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Sudan University of Science and Technology

College of Graduate Studies

**Technical Survey on Quality and
Adulteration of Ghee “Samm Baladi”
Marketed in Khartoum State**

مسح فنى لنوعية الغش فى السمن البلدى المسوق
بولاية الخرطوم

BY

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Verse

الاستهلال

قال تعالى :

{ وَإِذْ لَكُمْ فِي الْأَنْعَامِ لَعِبْرَةٌ لِّسْتَعْتَبُوا مِمَّا فِي بُطُونِهِ مِنْ نَيْزِ فَرَثٍ وَدَمٍ لَبَنًا

خَالصًا سَائِغًا لِلشَّارِبِينَ }

صدق الله العظيم

[سورة النحل الآية "66"]

Dedication

To souls of my parents

To soul of My Brother (Emad)

To My Lovely Family I dedicate this Research.

Acknowledgement

I thank God for giving me strength to accomplish this research.

I would like to express my sincere appreciation and thanks to my supervisor Assoc.Prof.Anas Mohamed Osman for this supervision, continuous encouragement and assistance.

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Abstract

The current Research was conducted to study the quality and adulteration of traditional Samn Baladi, in Khartoum state (Bahri, Khartoum and Omdurman).

Thirty (30) of "Samn" samples were collected randomly from different sales points in the markets of the State, divided in 3 groups, A, B and C, 10 samples per each group.

The analytical tests for moisture and fat content were performed to determine the composition and detect any possible adulteration, Reichert – Meissel and Polenske values for detection of adulteration by animal body fat and vegetable fat (oils), iodine value for the detection of carbohydrates addition .The acidity of the product was also determined .Sensory evaluation was also performed to determine possible alternation in the characteristics and acceptability.

The average moisture% of group A and B obtained was 3.68 ± 3.78 , 3.47 ± 8.27 respectively, which indicated adulteration by incomplete ripening of "Samn" due to insufficient temperature applied during the preparation of "Samn". Samples of group C were normal ($0.75\pm 0.41\%$).A significant variation was recorded between the average % of moisture of A, B and C samples ($P\leq 0.05$).

The average fat %obtained 96.00 ± 9.11 , 96.25 ± 8.31 and 98.94 ± 0.46 for A, B and C respectively, which is less than the value "Samn" should contain .Also significant difference was detected between the averages hereby ($P\leq 0.05$).

The Reichert –Meissel value for A, B and C was 32.42 ± 2.69 , 29.90 ± 4.50 and 30.42 ± 3.73 respectively, which was higher than the normal value (not less than 28) found in the product, indicating no adulteration using animal body fats. The statistical analysis revealed a significant difference in this case ($P\leq 0.05$).

The Polenske value obtained, 3.27 ± 1.10 , 3.3 ± 1.04 and 2.81 ± 0.70 for A, B and C samples respectively, the value obtained is higher than it in normal "Samn"(2.7), which indicates adulteration of the product with

vegetable fats\oils. Significant variation was also recorded hereby ($P < 0.05$).

The average acidity determined as lactic acid %, was 0.21 ± 0.16 for group A samples, 0.13 ± 0.12 and 0.16 ± 0.22 for group B and C respectively. The obtained values are found within the normal limits of acidity values given for normal Samn.

The tested samples of all groups showed no addition of any sort of carbohydrates to the Samn, hence no adulterants were added.

The sensory evaluation for the characteristics color, texture, odour and taste noticed, indicated that the result of the evaluation is fair (satisfactory) for all.

Finally certain recommendations were given.

Arabic Abstract : ملخص البحث :

أجرى هذا البحث لدراسة نوعية وغش السمن التقليدي (البلدي) في ولاية الخرطوم (بحري، الخرطوم وامدرمان). تم جمع 30 عينة سمن عشوائيا من نقاط بيع مختلفة من اسواق الولاية، وقسمت العينات الى 3 مجموعات A, B, C بواقع 10 عينات لكل مجموعة ومن ثم أجريت اختبارات معملية لتحديد محتوى السمن من الرطوبة والدهن بغرض معرفة التركيب او اى غش محتمل. وأيضا لتحديد قيم ريتشارد-ميسيل وبولونسكى للكشف عن الغش بإضافة دهون حيوانية او نباتية للمنتج، اختبار اليود لكشف الغش بإضافة مواد كربوهيدراتية مثل النشا أو الدقيق وغيرها .

كما تم تحديد الحموضة على اساس النسبة % لحامض اللاكتيك في السمن. تم اجراء اختبارات حسية للون والطعم والرائحة والقوام لتحديد اى تغيرات محتملة ومدى القبول للمنتج .

بلغ متوسط الرطوبة % 3.47 ± 8.23 و 3.08 ± 3.73 لعينات المجموعة A و B على التوالي والذي يشير الى الغش لعدم اكمال مرحلة النضج والتي تعزى لاستخدام درجات حرارة غير كافية عند الاعداد. عينات المجموعة C كانت طبيعية في محتوى الرطوبة اذ بلغ 0.75 ± 0.41 تم رصد فرق معنوي بين متوسطات الرطوبة % لعينات المجموعات A, B, C ($P \leq 0.05$)

نسبة الدهن % المتحصل عليها بلغت 96.00 ± 9.11 لعينات المجموعة A، 96.25 ± 8.31 لعينات المجموعة B، 98.94 ± 0.46 لعينات المجموعة C وهذا اقل عن النسبة التي يجب ان تكون بالسمن، ايضا رصد فرق معنوي لمتوسطات نسبة الدهن % بالمجموعات الثلاثة ($P \leq 0.05$).

