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Quality Assessment of Frying Oil from some Restaurants in Al Ahsa, Saudi Arabia

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تقييم جودة زيوت القلي في بعض مطاعم الأحساء بالسعودية

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ABSTRACT

The oxidation and hydrolysis products in frying oil greatly affect human health. The qualities of twenty-one samples of both fresh and in-use frying oils collected from some restaurants in Al Ahsa, Saudi Arabia were assessed. In fresh oil, the free fatty acids (FFAs), *p*-anisidine value (*p*-AV), total polar compounds (TPCs), and value of total oxidation (TOTOX), were of an acceptable quality, while the peroxide value (PV) in some samples was above the recommended limit. For the in-use oils, 9.5% had an FFA level above 2%. Additionally, the PV of in-use oil was 38% above the suggested limit, whereas 81% of in-use oil was beyond the recommended level for TOTOX. The in-use oil contained more conjugated diene and triene than fresh oil. The refractive index, viscosity, and colour increased in all in-use oil samples compared to fresh oil. Significant correlations between most parameters were observed. Institutions responsible for food control and inspection should consider these results for consumer health.

المخلص

تعد نواتج التحلل المائي والأكسدة للزيوت من المركبات التي تؤثر على صحة الإنسان. تم تقييم جودة إحدى وعشرين عينة زيت طازج وإحدى وعشرين عينة زيت أثناء القلي تم تجميعهم من بعض مطاعم مدينة الأحساء في المملكة العربية السعودية. قيم الأحماض الدهنية الحرة والبارا أنيسدين والمركبات القطبية الكلية وكذلك قيم الأكسدة الكلية في عينات الزيت الطازج كانت ذات جودة مقبولة، بينما قيم البيروكسيد كانت في بعض العينات أعلى من الحد المسموح للزيوت الطازجة. أظهرت الزيوت التي أخذت أثناء القلي أن 9.5% منها تحتوي على مستوى من الأحماض الدهنية الحرة أعلى من 2%. علاوة على ذلك سجلت 38% من هذه العينات قيماً أعلى من الحد المقترح المسموح به، في حين أن 81% من العينات التي أخذت أثناء القلي كانت في المستوى المرضي المسموح به من قيم الأكسدة الكلية. احتوت الزيوت التي أخذت أثناء القلي على كمية من مترافقات الداين أكبر من الزيوت الطازجة. ازدادت قيم كل من معامل الانكسار واللزوجة واللون في كل عينات الزيوت التي أخذت أثناء القلي بالمقارنة بالزيوت الطازجة. تمت ملاحظة وجود ارتباط معنوي بين معظم قياسات الزيوت المختبرة. وبناءً على هذه الدراسة، فإنه من الضروري أن تأخذ المؤسسات المسؤولة عن الرقابة والتفتيش الغذائي هذه النتائج بعين الاعتبار من أجل صحة المستهلك.

1. Introduction

Oils and fats are used as media to transfer heat from the fryer to food. Frying is a fast and inexpensive technique used at the household, restaurant, and industry levels, which create the unique sensory properties of fried foods. Frying of food at high temperatures leads to a palatable taste, attractive golden colour, and crispy food surface, which are highly favoured by many consumers. During the oil frying process, many chemical reactions occur including the hydrolysis of triglycerides due to the moisture of fried food, oxidation of oil due to exposure to oxygen and the oil's direct contact with the mineral cooking vessel surface, and thermal destruction of oil components due to exposure to high temperatures (Lina *et al.*, 2019; Song *et al.*, 2017). During frying, fried food absorbs significant quantities of frying oil that contains different compounds produced by the thermal chemical reaction in used oil (Li *et al.*, 2019; Sunisa *et al.*, 2011).

Scientific evidence suggests that many of these compounds pose serious health risks (Aladedunye and Przybylski, 2009). Scientists have identified several harmful compounds in frying oil such as volatile compounds and products of monomers, polymers and oil degradation products such as ketones, aldehydes, and oxidised triglycerides (namely epoxy, oxy, and hydroxyl-derivatives; and triglycerides in the form of dimeric, trimeric, or polymeric). Other compounds such as acrylamide, chloropropanol, and furan were also associated with frying (Li *et al.*, 2017; Lina *et al.*, 2019; Mesias *et al.*, 2019). The reactions of these chemicals also cause a loss of the oil's nutritional value through the breakdown of fat-soluble vitamins and essential fatty acids. Furthermore, unwanted changes in sensory properties (taste, flavour, and odour) may occur due to the presence of some primary and secondary oxidation products. The frying oil

quality must be studied carefully because the fried food delivers these oils to humans. The air of dining areas adjacent to cooking areas in restaurants also contains harmful products due to cooking oils (Dai *et al.*, 2018). The quality and safety of oil are assessed by several measurements. These measurements include chemical tests (hydrolysis and oxidation products such as FFAs, *p*-anisidine value, peroxide value, total polar compounds, and conjugated diene) and physical tests (refractive index, viscosity, and colour) as reported by Al-Kahtani (1991), Bansal, *et al.* (2010a), Karimi *et al.* (2017), Mudawi *et al.* (2014), Sebastian *et al.* (2014), Song *et al.* (2017), and Urbancic *et al.* (2014). These chemical and physical tests are highly affected by the time of heat exposure, frying temperature, the number of frying times, and type of oil (Song *et al.*, 2017; Sunisa *et al.*, 2011). Bansal *et al.* (2010b) also detected a linear increase in water-soluble compounds, viscosity, and refractive index by increasing the frying cycles in palm oil. During frying and storing, edible oil is exposed to oxidation via photosensitised oxidation and autoxidation (Choe and Min, 2006).

