DETECTION OF MILK AND SOME DAIRY PRODUCTS ADULTERATION

By

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ABSTRACT

Adulteration may be unintentional or intentional to increase the margin of profit. Sometimes the regulations or the standard specifications of the country help those adulterators by indirect ways which lead to the appearance of adulterated and low nutritional value dairy products in the market protected by these regulations or standard specifications. In this study, a survey has been done on some available cheese especially white soft cheese and processed cheese in the local market of Egypt. Also it was interest to find which method can be used to detect milk fat adulteration with vegetable oils.

Fifty six samples of some white soft cheese (34 samples) and processed cheese (22 sample) were collected from the local market and were analyzed for chemical composition (fat, protein, total solids) and starch presence. The obtained data revealed that most samples recorded very low protein content and high fat percentage. Besides, presence of starch in one brand of white soft cheese and in most samples of processed cheese. Allowance of low protein percentage in white soft and processed cheeses, as well as the high fat percentage and the expense of protein, the use of starch to increase the total solids and also the use of hydrogenated vegetable oil and fats is an inevitable consequence of the loose clauses in the Egyptian standardization specification.

On the other hand samples of standard butters (prepared from milk obtained from the herd of faculty of agriculture Cairo university), butter collected from the local market, vegetable oil and margarine were subjected to different analytical to find out the best methods that could be used to detect milk fat adulteration. Melting profile appearance and Liberman-Bruchared for cholesterol detection gave primary results stating the probability of adulteration. While Reichert-Meissl number gave positive result for detecting adulteration but can't deny its presence. To determine adulteration presence or absence, determination of sterols content has done been along which the determination of fatty acids composition.

Therefore it could be recommended reviewing the Egyptian standard specifications and some clauses should be changed to achieve minimal nutritional and healthy value requested in the dairy products. Also when determining milk fat adulteration we recommend melting profile appearance as a primary step, then determination of Reichart-Meissl number and if there is a probability for adulteration the sterol and fatty acids content should be done.

Key words: adulteration, white soft cheese, processed cheese, sterols, fatty acids

CONTENTS

LIST OF TABLES

LIST OF FIGURES

SUMMARY

Milk, which has known as cheapest and best acceptable nutritional source with its protein, fat, carbohydrate and reasonable vitamins content, all these benefits make it targeted for adulterators who adulterate it with many ways starting with the water, urea and starch addition ending with substitution to some of its components

Adulteration, on its general term is "offering or submitting a product low in the concurrent standards or specifications"; and when looking to this term on the field of food adulteration - specially milk and its products - we found it expand to include any act depressing the product quality by remove its high value components or substitute it with low value one or turned the product to unsafe food by using unhealthy additives. With this concept the adulteration appears into two types it may be intentional and it is the common one which has evolved and growing with the development of the analytical methods. The other type is unintentional type as incident contamination which is usually due to negligence, ignorance or due to lack of good or proper facilities.

Sometimes the regulations or the standard specifications of the country help adulterators by indirect ways. It supposed to issue specifications for products to ensure that the consumer will get the promising nutritional value from each product. But, negligent the standard specification in some items lead to depressing the products quality by producers, subsequently lead to depressing the nutritional value of products with unacceptable way.

So, this study was designed to through light on:

1- Weakness clauses in Egyptian standard specification especially in cheese field specifically white soft cheese and processed cheese by a survey study on some types of these varieties available in local market.

2- Methods that can be used to detect the adulteration of milk fat with vegetable oils or fats, such as qualitative, chemical and chromatographic methods.

The study contained the following points:

Firstly: Survey study on the chemical composition of some white soft cheese and processed cheese varieties available in the local market

a. White soft cheese varieties

Thirty four (34) white soft cheese samples (17 brands) were collected from the local market. Two brands (4 samples) were produced using milk fat and the other thirty samples were produced using vegetable oils. All cheese samples were analyzed for chemical composition (fat%, protein%, total solids %) and starch presence.

The obtained results revealed that:

1. There were significant increases in fat percentage at the expense of protein as it recorded an average of 7.85% and 22.33% for protein and fat respectively in samples that produced with milk fat. While the percentage in samples produced with vegetable oils were in the average of 5.6% and 24.72% for protein and fat respectively. It is worth mentioning that the lowest percentage of protein recorded was 2.57% and the highest percentage of fat was 31.84%.

2. All samples were lower than the old standard specification limit (10%). These lower protein values are due to the loose specifications which contributed in the appearance of unnutritional and unhealthy cheeses in the local market.

b. Processed cheese varieties

Eight brands (16 samples) of spreadable processed cheese, three of them were manufactured using milk fat, and the other five brands using vegetable oil. Another three brands (6 samples) of block processed cheese manufactured using vegetable oil. All cheese samples were purchased from the local market and all of them were analyzed for chemical composition (fat%, protein%, total solids%) and starch presence.