قيمة رقم ريتشارد - مايسل للمجموعات A, B, C بلغ 30.42 ± 3.73 و 29.90 ± 4.50 و 32.42 ± 2.69 على التوالي والذي هو اعلي من القيمة الطبيعية المقدره بالسمن (not less than 28) مما يشير الى عدم وجود غش باضافة دهون حيوانية للمنتج .

رصد التحليل الاحصائي وجود فروقات معنوية بين المتوسطات الثلاثة من هذه الحالة ($P \leq 0.05$).

قيمة رقم بولونسكى بلغت 3.27 ± 1.10 للمجموعة A، 3.30 ± 1.04 للمجموعة B، و 2.81 ± 0.70 للمجموعة C وهذه القيمة اعلي مما هو مقدر بالسمن (2.7) الذي يعنى غش السمن بإضافة دهون أو زيوت نباتية. رصدت اختلافات معنوية بين المتوسطات أيضا في هذا الحالة ($P \leq 0.05$).

الحموضة والتي قدرت على أساس النسبة المئوية لحامض اللاكتيك بلغت 0.21 ± 0.16 للمجموعة A، 0.13 ± 0.12 للمجموعة B و 0.16 ± 0.22 للمجموعة C. وهذه القيم تقع ضمن المدى المقدم لنسبة الحموضة بالسمن اظهرت نتائج التحليل الاحصائي وجود فروقات معنوية بين متوسطات نسبة الحموضة % لهذه المجموعات .

لم تظهر العينات المختبرة لكل المجموعات وجود اى نوع من المواد الكربوهيدراتية بالسمن مما يشير إلى عدم الغش بإضافة هذه المواد.

أما التقييم الحسى للون والطعم والرائحة والقوام الملاحظة فقد كان مقنعا حسب المحكمين .

أخيرا قدمت توصيات في هذا الخصوص .

CHAPTER ONE

1. INTRODUCTION

Fatty mammary products are those products that can be manufactured or prepared from milk, and are characterized as containing a high percentage of milk fat. Cream, butter, margarine and milk fat are considered as economically the most important components of food (Kenana2006).

According to Spreer (1998), cream products are products that enriched to a varying degree with milk fat, they are non acidified, acidified, whipped and may or may not have additives and also classified according to application, manufacturing process and the fat content.

Milk fat products include as major products ,cream ,butter and anhydrous milk fat ,samn and morta and other milk solids as secondary products as given by Abu zeid(2009).

Samn or ghee is by far the most indigenous milk product with a comparatively long storage life (Sharma 2006).

The concentration of fat in these products (cream, butter and Samn) to prolong the shelf life and in a direct correlation between the degree of concentration of the fat and the length of time saved (Almohandes2009).

In Sudan most of the milk producers process milk into many products such as sour milk (rob),yoghurt(zabadi) , cream(gishda) , butter (zibdeh) , cheese (jibna Beyda) and samn .Samn is widely consumed food commodity in Sudan and its production takes place in villages at house hold level(Abdel basit 2009).

Preparation of Samn is a food security strategy in nomadic life. Although Samn can be stored in certain earthenware jars (kelol)or simple leather bags

(tuggu ,helbatib), it is best stored in a unique dark ,heavy pot like container called butta .This container is build through an elaborate procedure from one of the compartment of camels stomach or from cows raw hide and mixture of ground ,roasted sorghum grain gum (Kadeha) and old ranges as a source of cotton fiber .The container is designed (by women)to reduce rancidity of the fat during the long storage period (Warda et.al.1998).

Food adulterants present in different food stuffs. Like other food stuffs, Samn is exposed to adulteration and different methods are applied in this respect.

Adulteration in food is normally present in its most crude form; prohibited substances are either added partly or wholly substituted. Normally the contamination/adulteration in food is done either for financial gain or due to carelessness and lack in proper hygienic condition of processing, storing, transportation and marketing. This ultimately results that the consumer is either cheated or often become victim of diseases. Such types of adulteration are quite common in developing countries or backward countries. It is equally important for the consumer to know the common adulterants and their effect on health (Elkholi1999).

Objectives:

1. To evaluate the quality of ghee "Samn".
2. To study the different methods of ghee "Samn" adulteration.

CHAPTER TWO

LITERATURE REVIEW

2.1 Definition of Ghee"Samn":

Samn or ghee is considered as one of the most important concentrated milk fat product and it is produced in some African and Asian countries, while in Europe its production is very limited, since the possibility of storing butter in cold stores is available (ELNimer2007).

Winton and Winton (2010) noticed that ghee is a clarified and ripened butter fat obtained from cows and buffalos milk in India and other eastern countries.

Samn or ghee is concentrated fat, obtained by the removal of water and solids from butter or cream and its making convenient method for the preservation of fat (NiiR 2007).

The Sudanese Standards and Metrology organization SSOM (2007) described Samn as that dense food stuff extracted from milk, cream or butter after the removal of the non-fat solids and moisture and has specific flavor and consistency.

Subhasish and Battacharyya (2006) stated that, according to PFA (1976), ghee means the pure clarified fat derived solely from milk or from curd or from butter or from cream, to which no coloring matter or preservatives have been added.

