

The relationship between the contact angle and some quality parameters of frying oils

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The changes in several physicochemical parameters including fatty acid composition, total polar compounds, colour changes and contact angles of sunflower oils in the deep-frying process were determined. A deep-fat fryer was used to fry chicken and fish for 270 minutes at $180\pm 5^{\circ}\text{C}$ without oil replenishment and oil samples were collected at 30, 90, 150, 210, and 270th minutes of the frying process. TPM values which represents the amphiphilic compounds in oils were higher in the used oil than in the control oil. The contact angle values that may influence the oil uptake during frying and post-fry cooling were lower in used oils than in the control oil. Cox and Totox values were higher in oils subjected to chicken frying for 270 minutes compared to oils used to fry fish for 270 minutes.

Keywords: Contact angle, correlation, deep fat frying, sunflower oil

INTRODUCTION

Frying process is one of the most widely used food processes to give the product the desired flavour and aroma [1]. During frying, oils are transferred to small cavities in food and replace water and/or steam. Many complex chemical reactions occur while food continues to be fried in heated oil causing the production of degradation products [2]. The changes in the physicochemical properties of fats during frying have been used as an indicator of the chemical degradation of the frying means. Determination of total polar materials (TPM), para-anisidin value (p-AV), peroxide value (PV), UV absorbance values (K_{232} and K_{270}), total oxidation (TOTOX) value and free fatty acids (FFA) are the most commonly applied methods used in the oil industry to evaluate the degradation of oils [3].

The physicochemical properties of fried oils are also influenced by frying because of the formation of surface active agents [4]. The surface and interfacial tension play a vital role in oil intake mechanisms as frying oil enters into small pores of food filled with water and/or steam [5]. There are many methods for determining the interfacial and surface tensions including the Du Nouy ring method, the Wilhemny plate method, the capillary rise method, the maximum bubble method, the Pendant drop and the Sessile drop methods [6]. There is no ideal surface tension determination method for all systems. Factors such as the quantity and characteristics of the sample, the measurement temperature, the surface tension or the interfacial tension should be taken into account when selecting the appropriate method, such as whether the time parameter is within the scope of the study or not [7].

Utilisation of surface properties of oils as an oil degradation parameter is not a

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common method and the contact angle of oils have been studied by very few researchers. In a study by Gil and Handel (1995), it was suggested that the increase in the oil intake of the frying doughnuts due to the decrease in the frying oil/water interface tension [8]. It was determined that there was an important correlation between the oil intake of French fries and the contact angle of sunflower oil that was measured on a glass substrate [9]. A significant decrease in the interfacial tension of olive and palm oils was also observed by Kalogianni et al. (2011) with oils subjected to repeated frying cycles [4]. Recently, surface tension, viscosity dynamic and kinematic contact angles of control and used oils were determined to examine the effects of oil degradation [5]. Frying has an important effect on the surface properties of oil and the effect needs to be predicted with different frying means to attain a better understanding of the oil absorption and rapid methods in order to estimate the quality of frying oil are great interest for researchers. Therefore, this study aimed to determine the physicochemical properties of oils used in deep fat frying and estimate the contact angles of oils used in different frying medium in oil degradation.

EXPERIMENTAL PART

CHEMICALS AND OILS

All reagents and solvents (Ethanol, methanol, cyclohexane, chloroform, acetic acid, potassium iodide, sodium carbonate, hexane, sodium thiosulfate, potassium iodide, starch reagent and isooctane) were purchased from Sigma-Aldrich. The analytical grade was used for all other unlabelled chemicals and reagents. Fish fillets, chicken breasts and sunflower oil were purchased from a local supermarket.

PHYSICOCHEMICAL PARAMETERS

Physicochemical parameters including FFA, PV, p-AV were measured based on the AOCS Official Method Cd 3d-63, Cd 8b-90 and Cd 18-90, respectively (AOCS, 2009) [10]. The TOTOX value was calculated using the following formula [11], [12]:

$$\text{TOTOX} = 2\text{PV} + \text{p-AV} \quad (1)$$

UV absorbance coefficients (K232 and K270) were determined according to the European Official Method of Analysis (Commission Regulation EEC N-2568/91, 1991) [13]. Firstly 0.25 g of sunflower oil sample was weighed and completed with 25 mL cyclohexane in a flask. The sample was homogenised with the help of vortex (Velp Scientifika, Europe) for 30 seconds and put into a quartz cuvette (10 mm). The absorption was measured at 232 and 270 nm in a spectrophotometer (Shimadzu UV-2450 UV-Visible

Spectrophotometer, Tokyo, Japan). Pure cyclohexane was used as a blank.

