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Quality evaluation and determination of heavy metal contents in palm oil

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Abstract

Palm oil is an edible vegetable oil, extracted from the fruit of the *Elaeis guineensis* which is used for frying foods in most of the restaurants and confectionaries as well as for cosmetic preparations in industries. As the quality of palm oil in Bangladesh is deteriorating day by day, ten brands (S-1 to S-10) of commercially available palm oils were collected from different local markets, their physico-chemical properties were tested and compared with the standard parameters stated by Bangladesh Standards and Testing Institution (BSTI). Results revealed that the acid value, free fatty acid value and relative density of all the palm oils (S-1 to S-10) were within the range of BSTI standard. However, the saponification value, peroxide value, iodine value, insoluble impurities and moisture content were much higher than the ranges of BSTI standard in all the samples. Moreover, lead content was higher than the standard value (>0.1 ppm) in brands S-1, S-4 and S-10. In addition, Copper and Iron contents were higher than the BSTI standards (0.1 and 1.5 ppm) in all the tested samples, whereas Cadmium content was below than the standard level (1.0 ppm).

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Introduction

Palm oil is an edible vegetable oil, which is rich in saturated fats and free *trans*-fats (Mukherjee and Mitra, 2009; McNamara, 2010). The oil is an extract of the fruit of *Elaeis guineensis* of *Arecaceae* family (Pande *et al*., 2012). Palm oil was originated in West Africa and since the late 20th century the majority of palm oil is grown in Southeast Asia (Henderson and Osborne, 2000; Lynn, 2002). It was added to Bangladesh edible oil market in early '70s. Palm oil is used in cooking foods and also used as an important ingredient in many processed foods. In addition, palm oil is used in soaps, toothpaste, waxes, lubricants and ink (Pande *et al*, 2012; Ismail, 2005). Palm oil is deep red in color as it contains abundant beta-carotene, which protects against vitamin A deficiency and certain forms of cancer (Solomons and Orozco, 2003; Nagendran *et al*., 2000; Radhika *et al*., 2003). Studies suggest that vitamin A level increases in blood due to consumption of red palm oil by pregnant women (Radhika *et al*., 2003). Palm oil is reported to improve the memory status due to the presence of tocotrienols antioxidants (Sen and Khanna, 2010; Ong, 1992). The palm kernel oil is high in saturated fatty acids. The fatty acids contents are about 50% and 80% respectively and are esterified with glycerol (Mukherjee and Mitra, 2009). POME (Palm oil mill effluent) is a source of microalgae growth specially in wastewater from palm oil milling process. *Chlorella* UMACC 283, cultivated in 5%

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POME, produces high content of lipid and biomass having good potential for biodiesel (Idris *et al*., 2018; Lik *et al*., 2018). Now-a-days, palm oil is increasingly being used in the foods, cosmetic, and agrochemical industries in Bangladesh. However, these oils are often adulterated and counterfeited with inexpensive poor quality oils having serious economic implications for the cosmetic and food industries, and potential health impacts to consumers. Substandard quality or improperly refined palm oils are entering in the market causing different types of complicated diseases to a large number of people. To ensure the quality of marketed palm oil, 10 different commercially available palm oils from the local market were collected and different physicochemical tests were carried out based on the parameters and guidelines stated by Bangladesh Standards and Testing Institution (BSTI). The main intension of this research work was to assess the quality of palm oils and identify the presence of adulterants as well as determination of the amount of heavy metals in locally marketed palm oils in Bangladesh.

Materials and methods

Reagents and instruments

Potassium hydroxide (KOH) was purchased from Merck Specialities (Pvt) Ltd, Mumbai. Hydrochloric acid (HCl), glacial acetic acid, potassium iodide (KI), sodium thiosulfate, phenolphthalein, starch, petroleum ether, diethyl ether were bought from Merck KGaA, Germany. Ethanol and chloroform were purchased from Sigma-Aldrich, Co., Germany. Hanus solution was prepared in the laboratory using iodine. Moisture analyzer was purchased from Mettler Toledo, USA. Instruments like reserved condenser and dryer were taken from Pharmaceutical Technology Laboratory, University of Dhaka. Atomic Absorption Spectrophotometry was performed in Bangladesh Council of Scientific and Industrial Research (BCSIR), Dhaka.

Sample collection

Ten (10) different commercially available palm oil were collected from different local markets of Bangladesh. Collected products/samples were labeled as S-1, S-2, S-3, S-4, S-5, S-6, S-7, S-8, S-9 and S-10. All collected products were preserved in a dark and dried place at room temperature.