Amr (1991) explained that Samn is the hydrous form of milk fat obtained from cream or butter of various animal species. While Blume (2013)

mentioned that rendering butter produces clarified butter or ghee, which is almost entirely butter fat.

The most important general requirement is that the fat is very pure and stable to autoxidation. To secure the good quality fresh milk should be used. Contamination by traces of copper is highly detrimental. The water content should not exceed 0.1%, because otherwise moisture droplets may form at low temperature. If the water content is higher (up to 0.4%), the product is usually designated (Walstra et al. 1999).

2.2 Classification of Samn

Samn can be classified according to the preparation methods applied. Accordingly following types are found;

2.2.1 Traditional Samn:

This type is produced by applying various traditional methods, and it is dominating over other types especially in developing countries. Such type is known as Samn Baladi in Sudan and ghee in India.

2.2.2 Industrial Samn:

Known as anhydrous milk fat or butter oil, which is prepared by applying modern techniques, whereby the milk fat is developed to produce this type. The high fat % is common for both Industrial and traditional types.

2.3 Composition and Properties of Samn:

2.3.1 Chemical composition:

Samn composed of not less than 97% fat, not more than 1% moisture and not more than 1% salt (Abu zeid 2009). (AlNimer 2007).

The SSMO (2007) gave 99.6%fat as lowest limit and 0.3%moisture as highest limit.

Subhasish and Battacharyya(2006) explained, the milk fat% ranges between 99-95%,the moisture not more than 0.5%and the un- saponifiable matter 3.2-7.4 IU\g ,19.34 IU\g and 26.48mg\g for carotene, vitamin A and Tocopherol respectively.

NiiR (2007) noticed that the butter fat and moisture are 99.5-99.8%, 0.1-0.3% respectively.

According to Codex Alimentarius (2001), the fat is not less than 99.6%, moisture not more than 0.2%.

Abulehia and Hamza (1995) noticed, the fat should not be less than 99.6%, the non fat- solids less than 0.3%and the moisture should not exceed 0.5%.

Also NiiR (2007) estimated the butter fat of ghee to range between 99.5 %and 98.8% and the moisture 1-0.3%.

Nath et.al. (1996) reported that ghee contains 0.2-o.4%cholesterol and this present in both free as well as in esterified form as noticed by Bindal and Jain (1972).

The slight variation between the components of the product may be related to the heat intensity used as well as original fat content found in the utilized raw milk used to produce cream or butter (Osman 2007).

2.3.2 Physicochemical properties:

According to Subhasish and Battacharyya (2006), the physicochemical properties are as follows:

Table 1: Physicochemical properties of samn:

Properties	Value
Melting point	Varies from 28-44°C
Solidifying	Varies from 15-28°C
Specific gravity	Varies from 0.93-0.94
Refractive index	Varies from 40-45
Reichert-Meissel(RM)value	Not less than 28
Polenske(P) value	Not more than 2
Saponification value	Not less than 220
Iodine value	Varies from 26-38

The Sudanese standards and meteorology Organization SSMO (2007) issued the following values:

Table2: Physicochemical properties of samn:

Properties	Value
Melting point	27-33°C
Saponification factor	220-235
Acidity (estimated as oleic acid W\W%)	0.4(highest limit)
Peroxide value (estimated as O ₂ \kg fat)	0.6(highest limit)
Iodine value	26-38

Soure: SSMO (2007)

Abu zeid (2009) explained the physicochemical properties of Samn as follows:

Table 3: Physicochemical properties of Samn:

Properties	Value
Refractive index	40-44
Saponification factor	Not less than 220
Reichert-Meissel value	Not less than 22
Polenske value	Not more than 2.7
Kreshner value	Not less than 19
Acidity	Not exceeding 10%

Soure: Abuzeid (2009)

3.3 Sensory Characteristics:

3.3.1 Flavor:

SSMO (2007) stated, the product should have an outstanding taste, free from rancidity or undesirable flavor.

The flavor of Samn is made up by volatile and non –volatile compounds as given by Iskandar et.al (1995).

According to NIIR (2007) ghee has agreeable taste and might have raw (bland) and slightly cocked (caramelized) flavor, depending on the quality of raw material and method of production.

Mahindra (2009) explained, the watery components of butter contributes to aroma during melting down, and when ghee is made by heating butter that has been derived from an unripend cream has rather flat flavor, .it is even worse when ghee is made by simply boiling down fresh unripend cream, since there is much water in cream (65%) than in butter (15%).

3.3.2 Color:

The color of Samn may vary from slight creamy white to dark creamy yellow or orange yellow, where by this variation in color is associated with the season of the year, absence of salt and type and amount of natural color added (Lambert, 1975).

The color of Samn should be white homogenous (SSMO, 2007), and of that from cow's milk is deep yellow (NIIR2007).

3.3.3 Odour:

The product should have a distinguished smell, and free from undesirable foreign smells as indicated by SSMO (2007).

ELNimer (2007) noticed a significant difference between the smell of traditionally processed Samn and that prepared by applying modern methods (Anhydrous milk fat, Butter oil), since the latter have not that distinguished odour of the native one.

In general Samn has a natural sweet pleasant odour as indicated by NIIR (2007).