Absorbance of turbid materials in used oil was determined and compared with the control. The turbidity of the control and used oils was determined using a Jenway turbidimeter (model 6035) in the wavelength range of 550-600 nm.

Colour changes in the oils were measured by Lovibond colour meter (RT850i device, USA). The resulting colour was defined as a colour space $L^*a^*b^*$ and chroma (c) that describes the vividness or dullness of a colour. Oil samples were measured in quartz cuvettes with 10-mm optical path length. The differences between the colour changes of the individual samples were expressed through the ΔE colour difference that is calculated with the equation below:

$$\Delta E = [(\Delta L^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2]^{1/2} \quad (2)$$

Where: ΔL^* , Δa^* , and Δb^* are the differences of these values between the samples control and after frying treatment.

FATTY ACID COMPOSITION AND CALCULATED OXIDABILITY INDEX

The European Official Methods of Analysis (EEC, 1991) was used in the preparation of methyl esters. The oil sample (0.1 g) was added in 20 mL centrifuge tubes and the oil sample was dissolved in 10 mL of pure n-hexane and interesterified to its methyl esters with the addition of methanolic potassium hydroxide (KOH) solution (2.8 g KOH in 25 mL methanol). Then the oil sample solution was vortexed for 30 s and centrifuged at 5000 rpm for 15 min. Clear supernatant was transferred into the vial via syringe and 0.45 μm syringe filter. After filtration supernatant was injected into the gas chromatography (GC) instrument. Analytical Conditions: Chromatographic analyses were carried out on an Agilent 6890 GC equipped with SP-2380 fused silica capillary column (60 m \times 0.25 mm \times 0.2; Supelco, Bellefonte, PA) and a flame ionisation detector (Agilent Technologies, Santa Clara, USA) equipped with Agilent 7683 auto-sampler. The GC oven was held at 120°C for 1 min and then, increased to 240°C at a rate of 6°C/min and, finally, kept at 240°C for 15 min. The temperature of the GC oven was held at 120°C for 1 min and increased to 240°C at a rate of 6°C/min, and kept at 240°C for 15 min. The temperatures of the detector and injector were 250°C.

The calculated oxidizability (COX) value of the oils was determined according to the formula explained by Moghaddam *et al.*, (2012) [14].

$$\text{Cox value} = \frac{1(18:1\%) + 10.3(18:2\%) + 21.6(18:3\%)}{100} \quad (3)$$

Where:

18:1% is the percentage of oleic acid content,

18:2% is percentage of linoleic acid content,

18:3% is the percentage of linolenic acid content [15].

THE CONTACT ANGLE AND TOTAL POLAR MATERIAL (TPM) MEASUREMENTS

The contact angle of used and control sunflower oils was measured by the sessile drop method using a KRUSS goniometer (Model DSA 100 E). A 1 ml syringe with a 22-gauge polymer needle (0.44 mm internal diameter) was used to form the drops on a PTFE surface with air as the surrounding media. The static contact angle was determined using Drop Shape Analyser Image software at room temperature described by Aydar et al 2016 [16]. Disposable syringes were used for every oil sample to avoid contamination that may influence the accuracy of contact angle measurements of oils. The volume was controlled using contact angle tools on the software and a drop of 4 μ L was dispensed, and the baseline was adjusted to measure the static contact angle. The surface was cleaned with acetone and left to dry before every measurement. The total time to determine the contact angle of oils did not exceed 60 seconds and the contact angle of sunflower oils did not change significantly (s.e \pm 0.1) during the 60 seconds of measurement.

