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DETECTION OF MILK FAT ADULTERATION USING DIELECTRIC PROPERTIES

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ABSTRACT

Nowadays, milk adulteration and, in particular, milk fat adulteration represents a big problem. Because of its high price, ghee and butter has always been the object of adulteration by adding less expensive vegetable oils or animal fat. The determination of fatty acids by gas chromatography (GC) followed by calculation of suitable ratios is commonly used to detect extraneous vegetable oils or animal fats, but with the introduction of some lipids sources in cow's and buffalo's diets, the detection of extraneous fat become difficult. The dielectric properties (Breakdown voltage (KV) test) of oils are electrochemical and thermo-physical property widely used to determine the shelf life of mineral oils used in transformers. This property was determined in some vegetable oils, some animal fats, milk fats and margarine oil to see whether there is a big variation between them easy to be used to distinguish between these oils and fats or not. Also, this property was determined in mixture of milk fat with different edible oils and animal fat in different ratios. The obtained results indicated that it could be possible to depend on the dielectric breakdown voltage as a rapid and easy method to detect milk fat adulteration with other vegetable oils and animal fats.

Keywords: Milk fat (Ghee), Adulteration, Vegetable oils, Margarine, Animal fats, Dielectric breakdown voltage.

INTRODUCTION

Butter and samna(Ghee) are of commercial importance because they are the most expensive source of milk fats and, consequently, they must have elevated standards of quality .Special regulation and strict standards for butter and samna have established that butter and samna must be obtained exclusively from cows ,buffaloes or other animal's milk (goats , sheep and camel).

Because of its high price, Ghee (samna) and butter have always been the object of adulteration by adding less expensive vegetable oils or animal fats (Cani et al.., 1994). The addition of extraneous fat to milk fat can be detected through several analytical methods. The determination of phytosterols allows the detection of small amounts of vegetable fat (Mariani *et al.*, 1994).

The analysis of fatty acids followed by calculation of suitable ratios is commonly used to detect extraneous animal fats, although fatty acids are strongly subject to variations due to natural factors such as season and period of lactation. In recent years, these variations have increased due to the introduction of some lipids sources (i.e. whole cottonseed and flaked soybean) in cows and buffalo's diets .consequently, the addition of extraneous animals' fats especially of small amounts to milk fat was hardly detectable.

From the above-mentioned review, it could be said that there is no single method by which it can be easily detect milk fat adulteration either by animal fat or by vegetable oils. Therefore, it has become urgent to think and search for a new method for detecting milk fat adulteration. It is well known that fats and oils are generally characterized by specific properties e.g. refractive index, density, saponification and iodine values in addition to electro chemical and thermo-physical properties known as dielectric. This property is very important for people working in the field of transformers as they usually use mineral oils for liquid insulation in high voltage equipments (transformers) to determine to what extent the mineral oils keep its dielectric properties.

Nowadays, mineral oils are still widely used as insulators for high voltage transformers. However, due to environmental consideration, many researches (Blackburn *et al.,* 2006 and Suwarno and Darma, 2008) have been carried out in an attempt to search the alternative of liquid insulating materials which are friendly to the environment; therefore, vegetable oils were tested for their dielectric breakdown voltage and now are widely used in a mixture with mineral oils (Lucas *et al.*, 2001). According to what previously reported, it was thought worthwhile to ask ourselves these questions "Does vegetable oils have the same dielectric breakdown voltage for animal fat or milk fat? Is it possible to distinguish between milk fat and other sources of fats, via dielectric breakdown voltage property, and as a result detect the milk fat adulteration.

Therefore, this study was designed to determine the dielectric breakdown voltage of vegetable oils, milk fat from buffaloes, cows and camel milks and animal fats and was compared with that of mineral oil and each other. Also dielectric breakdown voltage of milk fat mixed with other fat sources at different ratios was also determined in a trail to find out an easy and rapid method to detect milk fat adulteration.

MATERIALS AND METHODS

- **Mineral oil**: extra super oil produced by Petrolmen Oil Company was purchased from the local market.
- **Vegetable oil**: palm oil (shortening) and palm kernel oil (tekerline) were obtained from some dairy factories at 6.October city and El-Obour city. While sunflower oil, Soybean oil, Rawaby (vegetable samna) and margarine were purchased from one of the biggest super markets in Giza.
- **Animal fat**: Cows, sheep (fat tail), camel (hump), and lard (pork fat) were purchased from different butcher's shops in local market.