3.3.4 Texture:

Grains of large uniform size and a firm and non-greasy consistency is preferred, where by the grain size in Samn is influenced by several factors, such as rate of cooling, fatty acids make up and subsequent heating and cooling treatment (Srinivason and Lakrishnan 1984).

According to SSMO (2007), depending on the temperature the texture of Samn should be grainy or liquid and not solidified.

NIIR (2007) mentioned, the texture of the product, when no special conditions of cooling are adopted is pasty.

4. Nutritive value and Utilization:

4.1 Nutritive value:

Samn is the richest product in milk fat compared with other dairy products. It contains also a large amount of fat-soluble vitamins, A, D, E and K. It provides the human body with sufficient heat and energy.

Sharma (2006), noticed that ghee prepared by traditional methods vary low in fat –soluble vitamins A and D and contains appreciable quantity of these only under prescribed conditions.

Sserunjogi et.al .(1998) described the product as the best form of fat, helps in enhancing power, rejuvenates the skin inside and increases its glow, beside providing energy.

Ghee contributes significantly towards nourishment of people of all age groups. It is a good source of fat soluble vitamins (A, D, E, and K) and essential fatty acids (Rangappa and Achaya 1974).

The digestibility co-efficient or the rate of absorption is 96% which is better than any other animal or vegetable fat (Mohammed; 1979) according to Ahmed (2012).

4.2 Utilization and Uses:

NIIR (2007) summarized the utilization and uses of the product; it is used as cooking fat, in ice-cream manufacturing, in the confectionary industry and also used in manufacturing of various types of spreads.

According to Dirar (1993), the major uses of "Samn", among those who produce it, namely the nomads of Sudan are the following:

As a tapping formula (type of food) particularly made from Rob, many people drink Samn on its own in small quantities and given to children either pure or mixed with salt to rub the skin as a medication against fever and rheumatism.

5. Manufacturing Methods of Samn:

The manufacture of Samn is based on three technological steps, which involve the concentration of the milk fat, heat treatment of the obtained fat rich product and finally clarified (strained) to remove the non-fat solids and residues, then packed (Osman 2007).

5.1.2 Milk Fermentation Method:

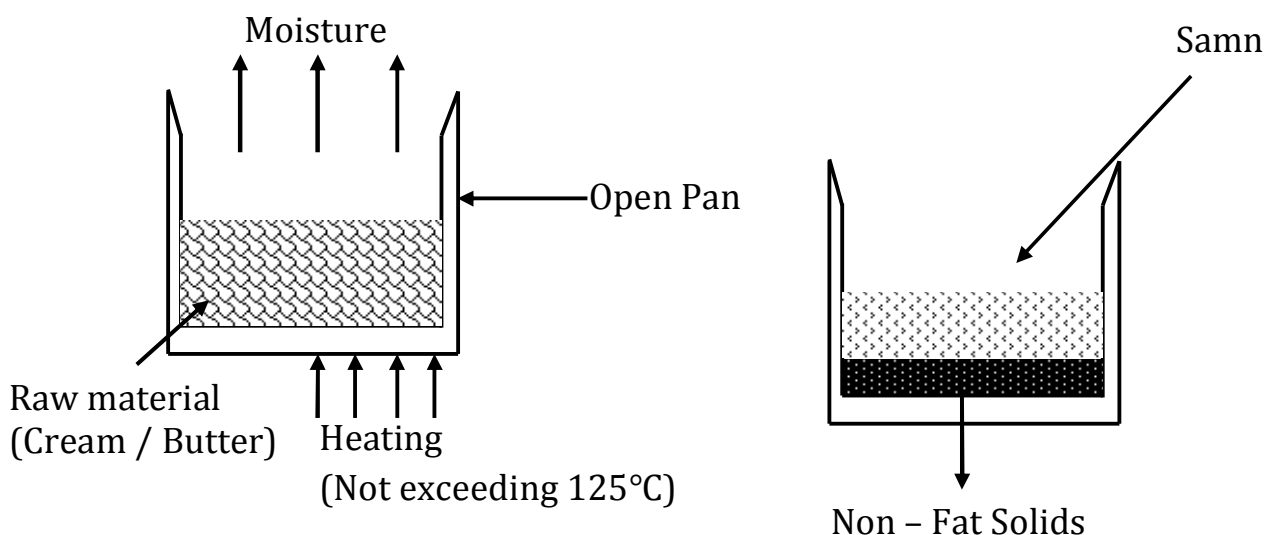
Here, the raw milk is left till it gets sour, the curd obtained then churned using a simple available device (siin, closed containers e.t.c) and as a result a curde butter (called Forsa in Sudan) is formed, which is then separated from the butter milk (called Rob), heated in open pan (Temperature varies between 100-125°C) to remove the moisture, clarified cooled and finally packed.

Abdel Malek (1978) defined forsa as the product of cream separation from soured milk by traditional churning method.

5.1.3 Melting Industrial Raw Material Method:

Ready processed cream or unsalted butter are heat treated at 110-125c in an open pan till the moisture evaporated and the non-fat solids precipitate at the bottom of the pan, (stirring helps here by).The obtained Samn is then separated from the non-fat solids (straining) and then packed.

Fig.2. Steps of production:



5.2 Modern Methods:

Such methods are applied in advanced dairy plants. The processed product is called anhydrous milk fat or butter oil. The milk fat is concentrated using standard separators. The manufacturing procedure varies from one method to another.