The method used in restaurants to evaluate frying oil depends on the human senses by observing the change in colour, foam appearance, and fumes. The oil can also be judged by the quality of the food produced by frying. However, this method cannot be trusted to assess the oil because the evaluator is usually the owner of the restaurant.

The safety and quality of frying oil in restaurants was reported by many researchers around the world, including Canada (Sebastian *et al.*, 2014), Kenya (Karimi *et al.*, 2017), and China (Li *et al.*, 2019). However, the most recent research paper on frying oil in Saudi Arabia was more than 26 years ago (Al-Kahtani, 1991). In Saudi Arabia, fast food restaurants are spread throughout many locations. These restaurants use frying oil to prepare many traditional foods such as

sambousek, falafel, potato, and chicken. There were approximately 14,553 fast food restaurants established in Saudi Arabia in 2015 (Statistics Portal, 2015). Studies on the quality assessment of frying oil are lacking in Saudi Arabia. Al-Kahtani (1991) examined the quality of 62 samples of cooking oil collected from fast food restaurants in Riyadh. However, even with the increase in fast food restaurants in recent years, references from this study have not been documented in the Eastern region, especially in Al Ahsa.

In addition, regulations and guidelines for the control of frying oil around the world are still not available. In Saudi Arabia, the Gulf Cooperation Council Standardization Organization (GSO, 2008) legislated (for refined oil) the maximum limits of FFAs (0.6 mg KOH/mL) and the peroxide value (10 meq/kg), but some oil measurements have not yet been reported.

Subsequently, the purpose of the present study was to assess the quality of frying oil collected from some restaurants throughout Al Ahsa City, Saudi Arabia by estimating the hydrolysis and oxidation products as well as physical characteristics to avoid the risks of these harmful compounds to consumer health.

2. Material and Methods

2.1. Oil Samples Collection:

Twenty-one samples, each of fresh and in-use frying oils, were collected randomly from twenty-one national and international chain restaurants in different locations of Al Ahsa, Eastern region, Saudi Arabia. Oil samples were taken with the approval of restaurant officials. The oil samples were collected into 50 mL dark glass bottles by using a glass pipette and automatic pipette filler. The oil samples were transferred to the laboratory in an icebox. After reaching room temperature, all the samples of collected oil were filtered using filter paper (Whatman No. 1) and kept frozen (-18°C) until analysis.

2.2. Frying Practices:

The description of frying oil collected from twenty-one restaurants in Al Ahsa are shown in Table (1). Frying processes varied according to the kind of restaurant and type of fried food products. Deep frying under pressure constitutes the most common frying technique for fish and chicken, while deep frying under atmospheric pressure is usually used for potatoes, falafel, sambousek, burgers, and sausage, and sometimes fish. Conventional open pan-frying is commonly used for falafel, potato, sambousek, and eggplant. Different kinds of frying oil (palm olein, soybean, and sunflower) are used to prepare fried foods (falafel, eggplant, sambousek, potato, chicken, burgers, sausage, and fish). Some oil producing companies labelled the oil additives, whereas others did not. The oil additives included antioxidants, crystallizing agents, and anti-foaming agents. All of the oil companies labelled the fatty acids composition (Table2).

Table 1. Description of frying oil collected from restaurants

No. of restaurant	Type of oil*	Fried food products	Additives*
1	Sunflower oil + palm olein	Fish	Antioxidant (TBHQ, BHT, Ascorbyl palmitate), Crystallizing agent (Sorbitan Tristearate) and Anti-foaming agent (Dimethyl-polysiloxane)
2	Sunflower oil + palm olein	Potato	TBHQ and Anti-foaming agent (Dimethyl-polysiloxane)
3	Palm olein	Potato	TBHQ and Anti-foaming agent (Dimethyl-polysiloxane)
4	Sunflower oil + palm olein	Falafel** + Potato + eggplant	Antioxidant (TBHQ, BHT, Ascorbyl palmitate), Crystallizing agent (Sorbitan Tristearate) and Anti-foaming agent (Dimethyl-polysiloxane)
5	Palm olein	Burgers + Sausage	Unlabelled
6	Palm olein	Falafel + Sambousek***	Unlabelled
7	Palm olein	Potato	Unlabelled
8	Palm olein	Potato + Sambousek	Unlabelled
9	Sunflower oil	chicken	Unlabelled
10	Sunflower oil+ palm olein + soybean oil	Falafel + Potato + eggplant	Anti-foaming agent (Dimethyl-polysiloxane)
11	Palm olein	chicken	Unlabelled
12	Palm olein	Falafel + Potato	Unlabelled
13	Palm olein	Potato	Unlabelled
14	Sunflower oil + palm olein	Potato	TBHQ and Anti-foaming agent (Dimethyl-polysiloxane)
15	Sunflower oil+ palm olein + soybean oil	Falafel + Potato	TBHQ and Anti-foaming agent (Dimethyl-polysiloxane)
16	Sunflower oil + palm olein	Falafel + Sambousek + Potato	Antioxidant (TBHQ, BHT, Ascorbyl palmitate), Crystallizing agent (Sorbitan Tristearate) and Anti-foaming agent