Pure milk fat (Samna or Ghee) were laboratory prepared from:

- Raw milk percured from cows and buffaloes herds maintained at faculty of Agriculture, Cairo University.
- Butter, manufactured from marketed milk at the dairy technology unit, at the faculty of Agriculture, Cairo University.
- Camel's milk purchased from a camel's farm (Ebel-Elkher) at Aborawsh, Giza, Egypt.
- Potassium hydroxide, diethyl ether and ethyl alcohol pure grade were obtained from El Nasr Pharmaceutical Chemicals Co., Egypt.

- For the preparation of milk fat : Each of Buffaloes and cows' milk was separated into skim milk and cream, the resultant cream was churned into butter, then butter was converted into Samna by boiling according to the method described by Fahmi (1961).
- Butter obtained from the dairy technology unit was converted into Samna following the above mentioned method.
- Camel milk was separated into skim milk and cream, then cream was converted into Samna by directly boiling because its cream was difficult to be churned.
- For the preparation of animal fat, cows, sheep (fat tail), camel (hump), and lard (pork fat) were heated at 110°C until completely melted. The melted fats were filtered while warm through filter paper Whatman no.1 to obtain tissue free lipids.

Margarine oil was obtained following two ways; by boiling in a way similar to the method of Samna, or by extracting the oil by diethyl ether (40-65 °C) in a separating funnel.

Acid value for all oils and fat samples was determined according to the method described by AOAC (1984).

Moisture content in oil and fat samples was determined using moisture analyzer (Sartorious MA 30 AG Gottingen, Germany).

Determination of the dielectric breakdown voltage of oil was done by using disk electrodes following the method described in the ASTM D877 standard (1980).

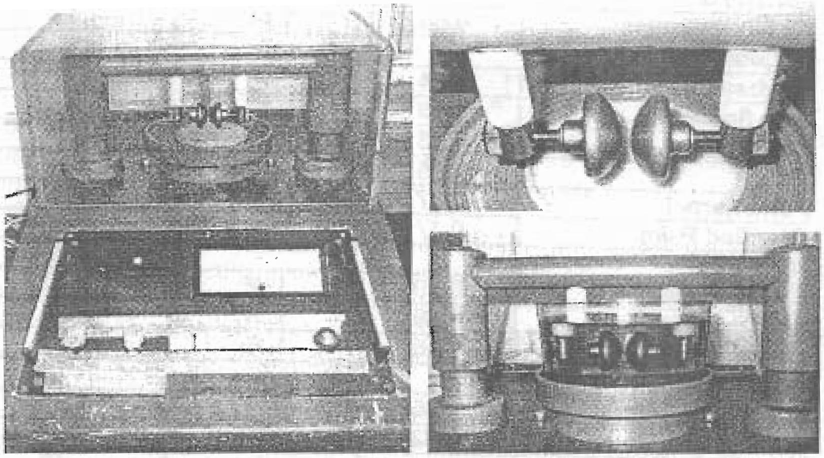

Figure (1): Equipment for determination of the dielectric breakdown voltage (KV) of oils (MGB OLPRUFGERAT OP Type: OP 80 H) from MGB SEISSEN. West Germany.

As shown in Fig (1), the disk electrode system utilizes 25 mm diameter square-edged disks separated by 2.5 mm. The cell is filled with oil to cover the electrodes to at least a depth of 20 mm and the sample is allowed to set for at least 2 minutes without agitation. A 60 Hz sinusoidal wave voltage is applied at a ramp rate of 2000 V/s until breakdown occurs as indicated by passage of a current through the sample of 20 mA. This occurrence is used to trip a relay within 3 to 5 cycles that stops the voltage ramping and maintaining the breakdown voltage. A series of determinations are done, which are then treated statistically to yield the final value.

Obtained data were statistically analyzed according to procedures outlined by Gomez and Gomez (1984) using MSTAT-C computer program. The differences among treatment means were compared by Least Significant Differences test (LSD)at 0.05 level of probability.

