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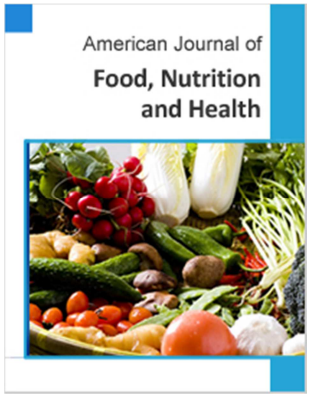

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Analysis of Representative Samples of UHT Sterilized Milk in the Egyptian Market

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Abstract
To study the properties of the UHT milk found in the local Egyptian market, Six representative UHT milk samples from the main Dairy plants were collected, Samples took the capital letters A, B, C, D, E and F. Samples were physco-chemically, microbiologically analysed as well sensory evaluation was done to follow their acceptability to the Egyptian UHT milk Specification No. 1623/2005. Results can be summarized as follows; Chemical composition of different samples before shaking are not the same after shaking. Total solids for fresh milks before shaking were 13.46, 14.11, 12.56, 11.93, 12.60, and 11.76% for A, B, C, D, E and F samples, respectively. Respective values after 180 days storage at room temperature were 14.36, 14.84, 13.32, 12.72, 13.16, 12.44%, respectively. Fat percentage for fresh and after 180 days of storage were (3.842/3.443), (3.76/3.320), (3.155/2.015), (3.114\2.704), (3.100/2.725) and (3.002/2.684) for A, B, C, D, E and F samples, respectively. Respective protein contents for fresh and 180 days old UHT were (3.290\2.654), (3.320\2.593), (3.230\1.220), (3.260\2.461), (3.200\2.513) and (3.150\2.485)% differences in such values is the tendency of protein to go towards the bottom. Ash contents before shaking ranged between (0.656%) and (0.784%). Sedimentation ratio increased with the storage period, the highest sedimentation values were for (C) sample being 6.703 gm. /200ml milk. The percentage of Saturated and Unsaturated fatty acids are not similar to the fat of cow milk. Expected that vegetable oils were added to milk before UHT processing. Organoleptic analysis scoring points decreased as the storage period advanced especially after 60 days of storage. Total scoring points for 60 days old UHT for different samples and after 180 days were (78.2/52), (81.2/50.5), (82.1/40.3), (87/53.7), (88.1/56.3) and (89.3/60.5) out of 100 for A, B, C, D, E and F treatment, respectively.

1. Introduction

Milk contains the main nutrients, such as fat, proteins, carbohydrates, minerals and vitamins, necessary to the early life stages: the high nutritional quality of milk facilitates to achievement of individuals' nutritional daily requirements. In 2011, the world cow milk production was nearly 606 million tons, and cow milk dominated the global milk production (84%). Consumption of milk and dairy products varies from country to country: in 2012, the per capita consumption of milk slowly increased in the world:

particularly, it increased in South America and Asia and decreased in Europe and Oceania (Gerosa & Skoet, 2012; OECD-FAO, 2012). Among heat treatments, pasteurization, a relatively mild heat treatment (at least 71.7°C for 15s), is sufficient to destroy disease-causing microorganisms: pasteurized milk shows a negative reaction to the phosphatase test and a positive reaction to the peroxidase test, and it must be preserved at ≤6°C. Pasteurization is used to extend the shelf-life of milk for six days, while UHT treatment, obtained by applying heat at high temperature (>135°C) for a short time (at least 1s), permits milk to be hold for a long period (90 days) at room temperature before being used EEC/1992 (Acocella, 1992). This "severe" treatment destroys all residual spoilage microorganisms and their spores, in order to prolong milk shelf life considerably. The growth of ultra-high-temperature (UHT) milk has been remarkable, increasing worldwide in the past 20 years especially in Europe, Asia and South America. Surprisingly, shelf-stable milk consumption in the USA. is very low compared with other regions in the world (Burton 1988; Kissell 2004). UHT processed fluid milk is very popular in other parts of the world; however, the U.S. population has been slow to accept it because of the "cooked" flavor in the UHT milk, their familiarity with fresh milk (Dairy Biz Archive, 2000) and the higher cost of UHT milk (Kissell, 2004). Ultra-high temperature (UHT) processing of milk results in a product with a long shelf-life when stored at room temperature (Valero *et al.*, 2001). The high temperature of the UHT process (140-145°C for 4-10s) and the long storage time can, however, result in changes in the sensory properties that can reach a limit beyond which the consumer will reject the product. Various enzymatic and physicochemical reactions occur in UHT milk and are responsible for the development of various off-flavours, sedimentation, gelation and discolouration of the milk (Shipe *et al.*, 1978; Celestino & Roginski 1997; Borle *et al.*, 2001). Sensory shelf-life studies often consider product defects, such as rancid and oxidised flavour in milk (Lawless and Claassen, 1993), as the critical attributes. These defects, however, are not always what determines the end of shelf-life, but rather changes in the levels of the desirable attributes or a combination of the two (Garitta, *et al.,* 2004). Although it is not expected that a product stored for several months should be exactly the same as the fresh standard, the sensory differences should be small enough for the acceptability of the product not to be altered significantly (Garitta, *et al.,* 2004). Probability of an individual failing before time x is reached. The "individual" in sensory shelf-life studies would not be the food itself, but rather the consumer. Therefore the failure function would be defined as the probability of a consumer rejecting a product at a time shorter than x. The focus of survival analysis used in shelf-life studies is therefore not on the food product and its deterioration but rather on the probability of a consumer rejecting the product stored for a certain time (Gambaro *et al.,* 2006; Hough, *et al.*, 2006; Hough *et al.*, 2003; Klein & Moeschberger, 1997).

In Egypt many attempts were paid to encourage the consumption of heat treated milk instead of boiling raw buffaloe milk. Pasteurized milk did not highly consumed because it has only 7 days shelf life and required refrigeration facilities, while UHT milk had the advantages of long shelf life at room temperature and more safe with the highly aseptic tetra pack containers. The Egyptian Standards (No. 1623/2005) dealt with some requirements of UHT milk, sometimes using powder milk and palm oils without mentioning their milk components. So the aim of this study is to make a survey study for the main UHT milk found in the Egyptian markets to follow their acceptance with the Egyptian Specification.

