DETECTION OF ADULTERATION IN MILK AND SOME DAIRY PRODUCTS

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ABSTRACT

Food adulteration is an act for debasing the quality of food with an admixture or through the substitution of inferior substances or by removing some invaluable ingredients from the food product. A total of 150 random samples of milk and some milk products (30 samples from each of raw cow's milk, UHT milk, Thick cream, Imported butter, Farmers Butter) were collected from different regions of Beni-Seuf city, Egypt and subjected to chemical analysis for detection of various adulterants. This study proved that the most prominent types of adulteration were addition of water, partial skimming or both. On the other hand, preservatives and commercial additives were absent in all milk and cream samples except gelatin which was found in 76.6% of cream samples. The mean iodine number of the imported butter and farmers butter samples ranged from 39.93 ±1.173 and 37.43 ± 0.718, respectively. All examined butter samples were free from cotton seed oil and starch while sesame oil was present in a percentage of 56.67% and 36.67% in the examined imported and farmers butter, respectively. In conclusion this qualitative analysis which has unfolded proved that some of milk and milk product procured did not conform to the legal standard and adulterated with some adulterants which decrease their nutritive value and may cause public health hazard.

Key words: Milk adulteration, cream, preservatives, commercial additives, iodine number, butter adulteration.

INTRODUCTION

Milk is a perfect human being food, produced by the mammary glands of mammals with high nutritional value providing the primary source of nutrition for young mammals before they are able to digest other types of food. Fresh milk is considered as a complete diet because it contains the essential nutrients as lactose, fat, protein, mineral and vitamins in balanced ratio rather than other foods (Khalid, 2006).

Cream is one of the most important dairy products defined either according to fat percent or heat treatment. It is considered as a primary product in manufacture of table butter, or for preparing ghee (Standard, 1966), also butter is considered the queen of fat specially when it comes from grass-fed cows, it is a rich source of vitamin A, D, E and K which are needed for the body and brain health and contain lecithin which assists in the proper assimilation and metabolization of cholesterol and other fat. It may be adulterated with addition of animal body fat hydrogenated oils and sometimes even the non-edible mineral oil (Tanmay *et al.,* 2017).

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Milk is a perishable commodity and is likely to be spoiled during summer season when weather becomes very hot. Also, due to unorganized and non-regulated marketing systems, the quality of milk is hardly maintained at consumer level, the middlemen through adding chemical preservatives such as penicillin, strepto-penicillin, formaldehyde, hydrogen peroxide, sodium bi-carbonate, etc. The adulterants and preservatives assume the proportion of health hazards for the consumers, particularly infants (Tipu *et al.,* 2007; Javaid *et al.,* 2009).

Adulteration of milk is done to increase its volume. Starch and other reconstituted milk powders are added to increase its viscosity. To increase the shelf life of milk ice and some chemicals like hydrogen peroxide, carbonates, bicarbonates, antibiotics, caustic soda and even the most lethal chemical formalin are being used. Urea adulterated milk is very harmful to the girls as it hastens up the process of puberty (Tariq, 2001).

Also, the adulteration of milk deteriorates the quality of milk, and may cause human health hazards, like gastroenteritis, nausea, vomiting, diarrhea, kidney damage and failure, acute failure of circulatory system, asthma, urticaria, metabolic acidosis, and convulsions in sensitive persons (Beall and Scofield, 1995; Mota *et al.,* 2003; Haasnoot *et al.,* 2004; Saad

et al., 2005; Ayub *et al.,* 2007; Rideout, 2008; Gwin *et al.,* 2009; Zhang *et al.,* 2009; Awan *et al.,* 2014).

So, this study was conducted to determine the chemical composition and detect various adulterants in milk and some milk products available in local market in different regions of Beni-Seuf city, Egypt.

MATERIALS AND METHODS

1. Collection and handling of the samples:
A total of 150 random samples of milk and some milk products (30 samples of each of raw cow's milk, pasteurized milk, thick cream, Farmers butter, Imported Butter) from different regions of Beni-Seuf city, Egypt during the period from February 2015 to Jan 2016 were collected in a clean, dry and sterile jars, and transferred to the laboratory with a minimum of delay, whereas they directly examined or held in the refrigerator until time is due.

2. Preparation of samples:

2.1. Milk Samples:
Each milk sample (500ml) was mixed thoroughly before being divided into 4 sub-samples. The first was used for detection of heat treatment, the second was used for compositional quality evaluation, the third was used for detection of preservatives, and the forth was used for detection of common commercial additives.