5.2.1 Processing method from cream:

According to Abu zeid (2009) this method involves the following steps:

Cream (30-45% fat) is pasteurized at 72°C, cooled to 55°C and then preconcentrated using special separators. Then the situation fat in water is converted into water in fat using a centrifixator to destroy the layers enclosing the fat globules. With the aid of standard separators the obtained butter is converted to anhydrous milk fat (butter oil) having not less than 99.5% fat. The product is then dried at 90°C, cooled to 30°C using a plate heat –exchange, packed and stored in a cool store at 5°C.

5.2.2 Processing method from Butter (1):

The so- called continuous unit system for ghee making is described by Suhasish and Battachanyya (2006) as follows:

Butter is received in a jacketed vat, melted by steam (40°C), preheated in a plate heat exchange and the obtained product is then flushed in air vapour separator to remove a part of the moisture and then pumped to the second plate heat exchange and heated to 115°C, flushed again in a vapour separator, then collected in a vat and finally clarified using a centrifuge.

5.2.3 Processing Method from Butter (2):

According to Abu zeid (2009) salted butter is washed to remove the salt, melted at 40°C, separated (99.5%fat) and then water (about 10%) is added at 90C° to get rid of the phospholipids, and then separated by a separator. The product is finally dried under vacuum by passing the hot Samn (90°C) to a vacuum room to decrease the moisture content.

According to Abdel Hamid et.al. (2001), samn produced from fat concentrated or fermented cream, compared with that made of butter, consumes longer preparation time, has high amount of morta, cooked flavor and smaller grains.

6. Packaging and Shelf life:

6.1 Packaging:

The Sudanese Standards and Meteorology Organization- SSMO - (2007) stated, Samn should be packed in clean dry containers, tightly sealed to prevent any possible external effects or interaction between product and the pack material.

Kanli (2007) explained, since milk fat is susceptible to deterioration due to exposure to light, air and metals, samn (which is made up entirely of milk fat) should be properly packaged promptly after production so as to retain its initial flavor and nutritive value. The packaging temperature as suggested by Abdel Hamid et.al (2001) 45-50°C and possible up to 60°C.

Abu Lehia and Hamza (1995) noticed, samn should be packed in stainless metallic packs, tightly sealed, dark colored, and stored in a cool store.

6.2 Shelf Life:

SSMO (2007) gave a shelf life of 12 months after the production date at 25°C. Zidan (2004) noticed, good prepared samn has a shelf life of 6 Months at room temperature, more than 1 year at 4°C and more than 2 years at 10°C.

The shelf life of samn varies from 3-12 months, 1-3 years and 1-4years at storage temperature of 22°C (room temperature) 10-15°C and 5°C respectively (Abu Iehia and Hamza 1995).

In order to prolong the shelf life of samn, sometimes several certain antioxidants substances are permitted to be added. As given by Fearson et.al. (1998), natural antioxidants are also believed to contribute to the extension of shelf life of ghee.

7. Deterioration of Samna:

According to Bushara (2012), Ada Hayat Tadriss (2010) and Abu Daaod et.al (2003), several factors influence samn deterioration and spoilage, such as:

- Excess moisture content.
- High storage temperatures.
- Contamination with metals (e.g. copper, iron).
- Exposure to light for long periods.
- Enzymes analysing fat.
- Addition of certain antioxidants.
- Improper pasteurization of cream.
- Improper clarification or separation.

Defects that appear in samn according to Bushara (2012), ELNimer (2007) and Sharma (2006) include:

Color defects:

Dark color due to overheating and discoloration due to low temperature during ripening, beside fat oxidation.

Texture defects:

Related to fat oxidation as a result of interaction between dissolved oxygen and unsaturated fat, and also to the influence of enzyme lipase which hydrolysis fat.

Compositional defects: Associated with non-fat solids and residues found in samn, when they are not completely removed during heat treatment.

8. Morta:

Morta is the by-product obtained as a result of processing samn.

Zidan (2004) defined morta as all precipitating substances during samn preparation from cream or butter with high amount of phospholipids and composes in average of 10-18% water, 43-60%fat, 20-35%organic non-fat substances and 10-20% ash.

ELNimer (2007) described morta as the secondary product when making samn and contains all non-fat substances that precipitate during samn preparation and has a high nutritive value for human consumption beside its utilization as supplement in the ration of animals.

9. Methods of adulteration and detection:

Different methods are used to adulterate samn that in turn result in certain defects and low nutritive value.

Ravi (2012) and ELNimer (2007) noticed that adulteration methods and how to detect can be given as follows:

9.1 Adulteration by adding animal fat or plant fat \oil:

9.1.1 Vegetable fat\oils:

Such adulterants are hydrogenated or non- hydrogenated vegetable oil. These oils are by far the most used, because of their resemblance in texture-grain, color and other characteristics of samn.

9.1.2 Animal body fat:

This type is not as common as with vegetable fat\oil. The obtained fats are mixed with samn in different proportions and difficult to detect visually.

9.1.3Detection:

Adulteration of samn with animal or plant fats can be detected by sensory evaluation to determine any alteration in texture, taste and odour compared with natural samn.