No. of restaurant	Type of oil*	Fried food products	Additives*
17	Sunflower oil + palm olein + soybean oil	chicken	TBHQ and Anti-foaming agent (Dimethyl-polysiloxane)
18	Sunflower oil + palm olein	Falafel + Potato+ eggplant	Antioxidant (TBHQ, BHT, Ascorbyl palmitate), Crystallizing agent (Sorbitan Tristearate) and Anti-foaming agent (Dimethyl-polysiloxane)
19	Sunflower oil + palm olein + soybean oil	falafel + Sambousek + Potato	TBHQ and Anti-foaming agent (Dimethyl-polysiloxane)
20	Palm olein	Falafel + Potato+ eggplant	TBHQ and Anti-foaming agent (Dimethyl-polysiloxane)
21	Palm olein	falafel + Sambousek + Potato	Unlabelled

*As described in the label, **Falafel is a deep-fried ball or patty made from ground fava beans or chickpeas, garlic, leek, and coriander, ***Sambousek is a pastry filled with meat, cheese, or vegetables,

Table 2. Fatty acids ratio of frying oil collected from restaurants*

No. of restaurant	Saturated fatty acid	Monounsaturated fatty acid	Polyunsaturated fatty acid
1	15-29	24-60	17-55
2	30-60	-	10-35
3	32-46	-	-
4	15-29	24-60	17-55
5	43-49	41-44	10-14
6	43-49	41-44	10-14
7	43-49	41-44	10-14
8	43-49	41-44	10-14
9	4.22	14-67	20-75
10	10.27	20.38	40-68
11	43-49	41-44	10-14
12	43-49	41-44	10-14
13	43-49	41-44	10-14
14	30-60	-	10-35
15	10-27	20-38	40-68
16	15-29	24.60	17-55
17	25-45	30-60	10-35
18	15-29	24.60	17-55
19	10-27	20-38	40-68
20	32-46	-	-
21	43-49	41-44	10-14

*As described in the label

2.3. Chemical Analysis:

2.3.1. Free fatty acids (FFAs)

FFAs of oil samples were estimated depending to the method of AOCS, Ca 5a-40 (AOCS, 1997), and a 5 g of sample was mixed into 50 mL of a 95% alcohol and petroleum ether mixture in a 250 mL conical flask by using a water bath. One millilitre of phenolphthalein was dropped into the mixture as an indicator, and the mixture was neutralised by 0.1 N KOH until the endpoint (pink colour) cleared. The FFAs are expressed as g/100 g oleic acid.

2.3.2. Peroxide value (PV)

Using the method of AOCS, Cd 8-53 (AOCS, 1998), the PV of frying oil samples was performed. One gram of oil was completely dissolved in 30 mL of a glacial acetic acid and chloroform mixture (3:2, v/v) in a 250 mL Erlenmeyer flask with a glass stopper. Half a millilitre of saturated potassium iodide was dissolved in the contents of the flask, and the flask was stopped and swirled for exactly one minute. 30 mL of distilled water was immediately mixed in. The flask was stopped and swirled vigorously. By using sodium thiosulphate (0.01N), the contents of the flask were titrated if the colour of the solution was a deep red orange until the colour lightened. One millilitre of 1% starch solution indicator was mixed well until the blue-grey colour faded. A sample was treated following the same procedure as a blank. The PV is expressed as meq O₂/kg oil.

2.3.3. *p*-anisidine value (*p*-AV)

Using the method of IUPAC, 2504 (1987), the *p*-AV was estimated. One hundred milligrams of oil were mixed with 25 mL of isoctane, then 0.5 mL of the *p*-anisidine reagent was mixed with 2.5 mL of this mixture. Following a 10-minute reaction time at ambient temperature, absorbance was measured by spectrophotometer at 350 nm (UV-1601, Shimadzu, Japan).

2.3.4. Total polar compounds (TPC₅)

According to the method of AOCS, Cd 20-91 (1998), the TPC₅ was assessed. 2.5 g of oil was put into a glass column (2.1 and 36 cm of diameter and length, respectively) packed with silica gel. The mobile phase (diethyl ether: petroleum ether, 13:87, v/v) was eluted to separate the non-polar fraction. The TPC content (%) was estimated as the TPC₅ mass fraction in the oil sample as a percentage.

2.3.5. Total oxidation value (TOTOX value)

Total oxidation is expressed as the TOTOX and was accounted as $TOTOX = p-AV + 2PV$.

2.3.6. Conjugated diene and triene

To evaluate the conjugated diene and triene in fresh and in-use oil samples, absorbance at 232 and 270 nm was measured, respectively. A 1% (w/v) solution of oil in isooctane was used to measure absorbance by a spectrophotometer, (UV-1601, Shimadzu, Japan).

2.4. Physical Analysis:

2.4.1. Refractive index

To estimate the refractive index of fresh and in-use frying oil samples, an Abbe refractometer (Milton Roy- K334610, USA) was used. The temperature was set at 20°C during the measurements.