2.2. Cream samples (Standard, 1997):
Each cream sample (250gm) was mixed and warmed at temperature 35°- 40° C in a water bath, then the sample was cooled to a room temperature.

2.3. Butter samples
Each butter sample (250 gm.) was mixed thoroughly before being divided into 2 sub-samples. The first was used for detection of iodine number, while the second was melted to perform the other tests.

3. Methods of examination:

i. Milk and cream samples

1- Detection of heat treatment: by using Storch′s test (Lampert, 1975).

2- Compositional quality evaluation of examined milk samples.

a. Determination of fat %: By Gerber method (FAO, 1977).

b. Determination of total solid % (Ling, 1963).

c. Determination of milk solids not fat % (Harding, 1995).

d. Determination of added water % (Ling, 1963).

3. Specific chemical tests for detection of preservatives.

a. Detection of formalin (Panda and Bindla, 1998).

b. Detection of salicylic acid (Ling, 1963).

c. Detection of Hydrogen peroxide (Pien *et al.,* 1953)

d. Detection of boric and borax (ling, 1963).

e. Detection of nitrate (Pond Water) (Sharma and Barui, 2011).

4- Detection of commercial additives:

1. Detection of starch and cereal flour (Kumar *et al.,* 1998).

2. Detection of urea (Act PFA, 1954).

3. Detection of ammonium sulphate (Kumar *et al.,* 2002).

4. Detection of detergent (Singhal, 1980).

5. Detection of coloring matter (Batis *et al.,* 1981).

6. Detection of gelatin in cream (AOAC, 2000c).

ii. Butter samples

1. Determination of Iodine number (AOAC, 2000a)

2. Detection of sesame oil (Baudouin's Test) (Recioand Olieman, 1996).

3. Detection of cotton seed oil (halphen's test) (AOAC, 2000b).

4. Detection of starch and cereal flour (Kumar *et al.,* 1998).

RESULTS

Table 1: Statistical analytical results of the examined samples based on fat percentage.

The examined Samples	No of the examined sample	Min	max	mean	±SE
Rawcow's milk	30	2.00	4.00	3.01	0.098
UHT milk	30	2.50	3.50	3.10	0.058
Cream	30	27.78	76.90	56.99	2.757

Table 2: Statistical analytical results of examined samples based on total solids percentage.

The examined Samples	No of the examined sample	Min	max	mean	±SE
Raw cow's milk	30	8.54	11.44	10.04	0.138
UHT milk	30	9.14	10.84	10.07	0.073

Table 3: Statistical analytical results of examined samples based on solids not fat percentage.

The examined Samples	No of the examined sample	Min	max	mean	±SE
Raw cow's milk	30	6.05	7.84	7.03	0.069
UHT milk	30	6.53	7.49	6.98	0.049

Table 4: Correlation between methods for adulteration of milk.

The examined Samples	Partial skimmed			Addition of water			Addition of water and partial skimmed		
	No.	+ve	%	No.	+ve	%	No.	+ve	%
Raw cow's milk	30	8	26.67	30	30	100	30	8	26.67
UHT milk	30	5	16.67	30	30	100	30	5	16.67

Table 5: Statistical analytical results of the examined samples based on detection of some preservatives.

Item	No of examined sample	Raw cow's milk	UHT milk	cream
		No of +ve	No of +ve	No of +ve
Formalin	90	N.D	N.D	N.D
Salicylic acid	90	N.D	N.D	N.D
Hydrogen peroxide	90	N.D	N.D	N.D
Boric acid	90	N.D	N.D	N.D
nitrate	90	N.D	N.D	N.D

N.D= Not detected

Table 6: Statistical analytical results of the examined samples based on detection of some commercial additives.

Item	No of the examined sample	Rawcow's milk		UHT milk		cream	
		No of +ve	%	No of +ve	%	No of +ve	%
Starch	90	N.D	0	N.D	0	N.D	0
Urea	90	N.D	0	N.D	0	N.D	0
Ammonium sulphate	90	N.D	0	N.D	0	N.D	0
Detergent	90	N.D	0	N.D	0	N.D	0
Coloring matter	90	N.D	0	N.D	0	N.D	0
Gelatin	30	-	-	-	-	23	76.67

N.D= Not detected

Table 7: Statistical analytical results of the examined samples based on their iodine number**.**

Samples	No of the examined sample	Min	max	mean	±SE
Imported butter	30	30.45	55.83	39.93	1.173
Farmers butter	30	30.45	44.00	37.43	0.715

Table 8: Statistical analytical results of the examined samples based on cotton and sesame seed oil and starch.

samples	No of the examined sample	Baudouin's Test		halphen's test		starch	
		No of +ve	%	No of +ve	%	No of +ve	%
Imported butter	30	17	56.67	N.D	0	N.D	0
Farmers butter	30	11	36.67	N.D	0	N.D	0

N.D= Not detected.