The chemical analysis tests are more accurate. They indicate that the soluble volatile fatty acids % increases, when animal fats are added , decreases by addition of the plant fat\oils (Reichert-Meissel value :not less than 28).The %of the un soluble volatile fatty acids decreases by adding animal fats , but increases by addition of vegetable fat\oils (Polenske value: not more than 2.7).

9.2 Adulteration by Carbohydrates:

Samn originally contains no carbohydrates. Small quantities of starch, flour or other from rice or potatoes are added to the samn for improving its consistency and better grain formation.

9.2.1. Detection:

One drop of iodine solution is added to the adulterated sample, the change in color of the solution from light brown to blue indicates the presence of carbohydrates in the product.

9.3 Incomplete Ripening:

When the moisture is not completely removed during the preparation of samn, a part of it is left, which means excess moisture in it.

9.3.1 Detection:

Carried out by observing the texture of the product. Not grainy texture is a sign of excess moisture. More accurately by performing chemical testes, since the moisture content should not be more than 1% in the product, otherwise it is adulterated.

9.4 Increasing weight by adding sodium chloride:

The NaCl %in samn should not be higher than 1% usually the salt is added to the bottom of the containers used for packaging.

9.4.1 Detection:

The detection is carried out chemically to determine the % of salt present in the product, or by taking a sample from the bottom of the pack and observing the presence of the salt.

Recently many advanced and sensitive techniques, such as thin layer chromatography, paper chromatography and gas liquid chromatography have developed for detecting samn adulteration, but they are expensive and time consuming.

CHAPTER THREE

3. MATERIALS AND METHODS

This study was conducted during the period April-September 2014 at the Department of Dairy Science and Technology, College of Animal Production Science and Technology, Sudan University of Science and Technology.

3.1 Sampling:

Thirty (30) samples of Samna produced traditionally were collected randomly from different sales points of the markets of Khartoum State. The samples were then divided into 3 groups, 10 samples per each group, (groups: Bahri, Khartoum and Omdurman) and then subjected to following analysis according to A.O.A.C. (2000).

3.2 Chemical Analysis:

3.2.1 Determination of Moisture:

Apparatus:

- Moisture dish.
- Dessicator.
- Air oven.

Procedure:

Weight accurately about 10 g of the sample in a moisture dish which has been dried previously, place in air oven for 1 hour at $105 \pm 1^{\circ}\text{C}$. Remove the dish from the oven; cool in the desiccator and weight. Repeat this procedure by keeping the dish in the oven for half hour each time, cool and weight till two successive weights do not exceed 1mg.

Calculation:

$$\text{Moisture \%} = m \frac{(m_1 - m_2)}{(m_1 - m)} \times 100$$

Where:

- M_1 : mass in g of dish with ghee before drying
- M_2 : mass in g of dish with ghee after drying
- M : mass in g of empty dish (IS 1966).

3.2.2 Determination degree of acidity:

Apparatus:

- Conical flask.
- Burette.

Chemicals:

- Ethanol alcohol.
- NaOH(.01 N).
- Phenolphthalein Indicator.

Procedure:

Weigh 10 g of ghee sample in a 250 ml conical flask. In another flask bring 50 ml of ethanol to the boiling point and while still above 70°C, neutralize it to phenolphthalein (using .05 ml) with 0.01 N NaOH. Add the neutralized alcohol to flask containing ghee sample and mix the contents of the flask. Bring the mixture to boil and while it is still hot, titrate with 0.1 N NaOH, shaking vigorously during the titration. The end point of titration is reached when the addition of single drop produces a slight, but a definite color change persisting for at least 15 sec.

Calculation:

Degree of acidity is the total titratable acidity present in the sample of ghee expressed as percentage:

$$\text{Degree of acidity} = \frac{N}{M} \times 100$$

Where:

- N = ml of 1N alkali used for titration.
- M = mass in g, of ghee sample(IS1966).

3.2.3 Iodine test: (ElNimer2007)

Apparatus:

- Test tubes.
- Pipette.

Reagents:

Potassium iodide 1%

Procedure:

2ml of sample were put in a test tube, then added 2 drops of lode solution, if the color converts to blue that means this sample contains starch.

3.2.4 Determiration of Fat: (Abu Daoad et. al, 2003)

Apparatus

- Gerber tube
- Centrifuge

Reagents:

- Sulfuric acid.
- Amyl alcohol.
- Distil water.

Procedure:

Weight 5g \pm 0.01 of ghee sample in Gerber tube then add 10cm³ distilled and put the tube in water bath (70°C) until water and 10cm³ H₂SO₄ all sample melts then 10cm³ amyl alcohol were added put Gerber tube in the centrifuge 1200 rotations per minute for 5 minutes.