2.4.2. Viscosity

The viscosity of oil samples was measured using a viscometer (Model LVDV-II+ Pro viscometer Brookfield, Middleboro, MA, USA). A circulating water bath (Thermo, Haake K10, Type-003-2859, Germany) was used to keep the temperature around $30 \pm 2^\circ\text{C}$. The spindle LV-2 (62) 20-100K was used for viscosity determination. The speed of the spindle was fixed at 50 rpm.

2.4.3. Colorimetric properties

To estimate the oil colour, a Hunter Lab (MiniScan XE Plus, Model No. 45/0-L, Reston, VA, USA) was used. Colour values were denoted as a^* (redness), b^* (yellowness), and L^* (lightness). The difference of total colour was calculated as follows:

$$\Delta E = [(a^i - a_0)^2 + (b^i - b_0)^2 + (L^i - L_0)^2]^{1/2}$$

a^i , b^i and L^i represent the a^* , b^* and L^* values respectively of in-use oil, and a_0 , b_0 and L_0 represent the a^* , b^* and L^* values respectively, of fresh oil.

Chroma (C^*) was calculated as follows: $C^* = (b^{*2} + a^{*2})^{1/2}$ (Bansal *et al.*, 2010b)

2.5. Statistical Analysis:

Statistical analysis was performed using SAS Statistical Computer Package V.9.1 (SAS Institute, Cary, NC, USA). The data of duplicate oil samples was calculated as the mean \pm SD. The data was subjected to analysis of variance (ANOVA one-way) in general linear models or the t-test. The Pearson linear correlation coefficient (r^2) was calculated to determine the relationships between different oil parameters.

3. Results and Discussion

3.1. Chemical Characteristics:

3.1.1. Free fatty acids (FFAs)

Chen *et al.*, (2013) mentioned that FFA content is one of the most significant parameters used to assess the quality of frying oil. The values of FFA from fresh and in-use frying oil samples are tabulated in Table 3 and 4, respectively. The values of FFAs in fresh frying oil were low in the range of 0.04 to 0.09% (Table 3). The low FFA values in fresh oil are due to the refining processes that take place during the manufacturing of oil that remove most FFAs from crude oil (Gustone, 2008). He reported that FFAs in fresh oil should be less than 0.1%. Thus, these values were within the permissible range in fresh oil. However, Gupta (2005) reported that the desired levels of FFA in fresh oil range from 0.03 to 0.05%. GSO (2008) reported that the maximum FFA of refined oil is 0.3%. Gustone (2008) and Karimi *et al.* (2017) reported similar FFA fresh oil values. The FFA value of in-use

oil ranged from 0.11 to 2.29% (Table 4), consistent with the findings of Karimi *et al.* (2017). The high FFA content in in-use oils may be due to hydrolysis of triglycerides in the presence of food moisture and high frying temperature. Aladedunye and Przybylski (2009), Bansal *et al.* (2010b), Chen *et al.* (2013), Sunisa *et al.* (2011), and Song *et al.* (2017) mentioned that the increase of FFA levels in frying oil are due to increased frying temperature, frying time, and frying cycles. The maximum accepted level of FFAs in frying oil varies between 0.5% and 1% according to Gupta (2005), whereas Tseng *et al.* (1996) reported that when the FFA levels are above 1%, the frying oil should be discarded. Accordingly, the results obtained from this study revealed that levels of FFA in 23.8% of in-use frying oil (Table 3) were beyond the recommended levels. Some countries have specific regulations for the maximum FFA level in frying oil. The USDA reported that if the FFA value is greater than 2%, frying oil should be discarded. The results of this study revealed that more than 9.5% of in-use frying oil had FFA values above 2%.

