Short note

Expired bakery products as a promising alternative source for biodiesel production

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Expanding the use of biofuels has led to attract attention to focus on the alternative sources of fatty systems. In the current investigation, the oil extracted from expired croissant samples has been investigated as a potential source for biodiesel production. Fatty substrates have been extracted from expired croissant samples. The physic-chemical properties of extracted oil were determined. The fatty acid composition of extracted oil was performed, then the calculated oxidation stability value (COX) of the extracted oil was calculated. Produced biodiesel was evaluated for density, moisture content, acid value, peroxide value, iodine index and oxidation stability 110°C. The oil yield from croissant samples was 27.8%, wt./wt. The predominant fatty acids in the extracted oil were oleic (34.50%), palmitic (27.02%), linoleic acid (25.42%), stearic (8.90%) and linolenic-acid (2.43%). Biodiesel yield (%) of the obtained biodiesel was 86.45%. The density, moisture content, acid value, iodine index and oxidation stability of obtained biodiesel complied with specifications established by ASTM D6751and EN 14214 standards for this type of fuel.

Keywords: Biodiesel, bakery products, croissant, extracted oil, Transesterification, COX value.

1. INTRODUCTION

Recently, energy demand has been increased due to modernisation and industrial growth. It is also expected that the global energy demand will increase by 49% in 2030 [1]. On the other hand, petroleum liquid reserves are depleting at an alarming rate [2] and these reservations could be drained within the coming 45 years [3].

Biodiesel is becoming increasingly important as a promising alternative fuel due to its renewability, biodegradability, nontoxicity, and environment-friendly nature [4]. Biodiesel is one of the sustainable energy sources derived from different sources such as vegetable oils, animal fat, and algae oil [5].

Shortenings, margarine, butter, and vegetable fats are the major sources of fat in confectionery and bakery products [6]. The amounts of fat in some bakery products from different countries varied from 9.4 to 31% [7]. These findings imply that some of bakery products are potential and significant sources of lipids. It was found that, household food waste has increased more than twice in the last five years [8]. Recently, it is estimated that 1.2-2 billion tons (approximately one-third of the food produced in the world for human consumption) is wasted [9].

Croissants are high fat bakery products, with flaky structure formed by repeated rolling and folding of butter or margarine in laminated doughs [10]. A croissant contains approximately 30-40% fat by weight [11]. Puff pastry is a popular confectionery product with a unique texture. Puff pastry is processed from a dough consisting of enriched with fat and other ingredients and the

finished puff product may contain about 30% fat on a dry matter basis [12], and thus, it is considered a high-fat food. Danish pastries are also produced from laminated dough with high levels of fats [13]. The Recent studies focus on low-cost alternatives, such as inedible resources as well as animal fat waste [14]. Thus, the use of oil waste and animal fats is increasing for industrial-scale biodiesel production, this makes the production process more sustainable [15]. Therefore, the oil extracted from expired croissant samples has been investigated in this investigation as a potential source for biodiesel production.

2. MATERIAL AND METHODS

2.1. COLLECTION OF EXPIRED CROISSANT SAMPLES

In total, 500 croissant-type) 85 g) samples were collected from a private factory in Buraidah, Qassim, Saudi Arabia. Samples were collected in 2020.

2.2. LIPID EXTRACTION OF EXPIRED CROISSANT SAMPLES

The croissant samples were cut into a slice having an even thickness of approximately 1.5 cm. Croissant slices were dried in an electric oven (Model: Leicester, LE67 5FT, England) at 60°C for 24 h. The dried croissant samples were ground in an electric grinder (Braun Model 1021) and passed through a 0.6 mm sieve. Croissant powder was extracted for 18 h. with n-hexane in a Soxhlet apparatus [16]. Solvent was removed at 50°C under reduced pressure using a rotary evaporator. The extracted oil was stirred with the anhydrous sodium sulphate for 5 min, and then filtered (vacuum filtration through a Whatman No 1 paper (Whatman International Ltd, Maidstone, UK). The obtained oil was packed in stainless steel vessels.