3.2.5 Determination of Reichert-Meissel and Polenske Value (IS 3508-1966):

Apparatus:

- Graduated Cylinders
- Pipette
- The assembly of the apparatus for the distillation
- Glass funnel

Chemicals:

- Glycerol 98%
- Dilute sulfuric acid
- Sodium hydroxide (50% (m\m))
- Ethanol 95%
- Phenolphthalein indicator 0.5%
- NaOH solution 0.1 N
- Barium hydroxide solution 0.1 N

- Silver sulfate
- filter paper

Procedure:

- A. Weigh 5 ± 0.01 g of ghee sample into a Polanski flask. Add 20 g of glycerol and 2ml of 50%NaOH solution. Heat the flask over a direct flame using a Bunsen burner, with continuous mixing, until ghee, including any drops adhering to the upper parts of the flask, is saponified, and the liquid becomes perfectly clear, avoid overheating during this saponification. Cover the flask with a watch glass.
- B. Make a blank test without ghee, but using the same quantities of reagents and following the same procedure, again avoiding overheating, and such overheating would be indicated by darkening of solution. Measure 93ml of boiling distilled water, which has been vigorously for 15 min, into a 100ml graduated cylinder.
- C. When the soap is sufficiently cool to permit addition of the water without loss, but before the soap has solidified, add water, draining the cylinder for 5sec, and dissolve the soap. If the solution is not clear (indicating incomplete saponification), or is darker than yellow (indicating overheating), repeat the saponification with a fresh sample of ghee. Add two glass beads, followed by 50 ml of the dilute sulfuric acid, and connect the flask at once with the distillation apparatus.
- D. Heat the flask without boiling its contents, until the insoluble acids are completely melted then increase the flame and distill 110ml in between 19 and 21 min. Keep the water flowing in condenser at a sufficient speed to maintain the temperature of the issuing distillate between 18 and 21°C.
- E. When the distillate reaches the 110ml mark, remove the flame and replace the 110ml flask by a cylinder of about 25 ml capacity, to collect

draining. Close 110ml flask with its stopper and without mixing the content; place it in water at 15c for 10 min so as to immerse the 110 ml mark .Remove the flask from the water, dry from outside ,and invert the flask carefully avoiding wetting the stopper with insoluble acids. Mix the distillate by four or five double inversions, without violent shaking.

- F. Filter through a dry 9cm open –texture filter paper (What man filter Grade 4) which fits snugly into the funnel. Reject the first running and collect 100ml in a dry volumetric flask, cork the flask and retain the filtrate for titration.
- G. Detach the still-head and wash the condenser with three successive 15ml portions of cold distilled water, passing each washing separated through the cylinder, the 110ml flask, the filter and the funnel, nearly filling the paper each time and draining each washing before filtering the next. Discard the washing.
- H. Dissolve the insoluble acids by three similar washing of the condenser, the cylinder, and the filter, with 15ml of neutralized ethanol, collection the solution in the 110ml flask and draining the ethanol after each washing. Cork the flask, and retain the solution for titration (for Polanski value).
- I. Reichert- meissle or soluble volatile acid value :pour 100ml of the filtrate (obtained in step F) containing the soluble volatile fatty acids into a titration flask ,add 0.1 ml of phenolphthalein indicator and titrate with 0.1N NAOH solution until the liquid becomes pink .
- J. Polanski or insoluble volatile acid value: Titrate the alcoholic solution of insoluble volatile acids after addition of 0.25 ml of phenolphthalein indicator, with the 0.1N barium or sodium hydroxide solution until the solution becomes pink.

Calculation:

$$\text{Reichert-Meissle Value} = 1.10(T_1 - T_2)$$

$$\text{Polanski Value} = T_3 - T_4$$

Where:

- T_1 = volume in ml of 0.1N barium or sodium hydroxide solution used for sample under step I,.
- T_2 = volume in ml of 0.1N barium or sodium hydroxide solution used for blank under step I,.
- T_3 = volume in ml of 0.1N barium or sodium hydroxide used for sample under step J,.
- T_4 = volume in ml of 0.1N barium or sodium hydroxide solution used for blank under step J; (IS 1966).

3.3 Sensory Evaluation:

Ten (10) trained panelists were chosen to evaluate color, texture, odor and taste of Samna samples, giving a certain degree (out of 100) for property tested for each sample. The average of the degree attained is then calculated. The evaluation is given in the following table according to Bhavan (1975):

Table4: Evaluation of properties quality

Quality	Score(out of 100)	Grade
Excellent	100-90	A
Good	89-80	B
Fair	79-60	C
Poor	59 and below	D

4.4 Statistical Analysis:

The data obtained will be analyzed using Statistical Package for Social Sciences (SPSS) Program (ANOVA) to determine possible variation among means.

CHAPTER Four

4. Result and Discussion

4.1 Result:

The obtained result is given in the following tables:

Table5: Compositional and physical properties

Parameters Group	Moisture%	Fat%	Rechert- meissle value	Polenske value	Acidity
A	3.68±8.78a	96.00±9.11b	32.42±2.69a	3.27±1.10a	0.21±0.16a
B	3.47±8.27a	96.25±8.31b	29.90±4.50a	3.30±1.04a	0.13±0.12c
C	0.27±0.41b	98.94±0.46a	30.42±3.73a	2.81±0.70a	0.16±0.22b
P-value	**	*	NS	NS	*

NS: Not- significant

Significant: $P \leq 0.05$

-Mean+SD values having different superscripts letters in the same raw are significant different ($p \leq 0.05$).

-Mean +SD values same superscripts letters in the same raw are not significant different ($p \leq 0.05$).

Table6: Adulteration by carbohydrates detection

Group	Result
A	Neg.(-)
B	Neg.(-)
C	Neg.(-)

Table7: Sensory evaluation result

Parameters Group	Color	Texture	Odor	Taste
A	73.53±12.24a	72.10±13.52a	67.88±15.50a	70.88±14.86a
B	71.21±14.29b	69.89±14.22b	64.32±13.69b	64.25±13.79b
C	70.21±11.73b	68.37±13.51b	64.70±13.45b	64.33±12.09b
P-value	NS	NS	*	**

Level of significance:

NS: Not significant at 0.05.