RESULTS AND DISCUSSION

Data presented in Table (1) and illustrated in Fig (2) summarize the average values of the breakdown voltage (KV) of mineral oil, vegetable oils, milk fat, animal fat and margarine, in addition to the moisture content and the acid values of these oils or fat as the breakdown voltage values is influenced by moisture and acid value of the oil or fat (Koch *et al.*, 2007).

Table (1): Dielectric breakdown voltage (KV) at 80°C, moisture % (w/w) and acid value (mg KOH/g) for mineral oil, vegetable oils, milk fats, animal fats and margarine oil.

Type of oil and fat	Moisture % (w/w)	Acid value (mg KOH/g)	Breakdown (KV)
Mineral oil:			
Extra super	0.68	1.44	53
Vegetable oils:			
Soybean	0.34	5.64	40
Sun flower	0.35	0.87	63
Olive	0.33	5.55	53
Palm	0.36	0.56	61
Palm kernel	0.37	0.40	44
Clarified Palm	0.37	0.43	40
Milk fats:			
Cow's Ghee	0.39	1.26	10
Buffalo's Ghee	0.69	1.57	8.5
Camel's Ghee	0.55	1.19	49
Animal fats:			
Lard (pork fat)	0.35	1.18	59
Cow	0.32	1.13	64
Camel (hump)	0.32	4	10
Sheep (fat tail)	0.32	0.42	44
Margarine oil:			
Ext. by Boiling	0.41	0.56	8
Ext. by Solvents	0.36	0.32	44.5

In general, there were considerable differences in breakdown voltage values of all samples. Animal cow's fat recorded the highest value (64 KV) while cow's and buffalo's milk fat in the form of ghee recorded the lowest values (10 & 8.5 KV, respectively). It was also clear that the vegetable oils were characterized by their higher values which were ranged between (40-61 KV). Vegetable oils breakdown voltage values are in agreement with those previously reported by Oommen (2002) and Blackburn *et al.* (2006).In regards to animal fat, cow recorded the highest value as previously mentioned, while lard and sheep recorded 59 and 44 KV respectively.

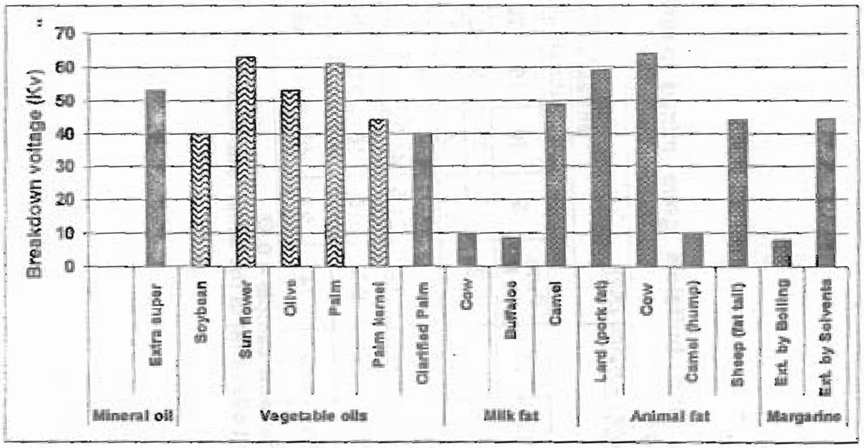

Fig (2): Comparison of dielectric breakdown voltage of mineral, vegetable oils, milk fats, animal fats and margarine oil.

Concerning camel values, it was very surprising as camel's milk fat recorded a very high value (49 KV), comparing with cow and buffalo's milk fat, while camel animal fat (hump) recorded a very low value almost similar to cow's milk fat (10 KV).On the other hand, margarine oil which was obtained by two ways recorded two different values. It recorded 8 KV similar to that of buffalo's milk fat when it was obtained by boiling and 44 KV when obtained by solvents extracting. This gap between the two values might be due to the presence of the emulsifying salts in the margarine oil prepared by boiling. These results could be considered as an indicator to the possibility to use this parameter to distinguish between any of these oils or fats. Furthermore, the probability to detect milk fat adulteration with other oils or fats is worthwhile to be investigated as a second part of this study.