2. Materials and Methods

Six representative UHT milk samples were collected from local markets produced by the biggest Dairy plants, samples were tagged as A, B, C, D, E and F samples. Samples were chemically, physic-chemically, microbiologically analysed, as well sensory evaluation was done by experienced panelists from Damietta Dairy plant and staff of Damietta, faculty of Agriculture.

2.1. Physico-chemical Analysis

The fat content was determined according to the AOAC (2005) by Rose-Gottlieb process extraction method using diethyl ether, petroleum ether and ethanol. Protein content was estimated using Micro-kjeldahle method according to AOAC (2005). TS and Ash contents was determined by gravimeter method (Barbano and Dellavalle, 1984). Acidity is measured using the method of AOAC (2005), NaOH N/9 with ph.ph. It is expressed as lactic acid%. The pH value of the milk was determined using a digital pH meter Microprocessor – based pH/mv/°C Bench Meter, Model Number HI 2211-02, calibrated with pH 4 and 7 buffer solution (AOAC, 2005). Alcohol test was conducted by the method of Tessema and Tibbo (2009), where equal amount of milk and 70-80% ethanol solution were mixed in a test tube to the examine quality of milk. Sedimentation test was determined according to Ramsey and Swartzel (1984). The viscosity was measured at 20°C under constant conditions using BROOK FILD D-V–E VISCOMETER Model RVDVE Serial Number E 6531997, Made in USA using Spindle, the viscosity expressed in centipois (cp). Analysis of Milk Fat for Fatty Acid Composition: GLC "Gas Liquid Chromatography" using GC Conditions, Device Model: HP (Hewlett Packard) 6890 GC, Detector: FID (Flame Ionization Detector)

2.2. Microbial Count

Preparation of samples: Each sample of UHT milk was thoroughly mixed before being subjected to bacteriological

examination. The surface of the retail packs was thoroughly swabbed with 70% alcohol for aseptic sterilization.

Total viable counts: The plate count agar media (Bridson, 1995) was used for the total viable count in UHT milk samples (AOAC, 2005). Plates were incubated for 24 h at 37°C.

Coliform counts were determined by pouring plate method on violet red bile agar medium, plates were incubated for 24h at 37°C.

Detection of *Bacillus spp* was determined using (Oxoid manual, 2010) Mannitol egg yolk phenol red polymyxin (MYP) agar medium was used for enumeration and isolation of Bacillus cereus. Plate count technique, (Spread plate inoculation). 0.1 ml amount from each prepared dilution of samples under investigation was transferred and evenly spread onto the surface of MYP agar plates. All plates were incubated at 30°C for 24 hours. According to the FDA method, typical *B. cereus* colonies on Mannitol Egg Yolk Agar (MYP Difco) supplemented with Polimix in B sulfate 0.1%, are surrounded by a precipitated zone which indicates lecithinase activity and a pink color is observed because mannitol is not fermented. The typical colonies were counted & recorded.

Spore forming bacteria: Is done by heating the UHT milk at 80°C for 20 minutes and using Dextrose Tryptone Agar medium (Oxoid manual, 2010). Inoculated plates were incubated at 32°C for 72 hours.

2.3. Organoleptic Evaluation

12 experienced panelists from Domiattia Dairy plant and members of the staff of Faculty of Agriculture, Damietta University done the organoleptic analysis, 10 degrees for colour 45 for taste and aroma 30 for structure (Body and Texture), 10 for appearance and 5 for container and closer. Data of TS, Fat, Protein and ash were before shaking. Data are the average mathematical of three replicates.

3. Results

Milk samples are kept at room temperatures (25-40°C), other copy of the samples are kept at refrigerator conditions (5-10°C). pH values decreased at room temperature more than those kept at refrigerator (Table 1) included the pH values at both temperatures. It is clear that as the storage period advanced the pH values gradually decreased. On the other hand samples kept at room temperature had lower pH values as compared with refrigerator conditions, The (C) sample had the lowest value of pH. pH values of room temperature sample reached (6.82/6.52), (6.80/6.55), (6.74/6.27), (6.71/6.54), (6.74/6.51), (6.76/6.59) after 180 days of storage at refrigerator. pH values were (6.82/6.58), (6.80/6.59), (6.74/6.36), (6.71/6.58), (6.74/6.56), (6.76/6.61) for room and refrigerator temperature of A, B, C, D, E and F treatments, respectively. The change in pH values are due to the heat treatment and storage conditions.

Table 1. *Effect of storage at room temperature or refrigerator on the pH values of different samples during 180 days.*

pH at temperature Conditions	Time (Days)	A	B	C	D	E	F
(25 – 40°C) *(5 – 10°C)*	0	6.82	6.80	6.74	6.71	6.74	6.76
(25 – 40°C)	15	6.80	6.78	6.72	6.69	6.72	6.74
(5 – 10°C)		6.81	6.79	6.73	6.70	6.73	6.75
(25 – 40°C)	60	6.77	6.75	6.66	6.64	6.66	6.68
(5 – 10°C)		6.78	6.76	6.70	6.67	6.70	6.72
(25 – 40°C)	90	6.65	6.74	6.65	6.65	6.68	6.70
(5 – 10°C)		6.74	6.71	6.62	6.61	6.62	6.64
(25 – 40°C)	120	6.68	6.65	6.55	6.59	6.58	6.63
(5 – 10°C)		6.70	6.69	6.58	6.64	6.65	6.68
(25 – 40°C)	180	6.52	6.55	6.27	6.54	6.51	6.59
(5 – 10°C)		6.58	6.59	6.36	6.58	6.56	6.61

The values of TS of different samples through 180 days of storage are tabulated in Table 2. Values of T.S. for fresh milks were 13.46, 14.11, 12.56, 11.93, 12.60 and 11.76% for samples A, B, C, D, E and F before shaking, While after 180 days values became 14.36, 14.84, 13.32, 12.72, 13.16 and 12.44%, respectively at room temperature. The slight differences in TS values through 180 days before shaking the samples may be due to the location of pipette from where the sample was taken.