DISCUSSION

Storch's test proved that all samples were randomly collected from dairy shops were in raw state, while all UHT milk samples were exposed for heat treatment. Similar results obtained by El-Bessary (2006) for raw cow's milk and Debnath *et al.* (2014) for UHT milk, while positive result was obtained by El-Loly *et al.* (2013) for raw cow's milk. heat treatment of the produced milk may protect consumers from being infected with pathogens, but it masks the unsanitary conditions under which milk is produced so it is act as a common type of adulteration also milk heating may result in off flavors and denaturation of protein (Draaiyer *et al.,* 2009).

The results given in Table 1 indicated that the fat percentage for the examined raw cow's milk samples ranged from 2.00 to 4.00 with a mean value 3.01± 0.098, while for UHT milk ranged from 2.50 to 3.50 with a mean value of 3.10± 0.058, and for thick cream samples it ranged from 27.78 to 76.0 with a mean value of 56.99±2.757.

The results were in agreement with those recorded by Mansour *et al.* (2012) and Amin (2016), while higher values were reported by Hossain *et al.* (2013) and Debnath *et al.* (2014), but the lower results were obtained by Sobeih (2000) and Fahmid *et al.* (2016) in raw cow's milk, while for UHT milk the results were in accordance with those reported by Bendale *et al.* (2015), but higher values were reported by Sobeih (2000) and Debnath *et al.* (2014), while lower values were obtained by Awan *et al.* (2014).

The low fat content in milk samples may be due to either reduced forage consumption and consequently decreases acetate and butyrate contents in rumen which are the major fat precursors, or to milk obtained after 4-5 lactations (animal's age), or failure

in stripping after milking, or attributed to adulteration by addition of water and /or partial skimming (Harding, 1995; Nickerson, 1995).

It is noticed from the obtained data in Table 2 that the total solids percentage for the examined raw cow's milk samples ranged from 8.54 to 11.44 with a mean value of 10.04± 0.138, while for UHT milk, it ranged from 9.14 to 10.84 with a mean value of 10.07± 0.073. These findings were significantly lower than the legal minimum of the Egyptian Standard (not less than 11.25%) (Egyptian Standards, 2005). The recorded results in raw cow's milk nearly similarly agreed with those reported by Mansour *et al.* (2012), while higher value were reported by Sobeih (2000), Fahmid *et al.* (2016) and Genzebu *et al.* (2016), but lower results were obtained by Faraz *et al.* (2013), where as higher values of heat treated milk were reported by Sobeih (2000), and lower values were obtained by Awan *et al.* (2014).

As shown in Table 3 the solids not fat percentage for the examined raw cow's milk samples was ranged from 6.05 to 7.84 with a mean value of 7.03±0.069, while for UHT milk it was ranged from 6.53 to 7.49 with a mean value of 6.98± 0.049, These results do not meet the legal Minimum of Egyptian standard (not less than 8.25%) (Egyptian Standards, 2005). Nearly similar results in raw cow's milk were reported by Fahmid *et al.* (2016) and Uddin *et al.* (2016), while higher value were reported by Sobeih (2000) and Amin (2016), but lower results were obtained by Faraz *et al.* (2013), while higher results of UHT milk are reported by Sobeih (2000) and Bendale *et al.* (2015), but lower values were recorded by Awan *et al.* (2014). The lower SNF content could be attributed mainly to adulteration by addition of water (Harding, 1995) as it decreases only by addition of water and not affected by partial skimming.

The data summarized in Table 4 mentioned that the water is added in varying degree in all types of milk samples which confirmed adulteration of milk by addition of water, but only about 8 (26.67%) out of 30 raw cow's milk sample and 5 (16.67) out of 30 UHT milk samples were adulterated by partial skimming of milk fat. Finally about 8 (26.67%) out of 30 raw milk samples and 5 (16.67) out of 30 UHT milk samples were adulterated by both addition of water and partial skimming of milk fat. The obtained results were in agreement with Chanda et al. (2012) and Swathi and Kauser (2015) in raw cow's milk and by Shaikh et al. (2013) in UHT milk, while lower results were obtained by Kandpal et al. (2012) and Debnath et al. (2014) for raw cow's milk and by Adam (2009) and Debnath et al. (2014) for UHT milk.