Data in Table (2) show the dielectric breakdown voltage (KV) for pure vegetable oils or animal fats and the pure milk fats (Ghee). It was clear that the dielectric break down values were higher for vegetable oils or animal fats as compared with pure milk fats. These results were promising, therefore pure milk fats of either cows or buffaloes being mixed with different ratios ranged from 5 to 35% of vegetable oils or lard or margarine oil, and the dielectric breakdown voltage was determined to see whether it will be constant or will be changed according to the % of addition or adulteration. Data in the same Table (2) and illustrated in fig 3, 4, 5 and 6 summarize these changes.

Table (2): Dielectric breakdown voltage (Kv) for cow's and buffalo's ghee mixed in different ratios with some vegetable oils, lard and margarine oil at 80°C.

Type of oil & Fat	Breakdown voltage (Kv)																
	Pure Veg. oil or Animal fat	Cow's Ghee Adulteration %								Buffalo's Ghee Adulteration %							
		Pure Milk Fat	5	10	15	20	25	30	35	Pure Milk Fat	5	10	15	20	25	30	35
Sunflower	55	8	9	13	22	26	31.5	38..5	50	9.5	16	31	35	42.5	51	59	65
Palm	43	8	9	13	16.5	24	31.5	35	37.5	8	8.5	9	16	32	36	47	51
Palm kernel	37	8	10	15	25	31	45	56	58	8	9	9.5	15	18	22.5	23	30
Lard	41	8	11	20	27	33.5	39	50	55	12	21.5	44.5	43	50.5	50.5	55	59
Margarine (Boiling)	8	11	20	29.5	30. 5	40	49.5	50	52	9.5	15	16	21	25	26	30	44
Margarine (Solvents)	39	11.5	20.5	30	30.5	40	49.5	50	50	14	22	40	46	46	46.5	51	53

1- LSD value at 0.05 level between different adulteration ratio in the same sample = 0.58

2- LSD value at 0.05 level between samples adulterated by different oils or fat at the same ratio = 0.47

As shown Table (2) and figs. 3 and 4 when cow's or buffalo's ghee were mixed with sunflower, palm and palm kernel oils, the dielectric breakdown voltage values increased from 8 to 50 , 37.5 and 58 in cow's ghee with the three oils respectively. With buffaloes ghee the values increased from 9.5 to 65, 8.0 to 51 and from 8 to 30 with sunflower, palm and palm kernel oils respectively.

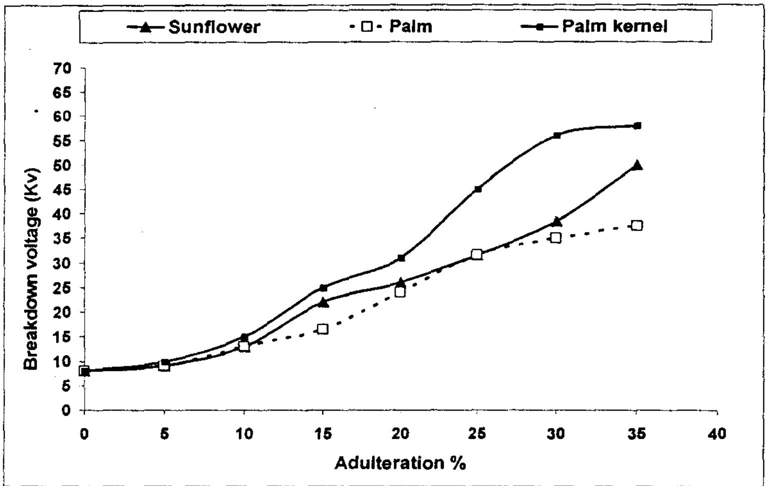

Fig (3): Breakdown voltage (KV) for cow's ghee adulterated by vegetable oils at different ratios.