Table 2. *Effect of storage temperature on TS of UHT milk during 180 days of storage.*

TS% at temperature Conditions	Time (Days)	A	B	C	D	E	F
(25 – 40°C) *(5 – 10°C)*	0	13.46	14.11	12.56	11.93	12.60	11.76
(25 – 40°C)	30	13.58	14.24	12.67	12.05	12.51	11.86
(5 – 10°C)		13.65	14.22	12.63	12.02	12.48	11.84
(25 – 40°C)	60	13.70	14.35	12.80	12.17	1262	11.97
(5 – 10°C)		13.73	14.31	12.72	12.10	12.55	11.93
(25 – 40°C)	120	14.01	14.60	13.04	12.43	12.88	12.18
(5 – 10°C)		13.98	14.47	12.99	12.27	12.76	11.77
(25 – 40°C)	180	14.36	14.84	13.32	12.72	13.16	12.44
(5 – 10°C)		14.18	14.65	13.10	12.47	12.96	12.30

TS of different samples at refrigerator of different UHT milk through 180 days before shaking before shaking, slight differences in TS was detected for the six samples, fresh and 180 days old samples TS values were (13.46/14.18), (14.11/14.65), (12.56/13.10), (11.93/12.47), (12.60/12.96) and (11.76/12.30)% for A, B, C, D, E and F treatments, respectively, the difference of total solids between the six samples is due to the standardized chemical composition of fresh milk before processing. After shaking the samples no changes in TS of each milk through the storage period is noticed. Total solids value after shaking were 13.55, 14.22, 12.48, 12.00, 12.60 and 11.85%. No marked effect on the TS values of the UHT milk when stored at refrigerator conditions or at room temperature. The difference in TS of the six samples is owing to the chemical composition of raw milk before processing. Very slight increase in TS as the storage period advanced, is due to tend layer of sampling from where it is taken, the fat tend to float on the surface of milk, while the protein to go down depending on the homogenization conditions. To some extent temperature storage had no marked effect on TS. No significant difference in the total solids value of UHT milk sample were detected at different storage period or at different temperature.

Table 3. *Effect of storage temperature on Fat content of UHT milk samples through 180 days.*

Fat content at two temperature Conditions	Time (Days)	Fat%					
		A	B	C	D	E	F
(25 – 40°C)	0	3.842	3.76	3.155	3.114	3.100	3.002
(5 – 10°C)							
(25 – 40°C)	30	3.768	3.743	3.142	3.105	3.000	2.956
(5 – 10°C)		3.756	3.747	3.134	3.000	2.900	2.868
(25 – 40°C)	60	3.714	3.715	3.036	2.908	2.877	2.884
(5 – 10°C)		3.614	3.623	3.032	2.809	2.853	2.784
(25 – 40°C)	120	3.642	3.653	2.836	2.886	2.816	2.754
(5 – 10°C)		3.564	3.575	2.724	2.794	2.712	2.662
(25 – 40°C)	180	3.443	3.320	2.015	2.704	2.725	2.684
(5 – 10°C)		3.355	3.221	2.023	2.655	2.695	2.504

The difference in fat values is owing to the homogenization conditions and the tendency of fat to float on the surface of the milk, for this reason the samples are well shaked before fat checking. The differentiation of milk fat in different layers was reduced by higher homogenization pressure (Chun *et al.*, 2013). Fat content of different samples through 180 days of storage are shown in Table 3. Fresh and 180 days old samples had (3.842/3.443), (3.76/3.320), (3.155/2.015), (3.114/2.704), (3.100/2.725) and (3.002/2.684)% fat content for A, B, C, D, E and F samples, respectively. No effect of storage temperature in fat content of different samples. After shaking the samples fat content of different fresh samples recorded 3.80, 3.78, 3.15, 3.10, 3.10 and 3.00%, respectively, the slight differences in fat content of different samples may be due to the location of pipette from where the milk sample was withdrawn. Fat contents of the six samples are under the legal standard specifications of Egypt (Fat content should be more than 3%). Before shaking the fresh and 180 days old samples had (3.842/3.355), (3.76/3.221), (3.155/2.023), (3.114/2.655), (3.100/2.695) and (3.00/2.504)% fat content for A, B, C, D, E and F samples, respectively. No effect of storage in fat content of different samples. After shaking the samples fat content of different fresh samples recorded 3.80, 3.78, 3.15, 3.10, 3.10 and 3.00%, respectively. Fat content of the 4 brands collected from UHT milk Bangladesh factories ranged between 3.2-3.5% fat.

Table 4. *Effect of storage temperature on the protein content of the UHT milk during 180 days of storage.*

Protein content at temperature Conditions	Time (Days)	Protein content					
		A	B	C	D	E	F
(25 – 40°C)	0	3.290	3.320	3.230	3.260	3.200	3.150
(5 – 10°C)							
(25 – 40°C)	30	2.958	2.912	3.200	3.196	3.144	3.000
(5 – 10°C)		2.966	2.926	3.100	3.127	3.158	3.115
(25 – 40°C)	60	2.922	2.940	2.816	3.089	3.064	2.853
(5 – 10°C)		2.933	2.852	2.952	2.829	3.097	2.922
(25 – 40°C)	90	2.826	2.775	2.764	2.619	2.847	2.783
(5 – 10°C)		2.854	2.700	2.880	2.752	2.966	2.863
(25 – 40°C)	120	2.763	2.617	2.454	2.513	2.702	2.527
(5 – 10°C)		2.791	2.632	2.567	2.659	2.810	2.650
(25 – 40°C)	180	2.654	2.593	1.220	2.461	2.513	2.485
(5 – 10°C)		2.662	2.612	1.320	2.582	2.600	2.533