Table 3. Chemical characteristics of fresh frying oil

No. of restaurant	FFA (%)	PV (meq/kg)	p-AV (meq/kg)	TOTOX value (meq/kg)	TPC (%)	Absorbance (nm)	
						232	270
1	0.08±0.01	2.3±0.95	1.2±0.1	4.7	10.5±0.8	1.01±0.31	0.34±0.05
2	0.04±0.01	1.6±1.0	1.5±0.3	4.6	4.9±1.3	0.72±0.22	0.32±0.05
3	0.05±0.00	1.1±0.9	0.3±0.1	1.7	5.2±0.7	0.60±0.25	0.30±0.07
4	0.05±0.00	5.4±1.8	1.7±0.1	8.8	6.4±1.5	0.55±0.18	0.27±0.01
5	0.08±0.00	4.5±1.7	2.3±0.4	9.1	4.7±0.9	0.34±0.13	0.22±0.01
6	0.07±0.00	2.6±0.7	1.3±0.3	5.2	5.8±1.4	0.52±0.00	0.40±0.11
7	0.06±0.01	3.5±0.7	1.8±0.5	7.1	6.8±1.5	0.45±0.09	0.36±0.02
8	0.05±0.01	5.3±1.8	2.9±0.6	11.1	4.5±1.4	0.35±0.11	0.25±0.05
9	0.08±0.02	4.9±0.7	2.8±0.5	10.5	4.5±0.4	0.60±0.20	0.21±0.05
10	0.05±0.00	2.1±0.3	0.9±0.1	3.9	4.4±0.1	1.52±0.15	0.45±0.03
11	0.04±0.02	1.4±0.6	0.7±0.2	2.8	8.4±2.1	0.49±0.14	0.21±0.03
12	0.05±0.01	3.6±1.1	1.3±0.2	6.2	6.6±1.5	0.67±0.10	0.32±0.07
13	0.08±0.10	4.2±0.8	2.5±0.3	9.2	4.8±0.5	0.82±0.02	0.52±0.05
14	0.05±0.00	3.9±0.9	1.8±0.1	7.5	7.7±2.2	0.75±0.27	0.21±0.01
15	0.09±0.01	4.0±0.3	1.7±0.3	7.4	2.5±0.7	1.41±0.13	0.52±0.01
16	0.09±0.02	5.3±1.6	2.6±0.2	10.5	17.5±1.8	0.97±0.18	0.55±0.02
17	0.05±0.00	4.9±1.5	2.8±0.4	10.5	6.7±1.6	0.77±0.05	0.40±0.10
18	0.06±0.00	3.5±1.9	2.7±0.2	8.9	7.1±0.9	0.57±0.07	0.34±0.00
19	0.05±0.00	2.2±1.3	2.0±0.1	6.2	7.5±2.9	1.63±0.07	0.41±0.09
20	0.05±0.01	2.1±0.5	1.5±0.4	5.1	4.2±0.8	0.53±0.10	0.28±0.03
21	0.07±0.01	3.6±1.1	2.0±0.2	7.6	8.4±1.7	0.90±0.27	0.54±0.07
Mean±SD	0.06±0.02	3.4±1.4	1.8±0.7	7.1±2.7	6.6±3.1	0.77±0.3	0.35±0.11

3.1.2. Peroxide Value (PV)

PV is a very important factor used to assess the quality of frying oil. The PV of freshly refined oil should be less than 1 meq/kg, and if the PV rises to 10 meq/kg, the oil is considered rancid (Gustone, 2008). However, Sulieman *et al.* (2006) suggested that the characteristics of frying oil quality should have a PV of less than 2 meq/kg oil. The average of PVs in fresh oil samples fell within the range of 1.1 to 5.4 meq/kg, as presented in Table 3. These findings may be because the oil is exposed to oxidation via autoxidation as well as photosensitized oxidation through frying and storing (Choe and Min, 2006). Matthaus (2007) mentioned that the oil oxidation depends on temperature, light, time, the presence of moisture, and metals. The PV of the in-use oil exceedingly ranged from 3.4 to 42.5 meq/kg oil; 38% of in-use oil samples had higher than the suggested limit of 10 meq/kg (Table 4). According to GSO (2008), the maximum allowed PV of refined oil is 10 meq/kg. The appearance of hydroperoxides is associated with the capability of fatty acids to oxidise and the antioxidant level (all oil sample contained antioxidants as an additive agent, Table 1). In-use oil samples number 1, 4, 9, 10, 15, 16, 18 and 19 generally had the highest PV. The increase in the PV of in-use frying oil may be due to the presence of a large proportion of polyunsaturated fatty acid (such as linoleic acid) characteristic of sunflower oil. Choe and Min (2006) reported that edible oil is exposed to oxidation via autoxidation and photosensitized oxidation during frying and storing.

Table 4. Chemical characteristics of in-use frying oil

No. of restaurant	FFA (%)	PV (meq/kg)	p-AV (meq/kg)	TOTOX value (meq/kg)	TPC (%)	Absorbance (nm)	
						232	270
1	0.14±0.04	13.5±1.3	51.4±84.4	93.9	21.9±1.7	2.11±0.52	0.63±0.14
2	0.15±0.00	12.7±1.7	40.6±3.7	123.1	21.4±2.5	1.97±0.55	0.56±0.12
3	0.16±0.00	7.9±1.1	57.6±4.1	118.5	6.0±0.6	0.55±0.21	0.31±0.08

No. of restaurant	FFA (%)	PV (meq/kg)	p-AV (meq/kg)	TOTOX value (meq/kg)	TPC (%)	Absorbance (nm)	
						232	270
4	1.71±0.10	21.5±1.1	48.5±5.2	131.8	15.3±3.6	1.72±0.78	0.45±0.01
5	0.74±0.07	20.6±3.1	55.6±2.6	109.9	10.3±2.7	0.89±0.46	0.41±0.06
6	0.52±0.08	15.5±2.7	47.2±4.2	103.5	17.9±2.3	1.76±0.11	0.55±0.02
7	0.19±0.05	12.3±0.9	45.6±2.7	12.4	19.6±2.7	0.98±0.23	0.66±0.19
8	2.07±0.04	3.4±0.9	4.5±1.0	198.3	5.9±0.5	0.78±0.05	0.63±0.11
9	0.11±0.05	42.5±3.5	77.9±7.6	137.5	33.6±5.8	1.45±0.40	0.73±0.05
10	0.22±0.07	16.9±1.2	60.3±5.3	93.9	13.5±0.5	2.02±0.95	0.71±0.05
11	0.55±0.5	3.7±0.9	5.6±0.8	13.0	7.2±1.3	0.55±0.23	0.35±0.04
12	0.95±0.05	11.2±2.5	25.1±3.1	61.4	7.8±2.0	0.91±0.21	0.66±0.06
13	2.29±0.71	12.9±1.5	33.6±5.8	80.1	10.6±1.9	1.34±0.17	0.77±0.15
14	0.64±0.04	16.1±4.2	36.7±4.8	89.5	15.7±3.5	1.50±0.65	0.87±0.14
15	1.17±0.50	20.3±1.7	29.8±6.3	79.9	7.7±1.8	2.97±0.59	0.92±0.05
16	1.98±0.23	17.5±2.5	33.2±3.8	83.9	7.7±1.8	2.97±0.59	0.92±0.05
17	0.87±0.14	7.8±2.1	10.1±0.2	28.0	5.5±1.2	0.71±0.34	0.37±0.12
18	0.98±0.31	12.5±2.0	25.7±5.2	63.9	11.3±2.4	0.91±0.24	0.48±0.05
19	0.78±0.03	15.1±2.9	35.8±3.5	86.7	10.5±2.1	2.95±0.71	0.61±0.14
20	0.55±0.04	3.9±1.4	7.4±1.2	18.7	5.2±1.1	0.52±0.20	0.27±0.12
21	0.78±0.05	4.1±1.3	6.8±0.8	17.7	5.9±0.4	0.87±0.05	0.50±0.01
Mean ±SD	0.84±0.67	13.9±8.6	35.2±20.4	83.1±47.5	13.0±7.3	1.38±0.73	0.58±0.18