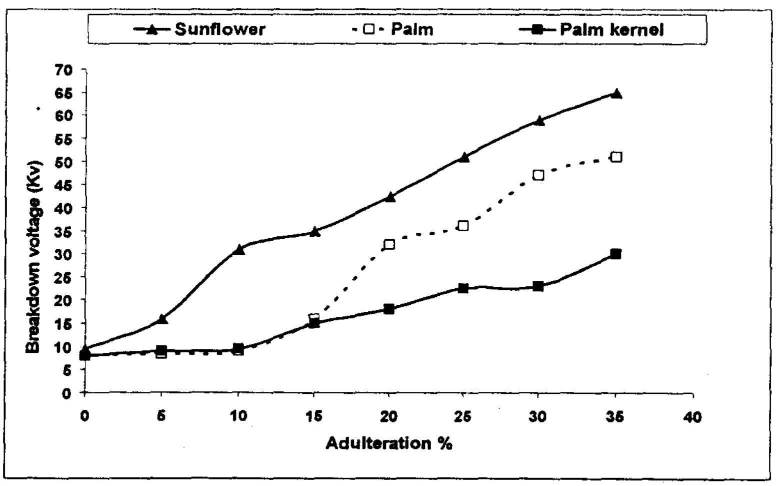

Fig (4): Breakdown voltage (KV) for buffalo's ghee adulterated by vegetable oils at different ratios.

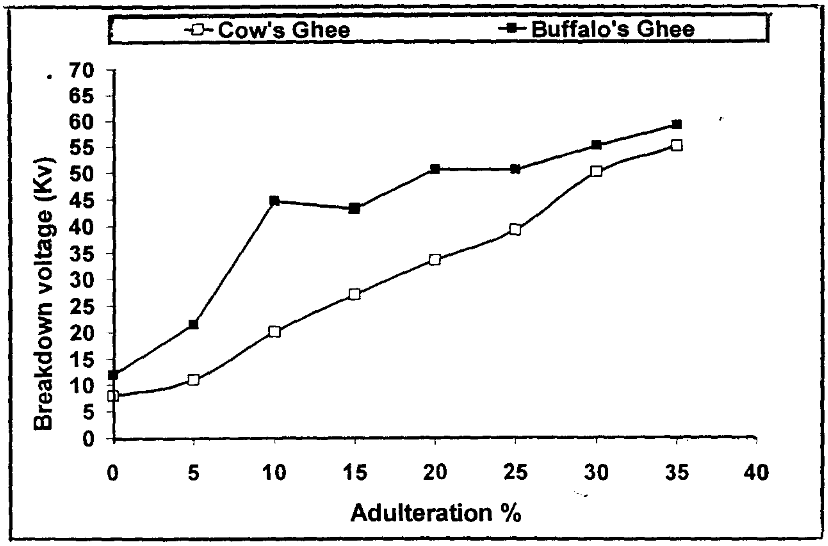

Fig (5): Breakdown voltage (KV) for cow's and buffalo's ghee adulterated by animal fat (lard) at different ratios.

This increment was significant from the first adulteration % as the LSD value was 0.58. Regarding the adulteration with animal fat (lard), the results in same Table and Fig (5) clearly indicate that cows or buffaloes milk fat dielectric breakdown values were positively and significantly affected by lard adulteration as the dielectric breakdown value for cows ghee increased from 8 and in direct relationship with the adulation % to be 55 with 35% addition. While buffalo's ghee appeared quick response as the dielectric breakdown value increased from 12 and reached 50.5 with 20% addition and reached 59 with 35% addition.

Concerning, the adulteration of milk fat with margarine which is commonly happened with butter, it is worthy to note that margarine oil recorded two different values, when margarine was boiled to get the oil by water evaporation, the margarine oil behaved in a manner like pure milk fat and recorded dielectric breakdown value equal to 8 which is similar to cows ghee. When margarine oil was extracted with diethyl ether, the dielectric breakdown value behaved in a manner like vegetable oil and the recorded value was 39. This variation was attributed as previously mentioned to the presence of emulsifying salts in the first state when cows or buffaloes ghee was adulterated with margarine oil obtained by boiling, the dielectric breakdown voltage was also increased significantly to end with 52 and 44 in case of cow's and buffalo's ghee respectively.