The obtained results revealed that:

1. There were a big gap between the sum of fat% and protein and the total solids %. This trend was found in one sample from those produced with milk fat and in all samples that produced with vegetable oils. This gap is covered by the addition of starch which can be used as food stuff according to the standard specification (No. 1132/2013) which allowed addition of food stuff in the range of 15%.

2. Protein content in spreadable cheese made with milk fat was acceptable as it ranged from 8.85% to 9.9% while it ranged from 1.64% to 8.9% in cheese samples with vegetable oils.

3. The low protein content, the high vegetable oil content and also the presence of starch in these cheeses are due to the absence of protein content limit in the present specifications, and the allowance of using vegetable oils without limitations as well as the allowance of adding food stuff at a the ratio up to 15%.

Secondly: Detection of butter adulteration by vegetable oils

a. The qualitative methods

Two butter samples represent the standard butter for both buffalo's and cow's milk fat were prepared from raw milk obtained from the herd of the faculty of Agriculture, Cairo Univ., Giza, Egypt. Both milks were separated using Alfa-Laval separator and the obtained creams were churned into butter. Another seven butter samples were purchased from the local market.

1. Melting profile appearance

The obtained results revealed that this test could be used to detect the possibility of milk fat adulteration by melting the butter sample at 60ºC in a cylindrical tube. The natural butter appears with an off white color and yellowish color in the upper layer of buffalo's and cow's butter respectively while the lower layer was white in both as it represent the butter solids not fat. The melted upper layer and the lower layer have changed in the tested samples in volume, color and presence of watery layer according to the melted butter characteristics and the method of adulteration.

2. Liberman-Bruchard color reaction for cholesterol detection

This method is depending on the reaction of cholesterol with various strong acids to give green color in the presence of cholesterol and the color concentration decrease when the cholesterol content decrease. The obtained results revealed that there were fluctuations in the obtained colors therefore it couldn't be dependable test to detect adulteration and this point need more research.

b. The physical and chromatographic methods

Standard buffalo's milk fat sample (BO) was prepared from raw milk obtained from melting standard buffalo's butter at 60ºC to get milk fat free from solids. In addition to one sample of hydrogenated palm kernel oil (HPKO) and five prepared samples by mixing BO with HPKO at the ratios (10 to 50%). All samples were subjected to determination of Reichert-Meissl number, fatty acids composition and sterol fractions to illustrate the effect of mixing vegetable oil with butter oil on the properties of milk fat.

1. Reichert-Meissl number

The obtained results revealed that there was apparent reduction in the values of Reichert-Meissl number for Butter fat with the increase of mixing percentage with HPKO as it lowered from 29.22 to reach 16.56 in 50% mixing sample that means 43.33% decrease. These results revealed that this test can detect the probability of milk fat adulteration with vegetable oils but it can't ensure the absence of adulteration.

2. Sterols determination in BO, HPKO and their mixtures

The obtained results revealed that determining of sterols by HPLC device gives apparent result on presence of milk fat adulteration with vegetable oils as cholesterol content in BO was 289.9 mg/100g fat while it was 11.7 mg/100 g fat in HPKO. By mixing BO with different ratios of HPKO cholesterol content gradually decrease as it reach 147.85mg/100gm fat in 50% mix. On the other side stegmasterol and β-sitosterol content gradually increased from 0.03 and 0.12mg/100g fat in BO and reached to 7.53 and 28.91mg/100g fat in 50% mix.

3. Fatty acids composition

Determination of fatty acid composition by GC device revealed that there was a pronounced change in fatty acid composition either by increase or decrease. Lauric (C12:0) was found in a very high ratio in HPKO (41.91%) and in a low concentration in butter fat, therefor its percentage obviously increased in mixed samples. On the other hand Palmitic (C16:0) was found in BO in a high percentage than HPKO as it was 29.63 and 8.72 % respectively, therefor its percentage gradually decreased in mixed samples and …etc.

In general Total saturated fatty acid increased and monounsaturated and polyunsaturated fatty acids decreased. Adulteration of milk fat could be detected using GC analysis for doubtful samples at the same time with standard sample.

c. Detection of milk fat adulteration in samples from local market

To apply the previous findings in detecting milk fat adulteration, three doubtful milk fat samples were purchased from the local market and were separated and the resultant cream were churned to butter then the obtained butter samples were melted and analyzed for its fatty acids properties according to the following sequence: Reichert-Miessl number, Sterol determination and Fatty acids composition.

1. The first step is determination of Reichert-Miessl number

The obtained results illustrated that Reichert-Miessl numbers were reduced in the first and the third samples compared to standard milk fat, on the other hand the second sample gave a nearly close result to that of standard milk fat. The above results give a primary indication to the presence of adulteration in first and third samples.