Determination of acid value

The acid values of the samples were determined by using titrimetric method of AOAC (Association of Official Analytical Chemists). For the determination of acid value, 10±0.5 gm of oil was taken in a 250 ml conical flask and 50 ml of titration solvent (prepared by mixing diethyl ether and ethanol in a ratio of 1:1) was added to it. The solution was titrated with 0.1 N alcoholic KOH solutions until a pink color was obtained which persisted for 15 seconds. The number of ml of KOH added was recorded. Acid value was calculated as mg of KOH per gm of oil (Ekwu and Nwagu, 2004).

$$\text{Acid value} = 5.611 \times V \frac{f}{w}$$

where, V= volume of 0.1N KOH solution in ethanol (ml), f= Factor of 0.1N KOH solution, w= weight of oil sample in g, Content of potassium iodide in 1ml of 0.1N KOH solution= 5.611

Determination of free fatty acid value

10±0.1 g of oil was measured and taken in a 250 ml conical flask. 50-100 ml of ethanol was also taken into the flak which had been neutralized after addition of 1ml of phenolphthalein. Then it was titrated with 0.1N KOH solution with vigorous shaking until a pink color was appeared (Ekwu and Nwagu, 2004).

$$\text{Free fatty acid, in term of palmitic acid} = \frac{2.56 \times T \times F}{M}$$

where, T= titre consumed (ml), F= Factor of titre, M= mass of sample (g)

Determination of saponification value

1.0 g of oil sample was measured and taken in a conical flask. 25 ml 0.5 N alcoholic KOH was also taken, and the mixture was heated under a reserved condenser for 30-40 minutes until fully dissolved. After cooling, phenolphthalein was added and titrated with 0.2 N HCl until the pink color end point was achieved. A blank titration was performed under the equal settings without oil (William and Vida, 2015).

$$\text{Saponification value} = \frac{(B\text{-}T) \times N \times 56.1}{w}$$

where, B= ml of HCl required by blank, T= ml of HCl required by oil sample, N= normality of HCl, W= weight of oil in g

Determination of peroxide value

5±0.05 g of sample was measured and taken into a 250 ml glass stoppered Erlenmeyer flask. 30 ml of the acetic acid-chloroform solution (acetic acid and chloroform in the ratio of 3:2) was also taken to the sample and the flask was swirled until completely dissolved (careful heating/warming on a hot plate may be necessary). Then 0.5 ml of saturated potassium iodide solution was added using 1 ml mohr pipette. The flask was stoppered and was swirled for exactly 1 minute. After that 30 ml of distilled water was immediately added and the flask was stoppered and shaken vigorously to liberate the iodine from the chloroform layer. Then titration was done with 0.1 N sodium thiosulfate. When the starting color of the solution was deep red orange, it was titrated slowly with 0.1 N sodium thiosulfate until the color lightened. Since the solution was initially a light amber color, 1ml of 1% starch solution was added as indicator by a dispensing device. The titration was carried out until the blue gray color disappears from the aqueous layer (upper layer). The volume of titrant was recorded. The blank titration was done in the same procedure without oil (Crowe and White, 2001).

$$\text{Peroxide value} = \frac{(S-B) \times N \times 1000}{W}$$

where, S = titration of sample, B= titration of blank, N= normality of sodium thiosulfate, W= weight of the sample

Determination of iodine value

About 0.25 gm of the sample was measured and taken in a conical flask. It was dissolved in 10 ml chloroform. 30 ml of Hanus solution was added and the mixture was allowed to stand for 45 minutes in dark place with occasional shaking. Then 10 ml of 10% KI and 100 ml distilled water were added and washed if any free iodine was present on the stopper. Then it was titrated with sodium thiosulfate until the yellow color turned almost colorless. Few drops of the starch indicator were added and titration was continued until the blue color totally disappeared. The volume of sodium thiosulfate was noted. Titration for blank was done (Firestone, 1994).

$$\text{Iodine value} = \frac{(B-A) \times N \times 0.127 \times 100}{W}$$

B= ml of 0.1 N sodium thiosulfate required by a blank, A= ml of 0.1 N sodium thiosulfate required by the sample, N= normality of sodium thiosulfate, W= weight of the sample

Determination of insoluble impurities

2.0 g of oil sample was measured and taken in a 250 ml conical flask. 20 ml of solvent mixture (petroleum ether: diethyl ether=1:1) was also taken into the flask. The flask was shaken vigorously, allowed to stand for 30 minutes at 30° C. The liquid was then filtered through dried and previously weighed Whatman number 1 filter paper. The filter paper was thoroughly cleaned with 10 ml of the solvent. The filter paper was then dried in an oven at 103 °C to obtain a constant weight. The increase in the weight represents the weight of the impurities and was expressed as the percentage of the initial weight as the following (William and Vida, 2015).

