Quality Assessment and Detection of Adulteration in Buffalo Milk Collected From Different Areas of Gandhinagar by Physico-Chemical Method

Jivraj Makadiya* and Astha Pandey

Institute of Forensic Science, Gujarat Forensic Sciences University, Gandhinagar-382007 Gujarat, India.

Abstract: The study was carried out keeping in view the recently emerging concern of adulteration of natural milk with various illegal substances to increase its marketability. This study explains in detail the hygienic status of milk supplied to various dairies, small hotels and other public and educational institutions. A total of 30 samples were collected from different areas in Gandhinagar and Ahmedabad, Gujarat India and tested for determination and extent of adulteration. Qualitative analyses were carried out on 50 milk samples; standard milk adulteration methods followed by FSSAI (Food Safety and Standard Authority of India) 2012 Manual. Following are the significant observations of the study: The extent of adulteration varied significantly with least percentage for Glucose (30%), Sodium chloride (46%), Sucrose (50%) and highest for ammonium sulphates (96%) & Urea (100 %). All percentage values are indicative of presence of these adulterants (trace, moderate and high amounts combined). This qualitative analysis which has unfolded proved that the milk procured did not conform to the legal standards and was adulterated with toxic chemicals which are injurious to health.

Keywords: Milk adulteration; Dairy; Qualitative analysis of milk-color test; Adulteration.

Introduction

Milk is a complex mixture and a liquid food, which can easily be adulterated. According to PFA-1954 (prevention of food adulteration act) definition, “Milk is the normal mammary secretion derived from complete milking of healthy milch animal without either addition thereto or extraction there from. There are many methods known for detection of adulteration in milk but the methods discussed below are simple but rapid and sensitive methods to detect adulteration. in Milk contains more than 100 substances that are either in solution, suspension or emulsion in water, the important being casein - the major protein of milk, lactose - milk sugar, whey and mineral salts [1]

Milk is an almost ideal food. It has high nutritive value. It supplies body building proteins, bone forming minerals and health giving vitamins and furnishes energy giving lactose and milk fat. Besides supplying certain essential fatty acids.

A national survey in India has revealed that almost 70% of the milk sold and consumed in India is adulterated by contaminants such as detergent and skim milk powder, but impure water is the highest contaminant. According to National Survey on Milk Adulteration conducted by FSSAI (India) in 2011, water is the most common adulterant followed by detergent in milk. A survey by FSSAI in 2012, 68% milk samples was found to be adulterated in which 31 % were from rural areas. Of these 16.7 % were packet or branded milk and rest were loose milk samples from dairies. In the urban areas, 68.9 % milk was found to be adulterated with water, detergent, urea and skim milk powder. In Gujarat, 89% milk was found to be adulterated. Despite the
laws governing the quality and sale of milk existing in India for decades, the adulteration of milk has not been checked completely. [1]

Water is an adulterant in milk which is often always added to increase the volume of milk which in turn decreases the nutritive value of milk which if contaminated poses a health risk especially to infants and children. Detergents are added to emulsify and dissolve the oil in water giving a frothy solution, the characteristic white color of milk. Detergents cause gastro-intestinal complications. Urea is added to milk to provide whiteness, increase the consistency of milk and for leveling the contents of solid-not-fat (SNF) as are present in natural milk. The presence of urea in milk overburdens the kidneys as they have to filter out more urea content from the body.

Hydrogen Peroxide is also added to milk to prolong its freshness, but peroxides damages the gastrointestinal cells which can lead to gastritis and inflammation of the intestine.

Starch, cereal flours or arrowroot are added to make up the density of milk to prevent detection of added water. Starch is also used as an adulterant and if high amounts of starch are added to milk this can cause diarrhoea due to the effects of undigested starch in colon. Its accumulation in the body may prove very fatal for diabetic patients. Carbonates and bicarbonates are added to milk too, this can cause disruption in hormone signaling that regulate development and reproduction. Cane Sugar is added to raise the density to prevent detection of extraneous water.