3.1.3. p-anisidine value (p-AV)

Oxidation forms successive compound groups, starting with primary oxidation compounds (fatty acid hydroperoxides) and followed by degradation to form secondary compounds (α , β unsaturated aldehydes) (Bansal et al., 2010b). The peroxide products presented during primary oxidation were declared unstable compared to the secondary oxidative products. These compounds caused a rancid smell in oil and can be detected using p-anisidine. Al-Kahtani (1991) and Sebastian et al., (2014) suggested that the p-AV is a critical and more credible parameter for the assessment of the oil oxidative state, because it measures the secondary stage of oxidation or the accumulation of secondary products. The same authors described that p-AVs are the most accurate and reliable value. In Table 3, the p-AVs ranged from 0.3 to 2.9 meq/kg in fresh oil samples, which are the same results found by Sebastian et al. (2014) and Karimi et al. (2017). An accepted p-AV of fresh oil ranged from 4.0 to 6.0 meq/kg according to Gupta (2005). The p-AV of fresh oil in the current study of fresh oil was of desirable quality. The p-AV of in-use oil fell in the range of 4.6 to 77.9 meq/kg (Table 4). Only two samples (9.5%) had a p-AV less than the recommended limit and were of an acceptable oxidative status. Gupta (2005) indicated that, if the p-AV of frying oil rises above 6 meq/kg, the oil is considered highly oxidised. The p-AV of frying oil increased with the number of frying cycles (Song et al., 2017). These findings were also verified by Karimi et al. (2017), who found that none of the tested frying oil samples used in restaurants had an acceptable oxidative status.

3.1.4. Total oxidation value (TOTOX)

The p-AV plus PV can be used as an indicator for the total oxidation state of frying oil (Van Der Merwe et al., 2003); this value is calculated by the TOTOX value. The TOTOX value in fresh frying oil samples ranged from 1.7 to 10.5 meq/kg (Table 3), indicating that the qualities of fresh oil samples were acceptable. As reported by Pereira De Abreu et al. (2010), the suggested standard TOTOX value is ≤ 19.5 meq/kg. For in-use oil, the results indicated that the TOTOX values in 81% of the samples were beyond the recommended level (Table 3). These results suggest that 19% of in-use oil assessed in this study had an agreeable oxidative status.

3.1.5. Total polar compounds (TPCs%)

TPCs reflect the levels of oxidative breakdown and hydrolysis products, such as FFAs, ketones, aldehydes, hydroperoxides, acids, alcohols, and epoxides present during oil storing and frying. According to Uriarte and Guillen (2010), the oil must be discarded if the TPCs are between 24 to 27%. In our study, the TPCs in fresh oil ranged from 2.5 to 10.5% (Table 3), indicating acceptable quality. For in-use oil, the TPCs ranged from 5.5 to 21.9%. These values were also within the recommended levels. These results revealed that all of the oil samples are of an acceptable quality. Aladedunye and Przybylski (2009), Song et al. (2017), and Urbancic et al. (2014) mentioned that

TPCs increased significantly with prolonged frying times and temperature. Li et al. (2017) found that the formation of TPC products in French fries, as a result of the reaction between carbohydrates, is enhanced with the increase of hydrophobic properties. Sebastian et al. (2014) obtained similar results while analysing oil samples collected from restaurants and found that TPCs ranged from 1 to 3% and 4.0 to 15.5% in fresh and in-use oil, respectively. However, Karimi et al. (2017) reported that the TPCs in most fresh and in-use oil samples collected from restaurants were above the recommended range.