$$\% \text{ insoluble impurity} = \frac{a}{w} \times 100\%$$

where, a= increase in the weight of filter paper, w= weight of the sample

Determination of moisture content

Moisture and volatile content of the oil were determined by using moisture analyzer. About 2 g of oil was taken on the aluminum pan of the moisture analyzer. Then the analyzer was set at a time of 5 minutes and the temperature at 110 °C. Then the analyzer was operated and reading was recorded. It automatically provides the reading of the moisture in percentage of the mass of the oil sample (Bittelli *et al*., 2003).

Determination of relative density

For the determination of relative density, the instrument used was called pycnometer. Its volume was 50 ml. First the weight of it was determined. Then it was filled with the oil samples and weighed using the electric balance. The difference of the two weights gives the weight of 50 ml of oil sample. In the similar method, weight of 50 ml water was determined. During the experiment, the room temperature was about 30 °C. Each measurement was carried out for at least three times. Then the average was determined. Then the relative density of the oils was calculated by following equation (Chang, 1988):

$$\text{Relative density} = \frac{\text{Mass of the 50 ml oil}}{\text{Mass of the 50 ml water}}$$

Microwave digestion

0.3 ml of sample was weighed and placed in a TFM vessel. The TFM vessel was introduced inside fume hood. Then 7 ml of 65% HNO_3 and 1 ml of 20% H_2O_2 were taken with the

sample. If the sample stays on the wall of the vessel, acid should be added drop by drop to wet it. Then the vessel was swirled gently to homogenize the sample with the acid. Then the vessel was closed tightly and inserted into the microwave cavity and was adjusted with the temperature sensor. Then the microwave program was started. The vessel was cooled to room temperature. Then the sample was transferred to marked flask. Then the sample was diluted with deionized water to 50 ml (Fernández-Martínez *et al.* 2015).

Determination of metals

The contents of Pb, Cd, Cu and Fe were determined by atomic absorption spectrophotometer (AAS) model AA-7000. The detection level of this instrument is 0.1 ppm. The samples were aspirated through the nebulizer and the reading was taken. For each case, the reading for blank was taken.

Results and discussion

The quality of palm oils was analyzed by evaluating physicochemical properties such as acid values, saponification values, peroxide values, free fatty acids, insoluble impurities, iodine values, relative density, moisture content and heavy metal contents. The results were presented along with the BSTI standard values. To determine the quality of different samples of oil in the local market, these properties are very important parameters.

Acid value

Acid value of oils indicates the amount of free fatty acids present in the oil which are normally formed due to hydrolysis or oxidation of oil by atmospheric moisture (Zheljazkov and Nielsen, 1996). The acid value of the vegetable oil should be less than 1 for a base, KOH catalyzed trans-esterification process. Fats and oils with higher acid value can cause gastric acidity, gastric discomfort, and mal-digestion. In addition, these will lose the consumer's acceptability due to presence of odd color and odor caused by rancidity. The acid values should remain below the standard value so that it can be used safely. According to the Table II, the highest acid value found was 0.33 (as KOH), mg/g in S-1 and the lowest was 0.142 (as KOH), mg/g for S-3 where the BSTI standard requirement for acid value is ≤ 0.5 (as KOH) (Table I), mg/gm. So, all the samples met BSTI standard.

Free fatty acid value

Free fatty acids are the fatty acids that are produced from triglycerides by hydrolytic reactions (Zheljazkov and Nielsen, 1996). The major free fatty acids in palm oil are myristic, palmitic, stearic, oleic and linoleic acids (Tan *et*

Table I. Different standard parameters and their requirements stated by BSTI (BDS 1770:2014) for palm oil

Parameters	BSTI standard
Acid value (as KOH), mg/g, max	0.5
Free fatty acid (as palmitic), percent by mass, max	0.25
Saponification value (as KOH), mg/gm	190-209
Peroxide value (as milliequivalents of oxygen per kg), max	8.0
Iodine value (as KOH), min	53
Insoluble impurities (percent by mass)	0.05
Moisture content (percent by mass)	0.1
Relative density	0.910-0.915
Lead (Pb) content, max in ppm	0.1
Cadmium (Cd) content, max in ppm	1.0
Copper (Cu) content, max in ppm	0.1
Iron (Fe) content, max in ppm	1.5