Values of total protein of different milk samples illustrated in Table 4, samples of fresh and 180 days old samples were (3.290/2.654), (3.320/2.593), (3.230/1.220), (3.260/2.461), (3.200/2.513) and (3.150/2.485)% for A, B, C, D, E and F treatments, respectively. Respective values for protein contents after shaking were 3.30, 3.20, 3.10, 3.15, 3.25 and

3.18%, respectively, similar to TS and F%, slight changes in protein content was recorded during storage which is may be due to the pipette location from where the milk sample was taken. Contrary to TS and F% contents, protein contents was affected by storage period for all treatments as storage period progressed, protein content gradually decreased. The sample C degradation may be owing to enzymatic hydrolysis, since microbiological analysis did not show any microbial enumeration on different Petri dish medium. Protein of samples of fresh and 180 days old samples before shaking kept at refrigerator temperature were (3.290/2.662), (3.320/2.612), (3.230/1.320), (3.260/2.582), (3.200/2.600) and (3.150/2.533)% for A, B, C, D, E and F treatments, respectively. Respective values for protein contents after shaking were 3.30, 3.20, 3.10, 3.15, 3.25 and 3.18%, respectively. During storage, certain decrease was observed for all samples, Egyptian standards excluded the limits of protein content.

Table 5. *Effect of storage period at room temperature on the ash content of the six UHT milk samples.*

Storage periods (Days)	A	B	C	D	E	F
Zero	0.656	0.667	0.634	0.706	0.737	0.752
30	0.665	0.674	0.654	0.727	0.706	0.765
60	0.690	0.687	0.664	0.746	0.718	0.784
90	0.657	0.652	0.685	0.765	0.691	0.765
120	0.673	0.667	0.698	0.696	0.702	0.746
180	0.662	0.685	0.732	0.729	0.743	0.706

Ash content values were shown in (Table 5) after shaking ash content values were 0.669, 0.675, 0.688, 0.703, 0.733 and 0.748% for A, B, C, D, E and F treatments, respectively. Before shaking there are some difference in ash contents of different samples. Again the difference is may be due to the location of withdrawn sample and not because of the period of storage. Fresh and 180 days old sample ash content were (0.656/0.662), (0.667/0.685), (0.634/0.732), (0.706/0.729), (0.737/0.743) and (0.752/0.706), respectively. Comparison results showed no noticeable differences are detected, so storage temperature had no effect on the content of ash of the milk during storage.

Table 6. *Effect of storage period at room temperature on milk precipitation by alcohol 70 and 80%.*

Time (Days)	Alcohol%	A	B	C	D	E	F
Zero	70	-	-	-	-	-	-
	80	-	-	-	-	-	-
15	70	-	-	-	-	-	-
	80	-	-	-	-	-	-
30	70	-	-	-	-	-	-
	80	-	-	-	-	-	-
45	70	-	-	-	-	-	-
	80	-	-	-	-	-	-
60	70	-	-	-	-	-	-
	80	-	-	-	-	-	-
75	70	-	-	-	-	-	-
	80	-	-	-	-	-	-
90	70	-	-	-	-	-	-
	80	-	-	+	-	-	-
105	70	-	-	+	-	-	-
	80	-	-	+	-	-	-
120	70	-	-	+	-	-	-
	80	-	-	+	-	-	-
135	70	-	-	+	-	-	-
	80	-	-	+	-	-	-
150	70	-	-	+	-	-	-
	80	-	-	+	-	-	-
165	70	-	-	+	-	-	-
	80	-	-	+	-	-	+
180	70	-	-	+	+	-	+
	80	-	-	+	+	-	+

A, B and E samples showed negative alcohol result for samples from zero to 180 days, after 165 days F sample precipitated by 80% alcohol and 180 days old sample precipitated by 70 and 80% alcohol. D sample precipitated at 180 days by both alcohols. C treatment gave positive results at 105 days and continued up to the end of storage.

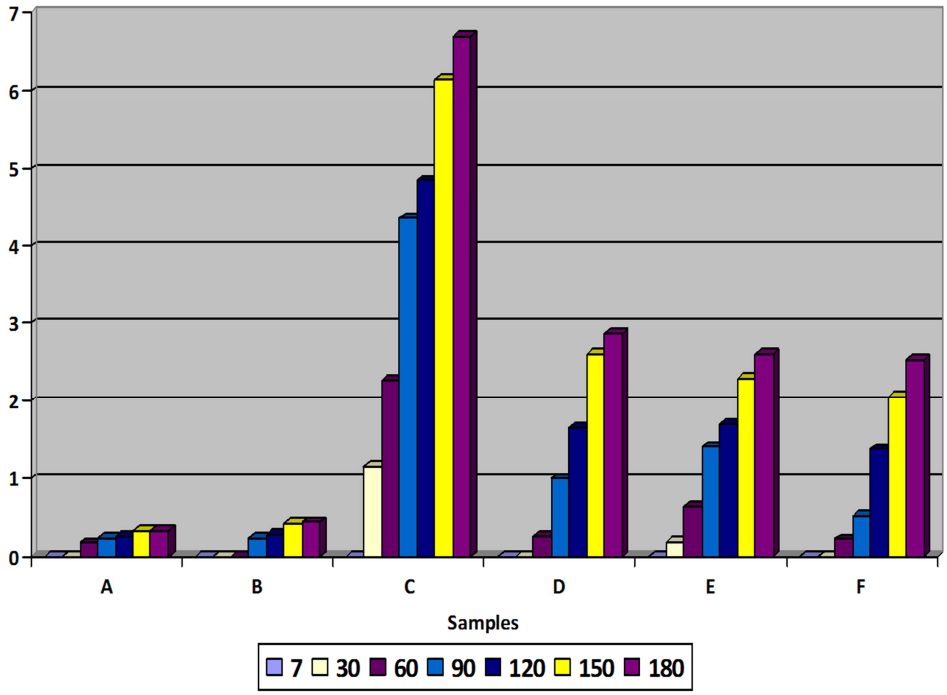

Figure 1. *Effect of storage at room temperature on sedimentation rate of different samples.*

The test was done only for room temperature samples. Data belonging the sedimentation are tabulated in Figure 1 which show that sedimentation was absent for all samples up to 15days of storage, C sample started sedimentation after 15 days. After 45 days A and B samples did not show sedimentation. Sample NO. B started sedimentation after 90 days. For all samples as storage period advanced sedimentation rate increased, 90 and 180 days old sample sedimentation rate were (0.235/0.325), (0.225/0.431), (4.353/6.703), (1.003/2.873), (1.406/2.610) and (0.520/2.544) gm per 200 ml of UHT milk for A, B, C, D, E and F samples, respectively. Egyptian standard (No. 1623/2005) excluded the sedimentation rate. Sample C had higher sedimentation value (6.703gm).