Generally, cow's milk samples had more subjected to adulteration by addition of water than buffalo's milk as it is low in fat content and any partial removal of fat will be appear. So, the retailers are pushed to addition of water to cow's milk than buffalo's milk (Mansour et al., 2012).

It is noticed that water is admixed with whole milk to increase the volume of milk during summer season, to successfully deal with the demand (Afzal et al., 2011). Water may be a source of microorganisms, harmful chemicals and poisonous substances. On the other hand, the addition of water decreases the milk solids not fat contents specially proteins which is very important for normal growth (Moore et al., 2012 and Santos et al., 2013). Also, skimming of fat inhibit the body from utilization of fat andfat-soluble vitamins as A, D, E and K which are very important for biological processes and normal growth of the body (Kartheek et al., 2011).

The result given in Table 5 indicated that all examined milk samples (raw cow's milk, UHT) and cream samples were free from the tested preservatives. The obtained results for formalin in raw cow's milk is in agreement with Abdel-Hameid (2002), El-Bessary (2006) and Amin (2016) while positive values were obtained by Wahba and Korashy (2006) and Debnath et al. (2014), also in UHT milk the results were similar to those reported by Debnath et al. (2014) and Prodhan et al. (2016), while positive results were obtained by Souza et al. (2011) and Debnath et al. (2015). Formalin causes damages of liver, lung tissues and increasing of total oxidant capacity (Aydin et al., 2015). The gained results for salicylic acid were similar with Wahba and Korashy (2006) and Amin (2016) in raw cow's milk and Lateef et al. (2009) in UHT milk, however Barham et al. (2014 a), Barham et al. (2104 b) recorded the positive results in raw cow's milk and Debnath et al. (2015) in UHT milk. Similar results for Hydrogen peroxide were reported by Debnath et al. (2014), while positive results were recorded by Debnath et al.

(2015) in raw cow's milk and UHT milk. Hydrogen peroxide has adverse effects on antioxidants balance in the human body leading to disturbance in the natural immunity and carcinogenic effect (Clare et al., 2003). The gained results for detection of Boric acid were fair agreement with El-Bessary (2006) and Debnath et al. (2014) in raw cow's milk and with Lateef et al. (2009) and Debnath et al. (2014) in UHT milk, while the positive results of raw cow's milk were recorded by Wahba and Korashy (2006), Mansour et al. (2012) and Amin (2016). Boric Acid causes nausea, vomiting, diarrhea, kidney damage, acute failure of circulatory system and even Death (See et al., 2010).

Result of detection of Nitrate is in fairly close agreement with El-Bessary (2006) in raw cow's milk and Bendale et al. (2015) in UHT milk, but positive results were recorded by Mansour et al. (2012). The overabundance dietary admission of nitrate lead to metahemoglobinaemia and the vivo production of the carcinogenic effect especially newborn infants (Gapper et al., 2004).

It is clearly evident to mention that our samples are free from chemical preservatives, but other residues as antibiotics or sulfa drug residues may be introduced into milk via treatment of dairy animals, their feeds, milking instrument and processing plants (Hubbert et al., 1996).

The results summarized in Table 6 showed that the examined additives couldn't be detected in all milk and cream samples with exception for gelatin that observed in creams sample at a percentage of 23(76.6%) out of 30 sample, which give it firmness and an appearance of richness (Stokes, 1897).

It is interest to note that the middlemen are added starch to synthetic and natural milk to adjust and or to increase the consistency and viscosity (Mudgil, 2008). Result of detection of Starch seems to be agreement with Debnath et al. (2014) and Uddin et al. (2016) in raw cow's and UHT milk, while positive results were recorded by Swathi and Kauser (2015) in raw cow's milk and Barham et al. (2015) in UHT milk. The adulterated milk with high amounts of starch can cause diarrhea due to the impacting of indigestible starch in colon and accumulation of indigestible starch in diabetic patients may prove very fatal (Singuluri and Sukumaran, 2014).

Addition of urea to milk, making the milk looked thick and concentrated giving a feeling of rich milk while, in fact it was low in fat and SNF and was poisonous due to the presence of excess urea (FAD, 2005 and Renny et al., 2005). The obtained results of detection of urea were agreed with Soomro et al. (2014) and Swathi and Kauser (2015) for raw cow's milk and with Bendale et al. (2015) for UHT milk, whereas the positive results were recorded by

Kandpal *et al.* (2012) and Faraz *et al.* (2013) for raw cow's milk and with Debnath *et al.* (2014) for UHT milk.