Table II. Acid value of different palm oil brands

Code name	Acid value (as KOH), mg/g	BSTI standard (as KOH), mg/g
S-1	0.33	
S-2	0.24	
S-3	0.142	
S-4	0.233	
S-5	0.283	≤ 0.5
S-6	0.233	
S-7	0.245	
S-8	0.294	
S-9	0.263	
S-10	0.272	

Table III. Free fatty acid value (as palmitic acid) of different palm oil brands

Code name	Free fatty acid (% by mass)	BSTI standard (% by mass)
S-1	0.0151	
S-2	0.011	
S-3	0.0065	
S-4	0.011	
S-5	0.013	≤ 0.25
S-6	0.011	
S-7	0.0112	
S-8	0.0134	
S-9	0.012	
S-10	0.0124	

Table IV. Saponification value of different palm oil brands

Code name	saponification value mg/g	BSTI standard mg/g
S-1	248.44	
S-2	242.60	
S-3	275.66	
S-4	240.76	
S-5	256.46	190-209
S-6	272.56	
S-7	220.05	
S-8	254.77	
S-9	286.00	
S-10	237.90	

Table V. Peroxide value of different palm oil brands

Code name	Peroxide value (mEq of O_2/kg)	BSTI standard (mEq of O_2/kg)
S-1	28.036	
S-2	9.681	
S-3	13.790	
S-4	23.680	
S-5	19.692	≤ 8.0
S-6	11.811	
S-7	20.592	
S-8	16.452	
S-9	18.321	
S-10	12.360	

al., 1997). In our research, the highest free fatty acid value was found 0.0151% by mass in S-1 (Table III) and the lowest one was 0.011% by mass in S-2, S-4 and S-6 (BSTI standard value ≤ 0.25% by mass). Here, all the samples met the BSTI standards. Moreover, the oil can be used as biodiesel in industries (Tan *et al*. 2009).

Saponification value

Saponification value is used in checking adulteration. Results from our study are summarized in Table IV. Saponification values of all collected samples (S1-S10) were found to be higher (220.05-286.00) than that of the BSTI standard specified ranges 190-209 (as KOH), mg/gm. The larger the saponification value/number, the better the soap making ability of that oil (Nielsen, 1994). Higher saponification value for triglyceride indicates higher medium chain fatty acids (Seneviratne and Dissanayake, 2011).

Peroxide value

Peroxide value is a common measurement of oxidation (Gray, 1978). Peroxide value, concentration of peroxide in an fat or oil, is useful for evaluating the extent to which spoilage has advanced during storage. It was found that peroxide values of all the brands (S-1 to S-10) were higher than that of the BSTI standard value, ≤ 8.0 mEq of O_2/kg (Table V). The higher peroxide value indicates the rancidity of oils due to relative higher oxidation in oils and also the presence of high level of free radicals at the same time. Actually, peroxide value may be increased due to auto-oxidation. Free radical reactions are chain reactions and are very detrimental for living beings. As radicals are highly reactive, they might be capable of damaging major cells of the body randomly which might be responsible for the development of chronic disease like cancer, atherosclerosis, and emphysema (Glavind *et al.,* 1952).

Iodine value

Iodine value (or iodine number or iodine index or iodine absorption value) of an oil or fat is the mass of iodine consumed by 100 g of the oil or fat. It is used for the determination of unsaturation in fatty acids. Higher iodine value indicates higher unsaturation (presence of double bonds). Saturated oil is good for making soap. Higher the degree of unsaturation more liquid the oil will be at room temperature. Increased degree of unsaturation also decreases stability. Table VI showed that the highest and lowest iodine values were in S-1 (51.99) and S-6 (27.71) respectively. However, all the values are lower than the BST accepted value, ≥ 53.0. So, the degree of unsaturation in the oils were lower.

Insoluble impurities

Poorly soluble impurities of oils or fats are those materials which remain insoluble and can be filtered off when the fat or oil is dissolved in petroleum ether or diethyl ether or any other suitable organic solvent. It is the measure of the presence of resins, minerals, dirt, and oxidized fatty acids, alkaline soaps of stearic and palmitic acids, and proteins that are suspended in the oil. The percentages of insoluble impurities in all the samples were found very higher than the acceptable value according to BSTI standard (Table VII). The causes of higher amount of impurities are lack of proper refining, low standard of packaging and storage condition.