Table 7. *GLC fatty acid analysis of the six UHT milk after 90 days of storage at room temperature.*

Code-Name of fatty acid	A%	B%	C%	D%	E%	F%
C6:0 Caproic Acid	0.1096	0.10957	0	0	0	0
C8:0 Caprylic Acid	3.6655	2.3431	3.94509	2.35059	2.1356	2.3862
C10:0 Capric Acid	0.8865	0.806	0.64921	0.70012	0.7224	0.7171
C12:0 Lauric Acid	17.9707	15.8885	3.8370	2.38629	2.0959	1.7398
C13:0 Tridecanoic Acid	6.5948	3.5254	6.64675	8.15159	8.3438	7.7993
C14:0 Myristic Acid	6.9924	10.2727	7.013677	6.7945	6.7920	6.7725
C14:1 Myristoleic Acid	6.2835	3.4837	6.76008	8.08751	8.4032	7.6877
C15:0 Pentadecanoic Acid	3.681	2.6948	3.96204	4.71704	4.8643	4.5084
C15:1 cis-10-Pentadecenoic Acid	4.6594	2.5599	4.90827	5.8964	6.0609	5.5863
C16:0 Palmitic Acid	17.8683	22.8935	29.0813	26.9015	26.3884	27.3276
C16:1 palmitolic Palmitoleic Acid	0.9059	3.0158	1.8122	1.9249	2.0585	1.8930
C17:1 Cis-10-Heptadecenoic Acid	0.000	0.000	0.000	0.000	0.000	0.000
C18:0 Stearic Acid	18.6789	15.9274	14.4303	10.1767	9.3746	8.7872
C18:1c ω9 Oleic Acid	7.5192	11.8325	11.5610	15.4348	16.0857	17.8882
C18:2c ω6 Linoleic Acid	0.6261	0.9529	1.1568	1.6869	1.6502	1.4099
C18:3α ω3 Linolenic Acid	0.000	0.49815	0.000	0.000	0.000	0.000
C20:0 Arachidic Acid	0.000	0.000	0.000	0.000	0.000	0.000
C20:2 cis-11,14-Eicosadienoic Acid	0.000	1.0381	0.000	0.000	0.000	0.000
C20:3ω3 cis-11,14,17-Eicosatrienoic acid	0.000	0.000	0.000	0.000	0.000	0.000
C20:4 ω6 Arachidonic acid	0.000	0.000	0.000	0.000	0.000	0.000
C22:0 Behenic Acid	3.5582	2.26695	4.2361	4.79106	5.02424	5.4966
Total	100	100	100	100	100	100
Un-Saturated	19.9941	22.9992	26.1985	33.0306	34.2586	34.4652
Saturated	80.0059	76.9998	73.8015	66.9694	65.7413	65.5348

Some UHT milk processor replace milk fat fully or partly to milk with vegetable oil to adjust the milk fat component without mentioned that on the containers, the GLC is a good analysis method to detect the type of fat added to the milk. It is well known that milk fat had 60-70% saturated fatty acids and 30-40% unsaturated fatty acids as well contain 4.0-6.0% short chained fatty acids. Samples D, E and F had higher unsaturated fatty acids being 33.0306, 34.2586 and 34.4652% unsaturated fatty acids for D, E and F samples, while saturated percentage were 66.9694, 65.7413 and 65.5348% for D, E and F samples, respectively.

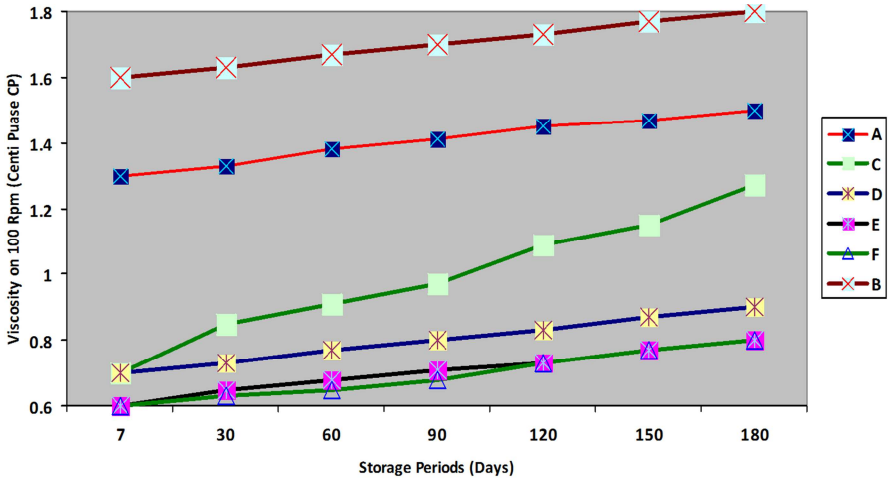

Figure 2. *Effect of storage at room temperature on viscosity of different samples.*

Values of viscosity at different storage time were tabulated in Figure 2. A and B samples had the highest viscosity at Zero time, there is a relationship between TS and viscosity values, as TS increased also viscosity increased for all treatments as the storage period advanced, the viscosity gradually increased values for fresh and 180 days samples were (1.30/1.50), (1.60/1.80), (0.7/1.27), (0.7/0.9), (0.6/0.8) and (0.6 and 0.80 CP) for A, B, C, D, E and F treatments, respectively. The higher sedimentation values samples had higher viscosity values. Samples A, B, C, D, E and F have normal viscosity, while sample C had great change of viscosity between fresh and 180 days old milk viscosity value. Under technological processing, results showed that the addition of 2 kg Recodan vegetable stabilizer is enough for one ton of recombined milk processed into UHT milk without high effect on viscosity while fresh milk needs only 1 kg of the vegetable stabilizer (Recodan)/Ton of fresh milk which highly alter the viscosity.