3.1.6. Conjugated diene and triene

Absorbance at 232 nm is correlated with the conjugated diene of polyunsaturated fatty acids, while absorbance at 270 nm measures the presence of primary and secondary oxidation (Urbancic et al., 2014). The absorbance of conjugated diene and triene at 232–270 nm, respectively, is used to assess oxidative stability (Che Man and Jaswir, 2000; Urbancic et al., 2014). The absorbance of conjugated diene and triene at 232–270 nm is shown in Table 4. In this study, conjugated diene and triene were present in the fresh oil samples in small quantities ranging from 0.34 to 1.01 and from 0.21 to 0.54, respectively (Table 3). However, in the case of in-use frying oil, the values ranged from 0.52 to 2.97 and 0.27 to 0.92, respectively (Table 4). The in-use oil samples came from restaurant numbers 10, 15, and 19 with a higher content of conjugated diene compared to other oils. This is because sunflower oil contains a high amount of polyunsaturated fatty acid present in linoleic acid. The increase in the conjugated diene and triene is associated with increased frying cycle numbers, which has been mentioned in previous literatures (Che Man and Jaswir, 2000; Che Man and Tan, 1999; Song et al., 2017; Urbancic et al., 2014).

3.2. Physical Characteristics:

3.2.1. Refractive index (RI)

The refractive indices of fresh and in-use oils are tabulated in Table 5. The refractive index recorded high values in all in-use tested oil samples compared to fresh oil. The increased refractive index of in-use oil samples was due to oil exposure to heat and/or light during frying. Arya et al., (1969) observed that exposure to heat and light caused an increase in the refractive indices of oil and fats significantly. They also observed a relationship between the status of peroxide expansion and the refractive index values of the autoxidised oil or fats. Bansal et al. (2010b) reported that oil degradation increases the refractive index values depending on the kind of food fried in the oil. Mudawi et al. (2014) observed that the refractive index exhibited a significant increase in both corn oil and sunflower oil from frequent frying.

Table 5. The physical characteristics (refractive index, viscosity, and colour) of fresh and in-use oil

No. of restaurant	Refractive index		Viscosity (Cp)				Colour						
	FO	UO	FO	UO	L*	a*	b*	Chroma (C*)	L*	a*	b*	Chroma (C*)	ΔE
1	1.4728	1.4731	13.7±0.5	21.7±0.3	85.05	5.28	18.53	19.26	65.29	4.88	51.52	51.75	38.46
2	1.4727	1.4735	17.3±0.2	22.9±0.5	78.25	7.58	14.28	16.17	74.25	3.52	43.09	43.23	29.37
3	1.4648	1.4650	18.8±0.3	20.1±0.4	51.29	4.08	17.13	17.61	50.25	3.04	44.29	44.39	27.20
4	1.4728	1.4738	17.1±0.1	29.1±0.4	63.05	2.46	8.91	9.24	59.36	2.33	61.41	61.45	52.63
5	1.4649	1.4657	19.6±0.3	28.7±0.3	60.21	3.89	13.08	13.65	55.32	3.28	63.17	63.26	50.33
6	1.4647	1.4656	19.7±0.3	28.6±0.5	67.56	3.67	14.51	14.97	36.75	3.26	34.11	34.30	20.00
7	1.4647	1.4658	21.1±0.6	26.6±0.6	65.25	3.65	15.10	15.53	61.38	2.54	32.83	32.93	18.18
8	1.4651	1.4668	19.1±0.3	31.4±0.7	51.36	3.02	11.38	11.77	40.25	3.56	35.97	36.15	26.99
9	1.4646	1.4659	18.7±0.3	28.7±0.3	50.78	4.21	17.45	17.95	49.45	3.69	71.54	71.64	54.11
10	1.4728	1.4730	14.1±0.4	19.1±0.3	66.54	6.36	15.47	16.73	60.25	4.78	68.52	68.69	53.44
11	1.4649	1.4651	19.4±0.3	20.7±0.3	53.62	4.78	16.36	17.04	51.02	4.12	39.10	39.32	22.90
12	1.4728	1.4736	17.1±0.3	25.1±0.2	61.07	5.54	18.29	19.11	59.23	3.32	41.27	41.40	23.16
13	1.4648	1.4656	20.4±0.5	24.9±0.5	57.56	5.54	21.58	22.28	55.87	3.36	37.55	37.70	16.21
14	1.4728	1.4738	14.9±0.5	29.1±0.4	62.74	7.01	19.58	20.80	58.68	5.55	45.62	45.96	26.40
15	1.4730	1.4740	14.2±0.4	29.9±0.7	61.47	7.45	14.23	16.06	43.68	4.21	67.89	68.02	56.62
16	1.4728	1.4735	17.1±0.1	22.9±0.6	60.12	6.75	17.44	18.70	54.07	4.25	75.84	75.96	28.77
17	1.4727	1.4733	14.1±0.2	19.9±0.5	60.37	4.58	15.24	15.91	58.87	2.69	35.19	35.29	50.10
18	1.4728	1.4636	13.5±0.1	24.1±0.6	65.35	3.98	17.58	18.57	55.88	4.52	61.57	61.74	45.02
19	1.4729	1.4738	17.1±0.4	21.1±0.3	67.43	4.32	19.88	20.34	62.25	2.57	70.11	70.16	50.53
20	1.4726	1.4732	17.6±0.3	19.2±0.5	77.84	3.25	14.24	14.61	62.35	2.41	50.88	50.94	39.79
21	1.4727	1.4733	17.3±0.4	19.6±0.4	71.09	4.44	12.54	14.58	63.28	1.87	38.10	38.15	27.30
Mean	1.4697	1.4700	17.2	24.4	63.71	5.38	15.85	16.71	57.36	3.51	50.93	51.07	36.07