Ammonium Sulphate is a chemical fertilizer, which is added to milk to raise the density of watered milk and increases the lactometer reading by maintaining the density of milk (Abbas *et al.,* 2013). The obtained results for Ammonium sulphate were agreed with Nirwal *et al.* (2013) in raw cow's milk and Lateef *et al.* (2009) in UHT milk, but positive results were recorded by Debnath *et al.* (2015) for raw cow's and UHT milk.

The detergents are added to milk to emulsify and dissolve the oil in water giving the frothy solution (foamy appearance), and give milk its characteristic white color (Centre for science and environment, 2006). Nearly similar results of detection of detergent were reported by Faraz *et al.* (2013) and Soomro *et al.* (2014) in raw cow's milk and Awan *et al.* (2014) in UHT milk, while the positive results were recorded by Debnath *et al.* (2014) in raw and UHT milk.

Iodine value (IV) is a measure of the extent of unsaturation of fat (Rangappa and Achaya, 1974 and Knothe, 2002). The presented data in Table 7 explained that the iodine number of the examined imported and farmers butter samples ranged from 30.45 to 55.83 with a mean value of 39.93 ±1.173 and 30.45 to 44.00 with a mean value of 37.43 ± 0.715, respectively. Our results approximately agreed with those previously achieved by Hallabo and El-Nikeety (1987), for the imported butter, and EL-Mossalami (2014) for farmers butter, while higher results were obtained by EL-Mossalami (2014)for the imported butter and Ibrahim (2006) for the farmers butter, whereas lower results were recorded by Sagdic *et al.* (2004), for the imported butter and Park *et al.* (2007) for the farmers butter, it was noticed that the iodine value did not differ significantly from one season to another and the lowering of the iodine values due to lack of green fodder in summer which compensated by administration of sufficient quantity of oil-cakes containing fats of high iodine values. (Kehar, 1956).

The data summarized in Table 8 revealed that 17 (56.67%) out of 30 examined imported butter samples and11 (36.67%) out of 30 examined farmers butter were positive for sesame seed oil while all examined butter sample were free from cotton seed oil and starch. Similar result for sesame seed oil was reported by Parmar (2005), but negative result was obtained by Rao *et al.* (2004). Positive results for presence of starch were recorded by Swathi and Kauser (2015).

It can be concluded from the present investigation that the milk and dairy product mayexhibit a wide array of hazardous impacts on human health. So in order to protect consumers from the hazards of adulteration. Periodical inspection of markets by inspectors of ministry of health should be enforced to control and to minimize the risks of adulteration and health ministry laboratories facilities need to be supplemented and upgrading to be capable of performing all related tests.

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الكشف عن الغش في اللبن وبعض منتجات الألبان

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الغش في الأغذية عن طريق خلط أو استبدال أو إزالة بعض المكونات التي لا تقدر بثمن من المنتج الغذائي بالمواد الرديئة هو وسيله تؤدي إلي خفض نوعية وجوده الأغذية. تم تجميع ١٥٠ عينة عشوائية من اللبن وبعض منتجات اللبن (٣٠ عينة من كل من اللبن الخام واللبن المعقم والقشدة السميكة والزبد المستورد والفلاحي) من مناطق مختلفة بمدينة بني سويف بمصر وخضعت للتحليل الكيميائي والكشف عن مختلف انواع الغش. أثبتت الدراسة أن أبرز أنواع الغش هي إضافة الماء، نزع الدهن أو النوعين معا. من ناحية أخرى، كانت المواد الحافظة والإضافات التجارية غير موجوده في جميع عينات اللبن والقشدة مع استثناء الجيلاتين الذي وجد بنسبه ٧٦.٦٪في عينات القشده. وقد تراوح متوسط عدد اليود لعينات الزبد المستورد والفلاحي بين ٣٩.٩٣ ± ١.١٧٣ و ٣٧.٤٣ ± ٠.٧١٨ على التوالي. وكانت جميع عينات الزبد التي تم فحصها خالية من زيت بذور القطن والنشا بينما كانت ٥٦.٦٧٪ و٣٦.٦٧٪ من عينات الزبد المستورد والفلاحي تحتوي علي زيت السمسم علي التوالي. وفي الختام اثبتت هذه الدراسه ان بعض عينات اللبن ومنتجاته المجمعه من الاسواق لا تطابق المعايير القانونيه وقد تم غشها مما يقلل من قيمتها الغذائيه وقد تسبب مخاطر صحيه عامه.