2.3. DETERMINATION OF OIL YIELD

The oil yield of expired croissant samples was calculated using the following equation:

$$\text{Oil yield (\%)} = W_{EO} / W_{EC} \times 100$$

Where:

W_{EO} = the weight of extracted oil (g),

W_{EC}= the weight of expired croissant samples (g).

2.4. DETERMINATION OF PHYSIC-CHEMICAL PROPERTIES OF EXTRACTED OIL

Extracted oil was analysed for water content (expressed as g/100g), AOAC n. 930.15 [17], acid value (expressed as mg KOH/g oil.), AOAC n. 940.28 [18], peroxide value (expressed as meq O2/kg oil) AOAC n. 965.33 [18], Saponification value (mg KOH/g oil), (AOAC n.920.160 [18] and Iodine value (g I_2/100 g oil) AOAC n. 993.20 [18]. The density (gr/ml) of oil was measured by a mass over volume measurement at 40°C [19]. Brookfield viscometer equipped with a thermo-container (Brookfield Engineering Laboratories, Inc.; Middleboro MA, USA) was used to determine the viscosity of the extracted oil samples at 45°C, according to the method described by Ismail et al., [20].

2.5. FATTY ACID PROFILE DETERMINATION

The fatty acid composition of extracted oil was carried out using gas chromatography (HP 6890), following the previous procedures described by Ali and El-Anany [16].

Oxidation value (COX).

The calculated oxidation stability value (COX) of extracted oil was calculated by applying the formula proposed by Fatemi and Hammond [21] as follow:

$$COX = \frac{[1\,(16{:}1\% + 18{:}1\% + 20{:}1\% + 22{:}1\%) + 10.3\,(18{:}2\%) + 21.6\,(18{:}3\%)]}{100}$$

2.6. BIODIESEL PRODUCTION

One thousand grams of extracted oil were placed into a 2000 mL round-bottom flask equipped with a condenser. After the oil was heated to 65°C, the solution of sodium hydroxide (5.0 g) in methanol (144.82 ml, 6:1 molar ratio of methanol to oil) was slowly added into the reaction. The reaction temperature of the transesterification treatment was set at 60°C. The reaction continued for 110 min. At the end of the incubation stage, the mixture was transferred into a reparatory funnel, left for 24 h to separate the glycerine and impurities from the biodiesel. The produced biodiesel was washed with deionised hot water (50°C) several times to remove the catalyst, glycerol, and impurities [22].

2.7. BIODIESEL YIELD

Biodiesel yield was calculated using the following equation [23]

$$\%\ \text{Yield} = \frac{\text{grams of biodiesel}}{\text{grams of oil}} \times 100$$

2.8. QUALITY PARAMETERS FOR BIODIESEL

The produced biodiesel was evaluated for density at 40°C (ASTM D 6751), moisture content, acid value, peroxide value, Iodine index and oxidation stability 110°C based on the previous procedures described by Ali and El-Anany, [24].

3. RESULTS AND DISCUSSION

3.1 PHYSICOCHEMICAL PROPERTIES OF EXTRACTED OIL

Table I shows the physicochemical properties of extracted oil. No water content of extracted oil was detected. The oil yield (%, wt/wt) from croissant samples was 27.8%. Recovery of oil is dependent on the

extraction technique, moisture content, type of solvent and extraction period [25]. The acid value of raw materials used for biodiesel production has an influential and effective role in the percentage of fatty acid methyl esters of the final product [26]. The acid value measures the content free fatty acids formed after the hydrolytic degradation of lipid molecules [20]. The acid value of extracted oil under investigation was 1.96 mg KOH/g oil (Tab. I). This result suggests that the triacylglycerol molecules were subjected to hydrolytic degradation process. Free fatty acids content significantly increased with the increase in the storage duration in all the food products [27]. Peroxide value of the fat extracted from croissant samples was 7.03 ± 0.07 meq peroxide/kg (Tab. I). Codex specification suggests that the peroxide value limit might reach 10 meq/kg when the products were at the end of their shelf life [28]. Saponification value measures the mean of the molecular weight of the glycerides expressed in milligrams of potassium hydroxide (mg KOH/g oil). Saponification value of the extracted oil was 196.0±4.7 mg KOH/g. This finding indicates that the saponification value of extracted oil was within the recommended values of 180-199 mg KOH/g oil [29]. The iodine value measures the degree of fatty acid unsaturation in oil components. Iodine values of oil extracted from croissant samples was 65.30±2.08 g I_2/100 g oil, this finding indicates that the obtained oil is considered non-drying oil.