No colonies were found on the plates of different media even sample C. The UHT milk normally heated at 80-90°C for 15 seconds followed by UHT treatment at 137°C for three seconds and packed aseptically. These process are able to destroy all the groups of tested microorganisms.

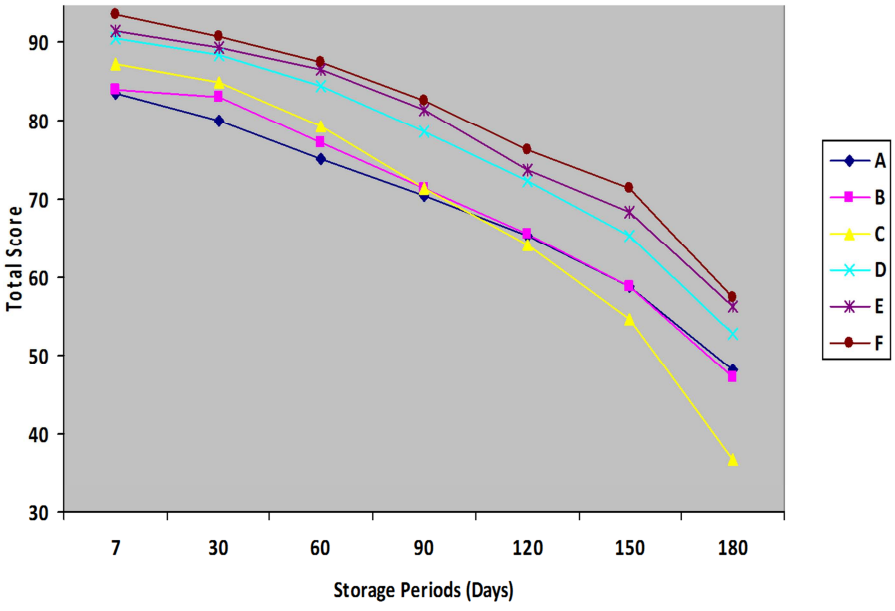

Figure 3. *Effect of storage at room temperature on organoleptic properties of different samples.*

Figure 3 included the average scoring points donated by the panelists. For colour, as the storage time progressed the density of white colour decreased, which means, higher level of Millard reaction. Samples No F, E and D gained the highest values 7.5, 7.4 and 7.3 out of 10, respectively at day 90, sample C, B and A scored 5.5, 6.8 and 6.5 scoring points out of 10, respectively. After 180 days of storage colour values were 4.5, 4.3, 4.0, 5.5, 5.4 and 5.8 for samples A, B, C, D, E and F, respectively. Similar to colour evaluation, taste and aroma scoring points (45 degrees) are decreasing as the storage period advanced Scoring points of fresh and 180 days old UHT milk samples were (40.8\22.3) (41.7\21.2) (42.3\15.1) (43.1\21.3) (43.5/23.1) and (44.1 and 24.5) scoring points out of 45 for A, B, C, D, E and F treatments, respectively. Body and Texture (30 degrees). Also Body and texture of UHT milk gradually decreased as the storage period advanced. The Body and Texture parameter required more proportional liquid, no coagulation, no precipitation, no forgners detected by eyes, normally, this is done for the tasted samples within natural day light, while smell and taste is done after gargelling with warm water. Body and texture scoring points were (25.5/15.7) (26.5/15.5) (23.5/12.5) (28.2/16.1) (28.3/17.3) and (28.7 and 19.5) scoring points for 90 and 180 days old UHT milk A, B, C, D, E and F, respectively.

Appearance scoring points are gradually decreased as the storage period advanced. Appearance as mentioned before are related to coagulation and precipitation. The absence of forgner bodies. Scoring points for fresh and 90 days old UHT milk were (8.5/6.8) (8.7/6.7) (8.5/5.8) (9.5/7.3) (9.7/7.5) and (9.8/7.8) out of 10 for samples A, B, C, D, E and F, respectively. Concerning containers and closing all six samples are similar to each other, since aseptic packing by tetra pack is very good system. Total scoring points for 60 and 180 old UHT milks are (78.2/52.0) (81.2/50.5) (82.1/40.3) (87.0/53.7) (88.1/56.3) and (89.3 and 60.5) out of 100 scoring points. The above results showed that it is preferable to make the validity of UHT milk after 3 months to keep the quality of the milk to each optimum.

4. Discussion

pH Values

Similar results were reported by El-Dakhakhny (1990), who found that the pH decreased with increasing the storage temperature and the highest decrease was after 180 days after incubation at room temperature, while Kawady (2004) concluded that the milk type and storage temperature had no significant effect on pH value, since storage period had significant effect on pH. This results agree with Aldubhany *et al.* (2014). Processing operations influences acid base equilibrium in milk. UHT treatment results in a pH decrease, due to conversion of lactose into different organic acids (Fox and Mc Sweeny, 1998). In milk, casein micelles are stable at natural pH that is 6.7. Lowering the pH facilitates aggregations of casein micelles and forms a gel, this result agreed with Ammara *et al.* (2009). Andrews *et al.* (1977) attributed the decrease in the pH of UHT milk is to reduction in the positive charge on the protein due to the reaction of the $-NH_2$ group of lysine with lactose in the Millard reaction. This might explain the larger decrease in the pH of recombined UHT milk and the larger decrease at higher storage temperature. AlKanhal *et al.* (1993).