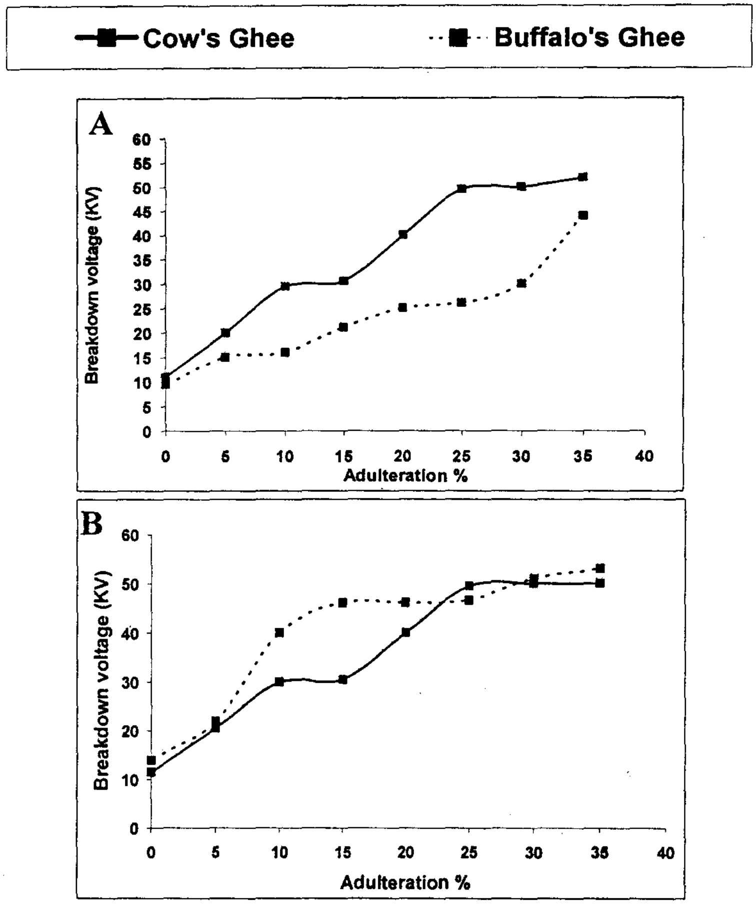

Fig (6): Breakdown voltage (KV) for cow's and buffalo's ghee adulterated by margarine extracted (A) by boiling or (B) by solvents at different ratios.

The same trend was also observed with margarine oil extracted by solvents as the dielectric breakdown voltage increased from 11.5 to be 50 with cow's ghee and increased from 14 to be 53 with buffalo's ghee.

These results clearly indicate that, it is possible to depend on the dielectric breakdown voltage test to detect milk fat adulteration either by vegetable oils or animal fats like lard and the author advise to extract oil with solvents when there is a doubt of margarine addition to avoid the effect of the emulsifying agent which might lead to a miss leading results.

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الكشف عن غش دهن اللبن باستخدام خواص العزل الكهربي

محمد عبد الغني ابراهيم الأعسر

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يمثل غش اللبن وخاصة دهن اللبن مشكلة كبيرة في الوقت الحاضر ونظرا لأرتفاع سعر دهن اللبن فإن الزبد والسمن غالبا ما يكونان هدف للغش بالدهون الأقل سعرا (الزيوت النباتية أو الدهون الحيوانية). ويعتبر تقدير الأحماض الدهنية بأجهزة التحليل الكروماتوجرافي والذي يتبعه بعض الحسابات من أكثر الطرق شيوعا لأكتشاف غش دهن اللبن بالزيوت أو الدهون الأخري. ولكن مع ادخال بعض المصادر الدهنية العديدة في تغذية الأبقار والجاموس أصبح الإعتماد علي هذه الطريقة لكشف غش دهن اللبن عملية أكثر صعوبة. ويستخدم في الوقت الحالي خواص العزل الكهربي (اختبار انهيار جهد العزل الكهربي) لتحديد فترة صلاحية الزيوت المعدنية المستخدمة في المحولات الكهربائية. وقد تم تقدير هذه الخاصية في بعض الزيوت النباتية وبعض الدهون الحيوانية ودهن اللبن وزيت المرجرين (Edible oils and fats) للوقوف علي معرفة مدي وجود فروق يمكن الإعتماد عليها للتمييز بينها. وقد تم أيضا تقدير هذه الخاصية في خليط من دهن اللبن وهذه الزيوت النباتية والدهون الحيوانية بنسب مختلفة. وقد أشارت النتائج المتحصل عليها الي امكانية الإعتماد علي هذه الخاصية كطريقة سريعة وسهلة للكشف عن غش دهن اللبن بالزيوت النباتية أو الدهون الحيوانية.