Sodium chloride (common salt) is added to make up the density (lactometer reading) of watered milk.

Like urea, ammonium sulphate is a chemical fertilizer, which is added to milk to raise the density of watered milk. Neutralizers such as caustic soda, caustic potash sodium carbonate, sodium bicarbonate and lime water etc. are commonly added to milk to neutralize the developed acidity in milk. Some of these chemicals (neutralizers) are also ingredients of detergents which are major components of synthetic milk. Sodium and potassium nitrates are oxidizing agents and hence act as preservative. Pond water also contains appreciable quantities of nitrates and such water is usually admixed with milk by rural milk producers or vendors. [2]

Materials and Methods

Milk samples

A total 30 of buffalo milk samples were collected from different local Street vendor (public and educational institutions) & dairies from different areas of Gandhinagar & Ahmedabad and were preserved at -4°C Celsius temperature under refrigerator. The samples were collected in 100 ml screw capped sterilized plastic bottles. Formalin (formaldehyde) was used for preserved milk sample (0.4% HCHO added 2-3 drop/100 ml milk sample). All the possible precautions were taken to avoid external contamination at the time of collection of samples and during processing. Milk samples (n=30) of Samples were collected in clean, dry and neatly labelled sample containers and transported to laboratory in cold chain.

Analysis of milk samples

The milk samples were analyzed for physical appearance and presence of adulterants. Color and pH of all samples were checked and the adulteration tests were done using the FSSAI manuals of milk analysis. Color tests were performed for detection of Cane sugar, Starch, Urea, Ammonium sulphate, Glucose, Skimmed milk, Gelatin, Sodium chloride, Nitrates, Hydrogen peroxide, Boric acid, Formic acid, Cellulose, Benzoic acid & Salicylic acid, Hypochlorates & chloramines, Saccharine, and Anionic detergents in Buffalo milk samples.

Results

The color of all milk samples observed were creamy white in appearance, texture of milk samples were smooth and oily and odour of milk samples were characteristics pleasant and milky. The milk samples were taken and pH strip was dipped in sample. The change in colour was noticed and was matched with pH scale. pH of all 30 milk sample were in between 6.7 – 6.9. All milk samples were of very poor quality.

Detection of adulterants in milk samples:
Table: A Detection of adulterants in milk samples.

<table>
<thead>
<tr>
<th>S. no.</th>
<th>Adulterants</th>
<th>Test</th>
<th>+Ve Sample</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Cane- sugar</td>
<td>Resorcinol test</td>
<td>15</td>
</tr>
<tr>
<td>2</td>
<td>Starch</td>
<td>Iodine Test</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>Urea</td>
<td>p-DMAB test</td>
<td>30</td>
</tr>
<tr>
<td>4</td>
<td>Ammonium sulphates</td>
<td>Nessler’s reagent test</td>
<td>29</td>
</tr>
<tr>
<td>5</td>
<td>Glucose</td>
<td>Modified Barfoed reagent &amp; Phosphhomolybic acid reagent</td>
<td>09</td>
</tr>
<tr>
<td>6</td>
<td>Skimmed Milk powder</td>
<td>Conc.HNO$_3$ reagent</td>
<td>-</td>
</tr>
<tr>
<td>7</td>
<td>Gelatine</td>
<td>Saturated picric acid solution</td>
<td>-</td>
</tr>
<tr>
<td>8</td>
<td>Sodium chloride</td>
<td>Silver nitrate + potassium chromates reagent</td>
<td>14</td>
</tr>
<tr>
<td>9</td>
<td>Nitrates (pond water)</td>
<td>DPA Reagent</td>
<td>-</td>
</tr>
<tr>
<td>10</td>
<td>H$_2$O$_2$</td>
<td>KI- Starch reagent</td>
<td>-</td>
</tr>
<tr>
<td>11</td>
<td>Boric acid</td>
<td>Turmeric papers test</td>
<td>-</td>
</tr>
<tr>
<td>12</td>
<td>Formalin</td>
<td>Chromatropic acid test</td>
<td>-</td>
</tr>
<tr>
<td>13</td>
<td>Cellulose</td>
<td>Iodine- zinc chloride reagent</td>
<td>-</td>
</tr>
<tr>
<td>14</td>
<td>Salicylic acid</td>
<td>0.5 % ferric chloride solution, concentric sulphuric acid</td>
<td>-</td>
</tr>
<tr>
<td>15</td>
<td>Saccharin</td>
<td>Acetic acid, sodium hydroxide, con. HCl, diethyl ether.</td>
<td>-</td>
</tr>
<tr>
<td>16</td>
<td>Hypochlorates &amp; chlorates</td>
<td>Potassium Iodide solution, Dilute HCl, Starch solution</td>
<td>-</td>
</tr>
<tr>
<td>17</td>
<td>Anionic Detergent</td>
<td>Methylene blue dye &amp; Chloroform reagent</td>
<td>-</td>
</tr>
</tbody>
</table>