2. The second step is to determine the sterols content

The obtained results revealed that there was a pronounced decrease in cholesterol content of the first and third samples compared with standard milk fat **[**as it was 219.78, 232 and 289.9 mg/100gm fat respectively] while stegmasterol and β-sitosterol content increase as it were 7.45 and 39.089 mg/100gm fat respectively in first sample and were 5.05 and 35.779 mg/100gm fat respectively in third sample compared with the standard milk fat. On the other hand second sample gave near results to that of standard milk fat for cholesterol, stegmasterol and β-sitosterol respectively. That indicates presence of adulteration with vegetable oils in samples one and three without determination of the vegetable oil type used.

3. The third step: determination of fatty acids composition

By comparing the fatty acids composition of those three samples with the fatty acids composition of common vegetable oils that mostly used in dairy products (palm oil, shortening, palm kernel oil and hydrogenated palm kernel oil) it would be clear that first and third samples show apparent increment in the fatty acid that characterize shortening Palmitic (C16:0), and decrease in the rest of fatty acids that absent from it as (C4:0), (C6:0), (C8:0) and (C10:0) or fatty acids that found in low content in it as (C12:0) and (C14:0).

Accordingly, one should carry out more than one test to prove whether there is adulteration or not. The melting profile appearance could use as a primary test. Followed by Reichert-Miessl number and finally determination of sterols and fatty acid composition.

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المستخلص العربي

الغش فى مجال الألبان ومنتجاتها قد يكون غشاً عارضاً وقد يكون أيضاً غشاً متعمداً بغرض زيادة هامش الربح. وأحياناً تساعد القوانين واللوائح فى البلاد على حدوث هذا الغش بطريقة غير مباشرة وبالتالى ظهور البان ومنتجات مغشوشة ومنخفضة القيمة الغذائية فى الأسواق والتى قد تحميها هذه اللوائح والمواصفات. ولذا تم فى هذه الدراسة عمل دراسة مسحية لبعض أنواع الجبن المتاحة فى السوق المحلى المصرى خاصة الجبن الأبيض الطرى والجبن المطبوخ وتم عمل دراسة حول الطرق التى يمكن استخدامها للكشف عن غش دهن اللبن بالزيوت النباتية بطرق وصفية وكيميائية وكروماتوجرافية .

تم تجميع عدد(٣٤ عينة) ممثلة لعدة أصناف من الجبن الأبيض الطرى (١٧ صنف) والجبن المطبوخ(٢٢ عينة) تمثل (١١صنف) وتم تحليل العينات لكل من الدهن والبروتين والجوامد الكلية والكشف عن وجود النشا. وأشارت النتائج المتحصل عليها إلى أن أغلب العينات سجلت نسب منخفضة جداً من البروتين ونسب مرتفعة بشكل ملحوظ من الدهن إلى جانب وجود نشا فى نوع واحد من الجبن الأبيض الطرى وأغلب أنواع الجبن المطبوخ. كما أن نسبة البروتين المنخفضة سواءً فى الجبن الأبيض الطرى أو الجبن المطبوخ ،ارتفاع % للدهن على حساب البروتين ،استخدام النشا لزيادة الجوامد الكلية وكذلك استخدام الزيوت النباتية المهدرجة لهو نتيجة حتمية للبنود غير المحدده فى المواصفات القياسية المصرية.

على الجانب الآخر تم تحضير عينات زبد قياسية من لبن ناتج من مزرعة كلية الزراعة جامعة القاهرة بالإضافة لتجميع عينات زبد متاحة فى السوق المحلى وكذلك عينات زيوت نباتية لمحاولة ايجاد أفضل الطرق التى من خلالها يمكن الكشف عن غش دهن اللبن بزيوت نباتية و اعطت عدة طرق وصفية مثل خواص الإنصهار واختبار ليبرمان للكشف عن الكوليسترول نتائج اولية تفيد احتمالية وجود الغش بينما أعطى اختبار رقم رايخرت مايسيل نتيجة ايجابية لوجود الغش ولكن لم يستطع نفيه وتم تحديد هذا الغش بواسطة تقدير محتوى الإستيرولات وتم تقدير محتوى الأحماض الدهنية لتحديد نوع الزيت المضاف.

ولذا توصى هذه الدراسة بمراجعة المواصفات الحالية وتغيير بعض بنودها للمحافظة على الحد الأدنى من القيمة الغذائية والصحية المطلوبه من المنتجات اللبنية. وعند الرغبة فى الكشف عن غش دهن اللبن بزيوت نباتية يجب الكشف عن خواص الإنصهار وتقدير رقم رايخرت مايسيل واذا وجدت مؤشرات مبدئية يتم التأكد بتقدير محتوى الإستيرولات ويليه تقدير محتوى الاحماض الدهنية عند الرغبة فى تحديد نوع الزيت المستخدم.

الكلمات الدالة: غش، الجبن الطرى، الجبن المطبوخ، الإستيرولات، الأحماض الدهنية