The density is a physical measure of adulteration of vegetable oils. Each oil has a specified density. Density of vegetable affected by fatty acid composition, minor components as well as temperature degree [30]. Density is an important factor which influences the design of unit operations such as distillation, heat exchangers, tubes, and reactors [31]. The density of oil extracted from croissant samples was 0.889 g/cm3 at 40°C. The absolute viscosity is one of the most important physical parameters of fatty systems. viscosity measures the resistance of oil to flow. It is necessary for pumping process, flow measurement, and heat transfer unit operations [32]. The viscosity value of oil extracted from croissant samples was 54.6 centipoises at 40°C.

Table I - Physicochemical properties of extracted oil

Parameters	Units	Value
Water content	g /100g	ND
Acid value	mg KOH/g Oil	1.96
peroxide value	meq/kg	7.03±0.07
Saponification value	mg KOH/g oil	196.0±4.7
Iodine value	(g I_2/100 g oil	65.30±2.08
Density at 40°C	g/cm3	0.889
Viscosity at 40°C	centipoises	54.6
Oil yield on dry weight basis	(%, w/w)	27.8

Table II - Fatty acid compositions of oil extracted from croissant samples

Fatty acid	CC:DB*	Extracted oil sample
Butyric	C 4:0	ND
Caproic	C 6:0	ND
Caprylic	C 8:0	ND
Capric	C 10:0	ND
Lauric	C 12:0	0.24
Myristic	C 14:0	0.60
Myristoleic	C 14:1	ND
Palmitic	C 16:0	27.02
Stearic	C 18:0	8.90
Oleic	C 18:1	34.50
Linoleic	C 18:2	25.42
Linolenic	C 18:3	2.43
Arachidic	C 20:0	0.89
Eicosenoic	C 20:1	ND
SFA		37.65
MUFA		34.50
PUFA		27.85
Calculated oxidation stability value (COX)		3.5

* Carbon content (CC) per double bonds (DB)
SFA, refers to Saturated Fatty Acids; MUFA, Monounsaturated Fatty Acids; PUFA, Polyunsaturated Fatty Acids.

3.2 FATTY ACID COMPOSITIONS OF OIL EXTRACTED FROM CROISSANT SAMPLES

Fatty acid composition of extracted oil is shown in Table II.

The oil extracted from croissant samples mainly composed of 34.50 wt.% oleic acid (18:1), 27.02 wt.% palmitic acid, 25.42 wt.% linoleic (C18:2), 8.90 wt.% stearic acid (18:0), 2.43 wt.% linolenic-ω3 acid (18:3), 0.89 wt.% arachidic acids (C20:0), 0.60 wt.% myristic acid (C14:0) and 0.24 wt.% lauric acid (C12:0). These findings indicate that the oil extracted contains 37.65, 34.50 and 27.85% saturated fatty acids (SFAs), monounsaturated fatty acids (MUFAs) and polyunsaturated fatty acids (PUFAs), respectively. These findings are in good agreement with those reported by Anwar et al., [33]. The viscosity of a fatty acid ester usually increases as chain length and saturation increases. On other side, cis double bonds cause a remarkable reduction of viscosity as esters [34]. In the current investigation, the calculated oxidisability COX value of oil extracted from croissant samples was 3.5. COX value is a useful element usually used for evaluation tendency of oil to undergo autoxidation [21]. This finding indicates that the oil extracted from croissant samples is almost stable which provides a particular resistance to oxidation.