SD	0.0040	0.0041	2.3	4.1	9.02	11.57	3.03	3.06	7.61	0.96	14.45	14.43	14.66
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FO, fresh oil and UO, in-use oil

3.2.2. Viscosity

The viscosity of fresh and in-use frying oils are shown in Table 5. Fresh oil viscosity varied between restaurants depending on the types of oil and the composition of fatty acids (Table 5). Kim *et al.* (2010) reported that oil viscosity varied depending on its molecular structure and its fatty acid constituents as saturated fatty acids increased oil viscosity while unsaturated fatty acids decrease it. In the current study, the viscosity of in-use oil was higher than fresh oil in all tested samples (Table 5). These findings may be due to oxidation produced during the frying process. This behaviour was also reported in previous studies (Bansal *et al.*, 2010b; Santos *et al.*, 2005; Tseng *et al.*, 1996). They reported that viscosity increases with increased frying time and frying cycle numbers.

3.2.3. Colorimetric properties

The colour of frying oil is commonly used as a visual indication of its quality and validity. A change in oil colour is a result of the incorporated results of chemical changes such as oxidation and polymerization (Maskan, 2003). The colours of fresh and in-use frying oil are shown in Table 5. The results revealed that the L* value was low in in-use oils compared to fresh oils in all tested samples, which may be due to the presence of carbonised food residues and oxidised fatty acids, as reported by Belbin (1999). The a*, b* and chroma (C*) values increased from fresh oil to in-use oil, which may be due to the degradation of natural fat-soluble pigments such as carotenoids and chlorophylls. Sikorska *et al.* (2007) reported that degradation of carotenoids and chlorophylls during storing and frying obviously affects oil colour. The total colour difference (ΔE) ranged from 18.18 to 58.77. Most samples with higher values of oxidative breakdown and hydrolysis products (peroxide, *p*-anisidine, and total polar compounds) recorded higher ΔE values and an increase in ΔE in-use oil compared to fresh oil (Karimi *et al.*, 2017). Additionally, Bansal *et al.* (2010b) found a linear correlation between the values of b*, C* and ΔE with the frying cycle numbers. Aladedunye and Przybylski (2009) reported that the colour of frying oil increased significantly as a function of frying time and temperature. The change in colour varied according to the type of food fried, duration of exposure to light, heat, and the oil type (Choe and Min, 2007).

3.3. Correlation Analysis:

Correlation analysis was used to explain the relations among the tested oil parameters assessed in the present study (Table 6). There were relatively high correlation coefficients between most parameters (PV, *p*-AV, TOTOX, TPCs, viscosity, and ΔE), with significant correlations between one another. A weak correlation between RI and other parameters was observed. FFAs recorded relatively high correlation coefficients with TOTOX, viscosity, and ΔE while it showed a weak correlation with other parameters. It is noteworthy that the viscosity had significant correlations with FFAs, PV, *p*-AV, TOTOX and conjugated diene (A_{270}). This result indicates that oil viscosity increases with an increase in FFAs, primary and secondary oxidation products, and conjugated diene.

Due to the presence of antioxidants (synthetic: TBHQ, BHT and ascorbyl palmitate, and natural: tocopherols and carotenoids), a crystallizing agent (sorbitan and tristearate) and an anti-foaming agent (dimethyl-polysiloxane), oxidative degradation continued. This might be because antioxidants evaporate and are volatile at the frying temperatures used and because of the foam that occurs during frying (Uriarte and Guillen, 2010).

Table 6. Correlation coefficient analysis between parameter assessed in this study (n=42 per parameter)

	FFA	PV	<i>p</i> -AV	TOTOX	TPC	A_{232}	A_{270}	Refractive index	Viscosity	ΔE
FFA	1.00	0.35 ^a	0.30 ^a	0.60 ^a	0.13 ^{ns}	0.28 ^{ns}	0.53 ^a	0.07 ^{ns}	0.62 ^a	0.60 ^a
PV		1.00	0.87 ^a	0.69 ^a	0.78 ^a	0.57 ^a	0.63 ^a	-0.08 ^{ns}	0.70 ^a	0.73 ^a
<i>p</i> -AV			1.00	0.76 ^a	0.75 ^a	0.56 ^a	0.58 ^a	-0.17 ^{ns}	0.67 ^a	0.70 ^a
TOTOX				1.00	0.54 ^a	0.50 ^a	0.57 ^a	-0.10 ^{ns}	0.78 ^a	0.69 ^a
TPC					1.00	0.47 ^{ns}	0.56 ^a	-0.001 ^{ns}	0.50 ^a	0.51 ^a
A_{232}						1.00	0.71 ^a	0.31 ^a	0.38 ^a	0.52 ^a
A_{270}							1.00	0.14 ^{ns}	0.62 ^a	0.54 ^a
Refractive index								1.00	-0.26 ^{ns}	0.13 ^{ns}
Viscosity									1.00	0.67 ^a
ΔE										1.00

Values represent the squared Pearson correlation coefficient (r^2) for the linear correlation analysis. a P< 0.0001, b P< 0.001, c P< 0.01, d P< 0.05, ns P< 0.05

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