Materials:

- ***test tubes***
- ***Starter culture***
- ***lml pipette***
- ***water bath***
- ***material for determination of titratable acidity (Fig.9)***

Fig. 9. Materials used to test inhibitory substances in milk

Procedure:

Three test tubes are filled with l0 ml of sample to be tested and three test tubes filled with normal milk.

All tubes are heated to 90 0C by putting them in boiling water for 3 - 5 minutes.

After cooling to optimum temperature of the starter culture (30,37, or 42 C), 1 ml of starter culture is added to each test tube, mixed and incubated for 3 hours.

After each hour, one test tube is from the test sample and the control sample is determined.

Assessment of results:

If acid production in suspected sample is the same as the normal sample, then the suspect sample does not contain any inhibitory substances;

If acid production as suspect sample is less than in the normal milk sample, then, the suspect sample contains antibiotics or other inhibitory substances.

3. QUALITY CONTROL OF PASTEURISED MILK

When milk is pasteurised at 63 C for 30 min in batch pasteuriser or 72 C for 15 seconds in heat exchanger, continuous flow pasteurisers, ALL PATHOGENIC BACTERIA ARE DESTROYED, there by rendering milk safe for human consumption. Simultaneously various enzymes present in milk, and which might affect its flavour, are destroyed.

In order to determine whether or not milk has been adequately pasteurised, one of the enzymes normally present in milk

phosphatase, is measured. A negative phosphatase result indicates that the enzyme and any pathogenic bacteria have been destroyed during pasteursation. If it is positive, it means the pasteurisation process was inadequate and the milk may not be safe for human consumption and will have a short shelf life.

- ***Test tubes***
- ***5 mls pipettes***
- ***1 ml pipettes***
- ***l00 ml volumetric flask***
- ***500 ml volumetric flask***
- ***water bath at 37 C***

Note: All glassware must be rinsed, cleaned, rinsed in chromic acid solution and boiled in water for 30 min.

Reagent:

Buffer solution:

Is mixed by 0.75g anhydrous sodium carbonate and l.75g Sodium bicarbonate in 500 ml distilled water.

Buffer-substrate solution:

Place 0.l5 g of di-sodium paranitrophenylphosphate(the substrate)into a clean 100ml measuring cylinder.

Add the buffer solution to make to 100 ml mark.

Store this buffer-substrate solution in a refrigerator and protected against light. It should not be used after one week. Prepare a fresh stock.

Procedure:

Pipette 5mls buffer-substrate solution into a test tube, stopper and warm the solution in the water bath at 37 C. Add to the test tube 1ml of the milk to be tested, stopper and mix well and place in water bath at 37 C. Prepare a blank sample from boiled milk of the same type as that undergoing the test. Incubate both the test samples and the blank sample at 37 C for 2hrs. After incubation, remove the tubes and mix them thoroughly.

Place one sample against the blank in a Lovibond comparator" ALL PURPOSES" using A.P.T.W. disc and rotate the disc until the colour of the test sample is matched and read the disc number.

Interpretation:

Disc Reading after 2 hrs incubation at 37 C	Remarks
0-10	Properly pasteurised
10-18	Slightly under pasteurised
18-42	UNDER PASTEURISED

> 42	NOT PASTEURISED

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