3.3 PHYSICOCHEMICAL CHARACTERISTICS OF THE OBTAINED BIODIESEL COMPARED TO ASTM D6751 AND EN 14214 STANDARDS

Table III shows the Physicochemical characteristics of the obtained biodiesel compared to ASTM D6751and EN 14214 standards. Moisture content has not been detected in the obtained biodiesel

samples. The presence of water in biodiesel causes microbial growth in storage tanks. This process could lead to the corrosion of metals, formation of sludge and slime, thereby causing blockage of fuel filters and fuel lines, which could, in turn, damage the vehicle fuel injection system [35]. The maximum amount of allowable water content in biodiesel as specified in the ASTM standard D6751 is 0.050% vol. [36]. Density is one of the most important physical properties of fuel products, because it is concerned with the mass of fuel that is injected into the combustion chamber and thus air-fuel ratio [37]. Density of the obtained biodiesel was 0.879 g cm-3. This value conforms to the density value recommended by the American Society for Testing and Materials [36] and European standard [38]. Viscosity parameter is one of the most important physical properties, required to identify the quality properties of biodiesel [19]. The dynamic viscosity at 40°C of the produced biodiesel was 23.61 centipoises at 40°C. Although viscosity is not compliant with biodiesel specifications, work is in progress to solve this problem by applying purification systems. The acid value is considered as an indicator of biodiesel deterioration. The high levels of free fatty acids have an influence on the engine fuel injection system and cause the corrosion of engine components [39]. The acid value of the obtained biodiesel was 0.48 mg KOH/g. This value was less than the maximum allowed acid value recommended by the American Society for Testing and Materials [36] and European standard [38]. Hydroperoxides are the initial products of oxidation process, these oxidations products increase the viscosity of biodiesel products [24]. Peroxide value of obtained biodiesel was 2.62 milliequivalent peroxides/Kg (Tab. III). Oxidation rancidity of biodiesel fuel is linked to its iodine value, which, in turn, is a measure of the unsaturation degree. Iodine value of obtained biodiesel was 61.17 g I_2/100 g. The maximum limit of iodine value was defined as 120 by biodiesel standard EN 14214. Oxidation stability is one of the most important properties of biodiesel fuel and primarily affects the stability of biodiesel during the storage period. The induction period of obtained biodiesel was 6.2 h. This value complies with international standards for biodiesel (six hours minimum to the European Standard EN 14214 and three hours minimum to the American Standard ASTM D6751. Biodiesel yield (%) of the obtained biodiesel was 86.45%. In this regard, Meneghetti et al. [40] reported that the maximum yield of biodiesel production using methanol was 90% with 1 h of reaction time at 60°C. The above-mentioned results indicate that the moisture content, density, acid value, peroxide value iodine number, and oxidation stability at 110°C of the obtained biodiesel meet the requirements of international standards for biodiesel.

4. CONCLUSIONS

The results indicate that the oil yield (%, wt./wt.) from croissant samples was 27.8%. The predominant fatty acids in extracted oil were oleic acid, palmitic acid, linoleic, stearic acid and linolenic-ω3 acid. The physico-chemical properties of the obtained biodiesel meet the requirements of international standards for biodiesel.

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Declarations Competing interests

The authors declare they have no competing interests.

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Table III - Physicochemical characteristics of the obtained biodiesel compared to ASTM and EN 14214 standards.

Characteristics	Values	ASTM specification	EN 14214
Moisture content %	ND	0.05 maxmum	–
Density (g cm^{-3})	0.879	0.87 to 0.90	0.86 to 0.90
Dynamic viscosity at 40°C (centipoises)	23.61		
Acid value mg KOH/g	0.48	0.8 maxmum	0.50 maxmum
peroxide value (milliequivalent peroxides/kg)	2.62	–	–
Iodine value(g I2/100 g)	61.17	–	120 maxmum
Oxidation Stability 110°C (hours)	6.2	3 minimum	6 minimum
Yields (%)	86.45	–	–

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