-Mean+SD values having different superscripts letters in the same raw are significant different ($p \leq 0.05$).

-Mean +SD values same superscripts letters in the same raw are not significant different ($p \leq 0.05$).

4.2 Discussion:

The laboratory analysis and sensory evaluation result of the Samn indicated the following:

Moisture Content:

The average moisture % of group A, B and C samples obtained was 3.6 ± 8.73 , 3.47 ± 8.27 and 0.75 ± 0.41 respectively. The average moisture% of group A and B is very high compared with that of group C. The values of both group A and B contradicts those given by SSMO (2007), Abu zeid (2009), ELNimer (2007). Since the moisture % in Samn should not be more than 1%, the values above (Group A and B) indicate adulteration of the product. This is associated with incomplete ripening of the product during preparation, due to not proper and insufficient heating temperature used and thus a part of the moisture is left (incomplete Removal) in the product. The samples of group C showed normal moisture content (less than 1%). The statistical analysis indicated no significant difference between the average percent of the moisture of group A and B, while a high significant variation is recorded between both and group C. ($P \leq 0.05$).

Fat Content:

The average fat% obtained for group A, B, and C was 96.00 ± 9.11 , 96.25 ± 8.31 and 98.94 ± 0.46 respectively. The high fat% was found in group C. In spite, all values recorded for the three groups are less than that required in the product (SSMO 2007), (NiiR 2007), (Subhasish and Batacharyya 2006), (Codex Alimentarius 2001).

The variation in the fat content of the product may be related to the insufficiency of the melting temperature applied during processing. The statistical analysis showed no significant difference between the average of the

fat% of group A and B while a significant difference was noticed between A, B and Group C. ($P \leq 0.05$).

Reichert-Meissle Value:

The average of Reichert-Meissle Value obtained, 32.42 ± 2.69 , 29.90 ± 4.50 and 30.42 ± 3.73 for samples of group A, B and C respectively. This value when increases more than 28, indicates that the % of the soluble volatile fatty acids also increase when the product is adulterated using animal fats. Furthermore, this value also decreases, when plant oils added. The tested samples of the three groups are thus not exposed to adulteration using animal fats. The Reichert -Meissle value obtained (not less than 28) agrees with those given by Subhasish and Batacharyya (2006) and Abu zeid (2009). The statistical analysis indicates no significant difference between values average of group A, B and C.

Polenske Value:

The Polenske Value obtained, 3.27 ± 1.10 , 3.30 ± 1.04 and 2.81 ± 0.70 for samples of group A, B and C respectively. As noticed by ELNimer (2007), Subhasish and Battacharyya (2006) and Abu zeid (2009), this value should not be more than 2-2.27, otherwise the product is adulterated by adding vegetable fat/oils, since the un-soluble volatile fatty acids found in the product increase by adding vegetable fats/oils and decrease by addition of animal fats.

The tested samples of all groups subject of current study were adulterated by vegetable fats/oils. The statistical analysis showed no significant difference between the averages of this value for group A and B, C.

Acidity:

The acidity as lactic acid % found in group A, B and C samples, was 0.21 ± 0.16 , 0.13 ± 0.12 and 0.16 ± 0.22 respectively. The values obtained are within the limits of acidity values as given by Abu zeid (2009), ELNimer (2007) and Abdel Hamid et.al. (2001). The statistical analysis revealed difference between the averages of acidity of group A, B and that of group C ($P\leq 0.05$).

Adulteration by Carbohydrates:

The laboratory analysis showed no carbohydrates e.g. starch, flour or other sorts in the tested samn samples of Group A, B and C .This means no adulteration by adding carbohydrates has been carried out.

Sensory Characteristics:

As mentioned above, the samples of all groups were exposed to adulteration by incomplete ripening due to varying heat temperatures applied during the preparation of samn, beside the addition of plant fat/oils adulterants. Hence an expected abnormal change in the sensory characteristics is expected. According to panelists and evaluation using the score method adopted by Bhavan (1975) (see table 4), following was noticed:

- **Color:** Fair
- **Texture:** Fair
- **Odour:** Fair
- **Taste:** Fair

However, significant variation was noticed between the averages given for a certain characteristics per each group (see table 7).

CHAPTER FIVE

5. Conclusion and Recommendations

5.1 Conclusion:

Concentrated milk fat products, such as cream, butter and samn or anhydrous milk fat, play a vital role in human nutrition. In Sudan, samn is dominating nutritionally over other concentrated milk fat products, and produced in a large scale compared with butter or cream .Jet, it is manufactured by applied known traditional methods. Production of anhydrous milk fat, butter oil on industrial basis is very limited. The most important consumer attributes of samn, are its flavor, odour, color , texture and composition .So, in order to produce a high quality samn, it is important to follow the proper procedures of processing, avoid the addition of any kind of adulterants and pay attention to packaging and storing. The current study investigates how far the produced samn meets the standard required.

5.2 Recommendations:

- To make sure that produced samn satisfies the standards required by Sudanese Standards and Meteorology Organization.
- More attention should be paid to the preparation of samn using traditional methods in terms of restrict hygiene measurements and complete ripening of the product.
- To apply modern processing methods, if possible.
- More research and studies should be carried out on traditional "Samn".

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