Total Solids Content

Our results are in agree with those obtained by Barbano *et al.* (2006) and Aldubhany, *et al*. (2014). Egyptian standardization (No. 1623/2005) recommended 8.25% MSNF for fresh milk and 8.5% for fresh standardized milk. Awall *et al*. (2016) collected 4 brand of UHT Milk available in Bangladesh market, he found significant ($p \leq 0.05$) differences in the SNF of the four brands and are not according to their legal specification BSI (2002). Hossain *et al.* (2011) showed variation in total solids may be resulted due to addition of water in milk. Rania (2001) collected UHT Milk from to three state of Sudan, she found differences in the TS between the three factories and found gradual slight decrease in TS during storage being (11.26/11.14), (10.77/10.55) and (11.27/10.77)% for 15 and 90 days old samples of factory 1, 2 and 3, respectively. Aldubhany *et al*. (2014) studied the effect of storage temperature on the chemical composition of UHT milk at (4±2°C), (22 ±2°C) and (37±°C) for 180 days. No significant difference in the total solids value of UHT milk sample were detected at different storage period or at different temperature, their results are agree with those obtained by Barbano *et al.* (2006).

Fat Content

Their legal specification BSTI (2002), BDS1702 (2002) minimum fat% requirements 3.25% (Awall *et al*., 2016). Ammara *et al*. (2009) found that the result of fat before shaking are 3.55, 3.66, 3.88, and 3.50% in last week (W12) it reached to 2.70, 3.50, 1.85 and 3.00% for sample I, II, III, and IV, respectively. The difference in fat values is owing to the homogenization conditions and the tendency of fat to float on the surface of the milk, for this reason the samples are well shaked before fat checking, the differentiation of milk fat in different layers was reduced by higher homogenization pressure (Chun *et al*., 2013).

Total Protein Content

According to Ammara *et al*. (2009) stated that protein of milk is highly affected by heating and well storage period. The principal changes in UHT milk during storage may be due to enzymes. Most of milk proteins coagulate after heating, hens the texture is changed during storage. Casein polymerization is greater at high storage temperature, but occurs significantly even under refrigerator condition. Awall *et al*. (2016) mentiond that protein content, of UHT milk is

highly affected by heating and storage period. The samples were analysed without shaking, so the difference in protein content is may be due to the tendency of protein to go down the bottom of container. This result agreed with those of Chun *et al.* (2013).

Alcohol Test

The alcohol test can be used to detect raw milk that it is likely to give a high level of the normal type of sediments, and there are indications that it may be useful in predicting the abnormal type milk (Sweetsur and white, 1975). Processing operations influences acid base equilibrium in milk. UHT treatment results in a pH decrease, due to conversion of lactose into different organic acids (Fox and Mc Sweeny, 1998). In milk, casein micelles are stable at natural pH that is 6.7. Lowering the pH facilitates aggregations of casein micelles and forms a gel, this result agreed with Ammara *et al*. (2009). Awall *et al*. (2016) who studied the evaluation of physic-chemical properties of four brands of UHT milk available in Bangladesh clot on boiling (COB) and Alcohol test, the four brands showed negative on both COB and alcohol tests which refer to the good quality of milk, she added that both of tests are important in milk processing for identification of abnormal milk, developed acidity and mineral balance in milk.

Sedimentation Value

Gowing back to (Table 1) the pH value was lower and acidity was higher for C sample than the other samples of milk. Ernani *et al*. (1997) produced reconstituted, UHT milk from whole milk powders that were manufactured from fresh (control) or stored at (4±1°C, 48±2 h) raw milk and stored for different periods at 25±1°C with longer storage at both 3±1°C and 25±1°C greater sediment and lower pH were observed UHT milk processed from older powder in milk powder. Rates of enzymatic reaction and oxidative reactions appeared greater in UHT milk stored at 25 ±1°C and in those processed from older powders and contributed to the development of off flavours in UHT milk with a prolonged storage period. Gelation was observed only at 25±1°C. Lipases and proteinases were reactivated during storage and there activity was greater in UHT milk. Processed from powder manufactured from stored raw milk. The taste of reconstituted UHT milk was affected may be lipolysis than by proteolysis. Ammara *et al*. (2009) collected 4 samples of UHT milk from Bangladesh Dairy Factories, results obtained from sedimentation test in the samples during storage period 3 months (12 weeks) shows that there is an effect of heat processing and subsequent storage period on sedimentation for all samples of UHT milk The changes started in week 2 of shelf life for sample 1 and 111 and sample 11 showed formation of sediments after week 6, sample 111 reached up to 7.1 gm/250 ml which is considerable changes and sample 11 formation of sedimentation after week 5. Grewal *et al*. (2017) studied the feasibility of using Fourier transform Infrared Spectroscopy (FTIR) to detect heat induced conformational rearrangements of proteins (protein- protein) and (protein–lipid) interactions was studied with accelerated shelf–life portals. Ultra–high temperature created whole (WM) and skim milk (SM) were stored at 20, 30, 40, and 50°C for 28 days. The changes leading to increased sedimentation in SM and WM at higher temperature (≥40°C) were observed during first 14 days of the storage period. Milk in samples stored at 40 and corresponding to conformation. Proteolysis happen C sample of our result has been attributed to endogenous enzymes such as plasmin or exogenous enzymes such as bacterial proteases (Datta and Deeth 2003). Psychrotrophic bacteria, and especially the *Pseudomonas sp.*, are particularly incriminated in this destabilization (Gaucher, *et al*. 2011).