Fig. A  Detection of adulterants (X- data: No. of positive samples; Y- data: Adulterants

Discussion

Milk is one of the most complete foods available in nature for human consumption. Milk contains all nutrients in balanced proportions to meet the demand of humans. Good quality milk is required for quality dairy products. The adulterated raw milk with adulterants is taken as defective and cannot be processed. By performing color test for cane sugar 15 sample out of 30 were positives for adulteration of sugar. Fructose in cane sugar reacts with resorcinol in HCl and gives red color.
From the starch test it is observed that none of the samples of milk contain starch. Therefore there is no adulteration of starch in the milk samples. From the urea test it is observed that all the samples of Milk contain urea. It is only a qualitative test, but quantitative can also be done by UV spectroscopy. From the ammonium compounds test it is observed that all the samples of milk contain ammonium compounds except sample-18. From the Glucose test it is observed that sample no 21 to 30 in which total 9 samples were adulterated with added Glucose. All the samples belonged to package dairy Buffalo milk of Amul & Madhur brands.

None of the samples responded positively to Skimmed milk test indicating they might be free of it. None of the samples responded positively to Gelatin test indicating they might be free of Gelatin. None of the samples responded positively to nitrate test. Hence nitrate (pond water) might be absent in the milk samples. None of the samples responded positively to synthetic color test of Hydrogen peroxide indicating they might be free of it. None of the samples responded positively to synthetic color test of Boric acid indicating they might be free of it. As given in table no.15 none of the samples responded positively to synthetic color test of Formic acid indicating they might be free of it. None of the samples responded positively to synthetic color test of Cellulose indicating they might be free of it. None of the samples responded positively to synthetic color test of Benzoic acid & Salicylic acid indicating they might be free of it. None of the samples responded positively to synthetic color test of saccharin indicating they might be free of saccharin. None of the samples responded positively to synthetic color test of Hypochlorates & chloramines indicating they might be free of it. None of the samples responded positively to synthetic color test of Anionic detergents indicating they might be free of it.

Out of 30 samples collected from different areas of Ahmedabad and Gandhinagar 50% of milk samples were found to be positive for cane sugar which was more in packed sample compared to loose sample. Thus it can be made out that for increasing the density the cane sugar might have been added. Similarly 100% milk samples showed positive for urea both in packed and loose sample. Out of 30 29 samples of milk showed positive for ammonium sulphate indicating 96% adulteration in milk. Glucose was positive in only 9 samples compared to 21 which were all negative indicating 30% of adulteration. Skimmed milk powder and Gelatin showed no presence in the milk samples indicating no adulteration of these samples with Skimmed milk powder or gelatin. Sodium chloride test was found positive in 14 samples indicating 46% adulteration in both packed and loose milk samples. The milk samples were found to be negative for hydrogen peroxide, boric acid, nitrates, formalin, cellulose test, saccharin, salicylic acid, anionic detergent etc.