Moisture content

Most of the stored products are hygroscopic and are influenced by the atmospheric relative humidity. Moisture content affects processability, shelf life and quality. So, accurate determination is important in quality ensuring of foods, pharmaceuticals and chemicals. Due to the higher value of moisture content, the product's shelf life is reduced by the hydrolysis and microbial activity. Table VIII shows that moisture contents in all the samples exceeded the BSTI standard value (0.1% by mass). The moisture content of oil is important as it determines the stability of the oil. It depends on the storage condition, packaging and refining process etc. Moisture content of a sample of material depends on its hygroscopic nature. Hygroscopic action is the amount of moisture/water a material will absorb relative to ambient temperature and humidity conditions. Temperature and humidity conditions of butter oil and ghee should be controlled. From the results, it can be assumed that the moisture contents of all the samples did not meet the BSTI standard because it was not sold or stored under appropriate conditions.

Relative density

As relative density is more or less constant value for any substance, it is one of the important quality parameters. Oils with the lower density are highly appreciable to consumers. Convenient use of the oil depends on its density. Change in the density can be due to impurity, change in the composition (during storing or mixing of adulterants), rancidity or improper refining. So, significant

Table VI. Iodine value of different palm oil brands

Code name	Iodine value	BSTI standard
S-1	51.99	
S-2	49.804	
S-3	46.055	
S-4	32.875	
S-5	28.084	≥ 53.0
S-6	27.71	
S-7	30.72	
S-8	29.95	
S-9	44.88	
S-10	42.92	

Table VII. Insoluble impurities in different palm oil brands

Code Name	Insoluble impurities (% by mass)	BSTI standard (% by mass)
S-1	3.55	
S-2	5.00	
S-3	2.007	
S-4	3.24	
S-5	5.18	0.05
S-6	4.35	
S-7	4.26	
S-8	4.02	
S-9	3.05	
S-10	2.94	

Table VIII. Moisture content of different palm oil brands

Code name	Moisture content (% by mass)	BSTI Standard (% by mass)
S-1	0.63	
S-2	0.76	
S-3	0.62	
S-4	0.51	
S-5	1.00	0.1
S-6	0.84	
S-7	0.88	
S-8	0.59	
S-9	0.66	
S-10	0.72	

Table IX. Relative density of different palm oil brands

Code name	Relative density	BSTI ntandard
S-1	0.9095	
S-2	0.9099	
S-3	0.9087	
S-4	0.9036	
S-5	0.9109	0.910-0.915
S-6	0.9093	
S-7	0.91055	
S-8	0.9116	
S-9	0.9121	
S-10	0.9095	

change in the density will question the quality of the oil but slight changes are common, and these may be allowed. Table IX shows that at room temperature of 25°C, the highest and the lowest values of the relative densities were 0.9121 and 0.9036 represented by S-9 and S-4 respectively. All the values found for the samples were almost similar to that of the BSTI standard (0.910-0.915).

Heavy metal contents

Table X shows that the detected level of all the other metals (Pb, Cu and Fe) were far above than that of the BSTI standard except Cd. Presence of these metals in excess is hazardous for human health. The metal contents may be of natural origin or due to contamination during different steps of processing, poor purification, packaging and storage condition. Many reports described the deleterious effects of trace metals on the flavor and oxidative stability of oils as some metals catalyze oxidation of fatty acid causing the decrease of shelf life and nutritional values (Gonzálvez *et al*., 2010).

Table X. Heavy metal content of different palm oil brands

Code name	Pb content (ppm)	BSTI standard (ppm)	Cd content (ppm)	BSTI standard (ppm)	Cu content (ppm)	BSTI standard (ppm)	Fe content (ppm)	BSTI standard (ppm)
S-1	4.917		BDL		0.850		8.567	
S-2	BDL		BDL		0.967		10.633	
S-3	BDL		BDL		14.95		58.983	
S-4	7.883		BDL		4.5		19.883	
S-5	BDL	0.1	BDL	1.0	11.567	0.1	44.583	1.5
S-6	BDL		BDL		2.25		7.883	
S-7	BDL		BDL		4.183		37.717	
S-8	BDL		BDL		6.433		36.683	
S-9	BDL		BDL		2.417		20.233	
S-10	1.933		BDL		0.817		1.033	

*BDL= below the limit

Conclusion

Study was conducted to evaluate the quality of palm oils in the Bangladeshi markets. In this study, different brands were collected from different shops of different markets located in various areas. BSTI (Bangladesh Standard and Testing Institution) is the government authority to evaluate the quality of all types of foods and food products. It has set the quality parameters and their acceptable values for goods. Therefore, the marketed brands should need to fulfill the BSTI requirements. From the study, it was noticed that almost all the samples tested in current project deviated from BSTI standard requirements. Besides, the amounts of Pb, Cu and Fe were present in all the samples above the standard limits of BSTI. So, the study showed that the quality of palm oil is not up to the marks. Therefore, more concern and vigilance is needed by the authority to ensure the proper quality of marketed palm oil in Banladesh.

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