Viscosity

Ernani *et al*. (1997) processed reconstituted UHT milk from whole milk powders that were manufactured from fresh (control) or stored (4±1°C/48±2h), no difference in viscosity scores was observed in UHT milk samples stored at different temperature (3±1°C or 25±1°C) for the same period however viscosity measured instrumentally was greater in samples stored at refrigeration temperature. Aldubhany *et al*. (2014) showed that viscosity values increased gradually during storage from 1.337 to 1.877, 1.382 to 2.07 and 1.393 to 2.237 CP after 6 months of storage at 4±2°C, 22±2°C and 37±2°C, respectively, for UHT milk samples stored at different temperatures. The significant increase in viscosity started after 30 days of storage at all storage temperatures, while the highest changes were reported after 120 and 90 days of storage at 22 and 37°C, respectively. These results concluded that the storage period had a great significant effect ($p \leq 0.05$) on the viscosity of stored UHT milk samples even at refrigerated temperature. These results are agree with the corresponding results determined by Ernani *et al*. (1997). Also, Kawady (2004) concluded that the milk type and storage period had significant effect on viscosity, while the storage temperature had no significant effect on viscosity. El-Dakhakhny (1990), found that the different storage temperatures and storage periods had clear effect on viscosity (Hammad *et al*. 1993). The initial viscosity of FUHT milk (1.8 mPa s) was lower than that of RUHT milk (2.52 mPas) and remained lower throughout storage. The viscosity of UHT milk increased with time of storage at all temperatures, The increase in viscosity of FUHT milk was higher at high temperature, but for RUHT milk, viscosity was lower at high temperature, this was in spite of similar trends in proteolysis, lipolysis and fat separation in both types of milk. None of the milk samples gelled and the highest viscosity (3.9 mPa s) was for RUHT milk stored at 6°C for 25 weeks. Ernani *et al*. (1997) studied the viscosity of UHT milk produced from whole milk powders. They found that refrigerated storage of raw milk had no significant effect on viscosity of the resultant UHT milk ($p > 0.05$; means of 2.13 mPa s for control compared to 2.14 mPa s for UHT milk obtained from raw milk subjected to refrigerated storage). A slight change in viscosity during storage of UHT milk at 3±1°C and 25±1°C

was observed. UHT milk stored at refrigeration temperature had higher ($p \leq 0.01$) viscosity (mean of 2.18 mPa s for combined storage periods) than that stored at the higher temperature (mean of 2.12 mPa s). At 25±1°C, the highest viscosity value was observed at the third month of storage (2.16 mPa s), while at 3±1°C, this was observed at the fifth month (2.26 mPa s). Studies on directly heated recombined UHT milk (Renner 1988 ;Mittal *et al*., 1988; Alkanhal *et al*., 1994) showed similar results with regard to the effect of storage temperature, i.e. viscosity was greater in samples kept at refrigeration temperatures (5 or 6°C) than those at a higher temperature (30°C). Other authors (Ashton, 1966; Harwalkar & Vreeman, 1978; Mittal *et al*., 1990; Reddy *et al*., 1991) have reported increased viscosity in stored UHT milk while Sur & Joshi (1989) did not find much change in viscosity of UHT whole milk (ranging from 1.97 to 2.44 mPa s) during storage at 22 and 37°C for 5 months.

Fatty Acids Content

It is expected that UHT milk from fresh cow milk, while A, B and C samples had higher saturated and short chained fatty acids approximately short chained fatty acids ranged between 4.0 and 6.0%. Although Choi (1993) studied the cause and mechanism of the formation of free fatty acids in UHT process milk during storage, he found that the ratio of FFA (C_{10}, $C_{18:1}$ and $C_{18:2}$) were statistically significant. The degree of the difference was extremely small, therefore one may speculate that the same kind of chemical or enzymatic mechanisms were involved in liberating fatty acids from milk fat at booth temperature from zero to 12 week and temperature at 23 and 35°C. C_4(10.1/8.9-8.4), C_6(4.8/4.5-4.2), C_8(3.6/3.3-2.9), C_{10}(5.2/4.8-4.4), C_{12}(4.0/4.2-4.4), C_{14}(8.4/8.7-9.5), C_{16}(22.7/23.4-23.4), C_{18}(12.2/13.5-14.8), $C_{18:1}$(26.3/25.7-24.8) and $C_{18:2}$(2.7/3.0-3.2). In our researches unsaturated percentage were 19.99, 22.99 and 26.20% for A, B and C samples expected to be partly or fully vegetable source of oils. Alcalá *et al.* (2013) analysed powder whole milk (PWM) on GLC, our results for the six sample were compared with their results, oleic and (PWM) is lower (3.53%) than our results 8.13, 13.27, 12.71, 17.11, 17.13 and 19.3% for A, B, C, D, E and F samples, respectively.

Microbiological Analysis

The higher acidity found in some samples may be due to the enzymatic left during cold storage of raw milk and survived the high temperature during UHT processing. Similar results were found by Ammara *et al*. (2009), who analysed samples of UHT milk found in the local market of Pakistan. No colonies were found on selected media of A, P, C coliform, *B. cereus*, *B. subtilis* and spore formers bacteria.

Organoleptic Properties

Gaewalin *et al.* (2008) compared the differences in flavor and texture of 37 commercially available UHT and sterilized milk samples including whole 2% reduced-fat and low-fat milk obtained from markets in seven countries: France (n=2), Italy (n=11), Japan (n=1), Korea (n=2), Peru (n=3), Thailand (n=13) and the U.S.A. (n=5). Five highly trained panelists used flavor and texture profiling to describe the sensory properties of each milk sample and suggests that companies manufacturing processes for UHT milk may have more impact than country or fat content in determining sensory properties of UHT milk. Richards *et al.* (2016) showed that the sensory quality of the milk deteriorates over time. This coincides well with literature that states that different aroma, flavor and textural changes occur in UHT milk during storage and ultimately limits the shelf-life of the milk. These changes include a decrease in favourable attributes associated with the milk, e.g., the decrease in the sweet aroma and taste in UHT milk (Clare *et al*., 2005) and an increase in unfavourable attributes, e.g., off-flavour development and gelation (Borle *et al*., 2001; Celestino *et al.,* 1997; Shipe *et al*., 1978).

5. Conclusion

The six samples had partly or totally vegetable oil, they are not highly accepted with standard specification, on the storage period extended the quality markedly decreased, recommended to make the validity only three months instead of six months to encourage Egyptian people for consuming UHT milk.

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