Conclusion

On the basis of data obtained in the present study, conclusion may be drawn that milk quality is not completely as per standards and adulteration in milk is still in practice and has not been checked completely. It is increasing very fast in Gujarat. Consumption of lower quality milk may lead to serious human health problems. To eradicate this malpractice by local dairy owners which is deep rooted in the cities more than rural areas, steps should be taken from the door steps of local consumers. The consumers must be more active against milk adulteration going on in whole country. It is important to have a quality control system that regularly check and ensure that only good quality milk is sold. The consumers and the milk sellers combined effort will help to decrease the adulteration practice.

The extent of adulteration varied significantly with least percentage for Glucose (30%), Sodium chloride (46%), Sucrose (50%) and highest for ammonium sulphates (96%) & Urea (100 %). This portrays that most of the milk samples were prepared with added adulterants during their production and processing or added intentionally according to one’s own choice to generate money. In a country such as India where milk and milk products play an important role in different foodstuffs, this analysis carried out should brings about more awareness to the general public about the malpractices or negligence in milk production.

In the above study an effort was made to systematically analyze the Buffalo milk samples. From the study it was found that the quality of most of samples was bad and hardly few sample s were found to be none adulterated.

Most common adulterants which were found in buffalo milk sample were Urea, Ammonium sulphates, Glucose, Cane sugar & Sodium chlorides, from the color test it was concluded that the preservatives in the Buffalo milk samples were not found.
From the above test it can be concluded that color test are good methods to detect the adulterants & further confirmation of the adulterants could be made by analytical methods, which could not be performed due to shortage of time.

The future scope lies in the use of FTIR in the detection of adulterants in the milk samples and other organic compounds can be detected by HPLC. Nanotechnology can also be used to detect the presence of melamine in the milk samples. The present dissertation work is useful to the forensic fraternity receiving the milk samples in the lab to check the adulterant.

References

7. Dairy Products School of Agriculture, India Gandhi National Open University. Vol-1
8. Foster Dee Snell, Wiley India, Encyclopaedia of Industrial Chemical analysis milk & milk products. Vol-26
10. State Institute of Vocational Education Director of Intermediates Education Government of Andhra Pradesh 2005, Quality Control of Milk & Processing.
16. FSSAI (Food Safety & Standard Authority of India) 1-13-2011 National Survey on Adulteration of Milk, New Delhi India.
18. P.J. John, Neela Bakore, et.al 2001 India worked on Assessment of organochlorine pesticide residue levels in dairy milk and buffalo milk from Jaipur City,Rajasthan, India, Environment International journal. p231-236
22. AOAC 2000, Dairy Products In Official Methods of Analysis. AOAC International, Gaithersburg, Maryland, USA.
23. PFA Act (1954) Prevention of Food Adulteration Act And Rules. Govt. Of India Publication As Amended Upto Date.
27. Mohd Aslam et.al 2013 Quantification of Organochlorine Pesticide Residues in the Buffalo Milk Samples of Delhi City, India Journal of Environmental Protection, p 964-974
30. Dr. Rajan Sharma et.al, January 2011, Chemical Analysis of Value Added Dairy Products and Their Quality Assurance Dairy Chemistry Division National Dairy Research Institute (Deemed University) Karnal (Haryana) India, p184-188
32. Asif Mahmood et.al 2010, Pakistan Journal of Nutrition A Comparative Study on the Physicochemical Parameters of Milk Samples Collected from Buffalo, Cow, Goat and Sheep of Gujrat, Pakistan vol-9, p1192-1197
36. Chlorpyrifos, DDT, Dichlorovos - Wikipedia, the free encyclopaedia.
38. Dr. Anjali aggarwal et.al 2008, Handbook of milk analysis, National Dairy Research Institute karnal Haryana India.

*****