Total polar materials that are indicative of all kinds of oxidation and hydrolysis substances in oils were quantified by Testo 270 (Testo, Lenzkirch, Germany) [17]. The total polar compounds measurements were conducted directly on the oil; oil samples were heated previously in a water bath to reach 50 \pm 5 $^{\circ}$ C, the temperature of the oil was observed on the display for 10 seconds. All tests were carried out in triplicate.

FRYING TREATMENT

5 L of sunflower oil (SO) was placed in a 10.8 L stainless deep fryer (24 \times 30 \times 15 cm) with temperature

control, and then the oil was heated to and kept at 180 \pm 5 $^{\circ}$ C. Then fish fillets and chicken breasts (500 g \pm 10 g) were fried for 10 min. 3 pieces of fish fillets and chicken breasts were used in each test. The frying procedure was held constantly for 3 continuous days (4.5 h per day). Deep-fried oil (150 mL) was collected at 30th minutes of frying and after every 60 minutes until the 270th minutes. Before analysis, all oil samples were stored at 4 $^{\circ}$ C in darkness using dark coloured bottles to avoid any degradation or contamination.

STATISTICAL ANALYSIS

Three replicates were performed for each frying treatment and each treatment was conducted on a different day. Data was analysed by analysis of variance (ANOVA) using SAS, 9.2 software (SAS Institute, Cary, NC). Tukey's HSD (α = 0.05) was applied on measured physicochemical and surface properties for all the oil samples to evaluate multiple comparisons. The linear relationship between the contact angle and other quality parameter variables were estimated using the Pearson correlation coefficient by following the PROC CORR procedure in SAS

RESULTS AND DISCUSSION

PHYSICOCHEMICAL PARAMETERS

Table I and Table II summarise the data relative to some physicochemical parameters of oils used in chicken and fish frying, respectively for 270 minutes. The parameter K_{270} , a marker of secondary oxidation products, increased in both oils and the increase in K_{270} during frying did not change statistically ($p > 0.05$). While time for chicken frying has no significant effect on the K_{232} value that is an indicator of the first oxidation products, K_{232} values were found significantly different in oil used during fish frying ($p > 0.05$).

The frying time promoted the occurrence of hydrolysis in the samples since changes in free acidity values

Table I - Physicochemical properties of sunflower oils used in chicken frying

	Control	30 min.	90 min.	150 min.	210 min.	270 min.
FFA	0.21 \pm 0.00 ^c	0.20 \pm 0.00 ^c	0.30 \pm 0.02 ^b	0.38 \pm 0.05 ^a	0.35 \pm 0.04 ^{ab}	0.41 \pm 0.02 ^a
K ₂₃₂	2.54 \pm 0.02 ^a	2.54 \pm 0.03 ^a	2.56 \pm 0.04 ^a	2.57 \pm 0.04 ^a	2.59 \pm 0.03 ^a	2.61 \pm 0.02 ^a
K ₂₇₀	2.31 \pm 0.06 ^a	2.41 \pm 0.03 ^a	2.47 \pm 0.03 ^a	2.47 \pm 0.05 ^a	3.03 \pm 0.05 ^a	3.04 \pm 0.12 ^a
p-AV	1.15 \pm 0.00 ^f	2.71 \pm 0.02 ^e	5.11 \pm 0.11 ^d	6.09 \pm 0.13 ^c	9.33 \pm 0.04 ^b	11.64 \pm 0.01 ^a
PV	1.18 \pm 0.17 ^d	2.08 \pm 1.02 ^{dc}	3.72 \pm 1.55 ^{bc}	5.15 \pm 0.84 ^{ab}	6.03 \pm 0.71 ^a	5.65 \pm 1.45 ^{ab}
L	132.16 \pm 1.83 ^a	129.15 \pm 2.01 ^a	104.84 \pm 2.93 ^b	82.55 \pm 2.13 ^d	92.46 \pm 0.53 ^c	101.25 \pm 0.79 ^b
c	255.28 \pm 34.18 ^a	235.33 \pm 33.14 ^a	132.50 \pm 29.93 ^b	49.61 \pm 27.77 ^{bc}	52.88 \pm 29.15 ^{bc}	32.42 \pm 22.47 ^c
Δ E	0.00 \pm 0.00 ^e	20.48 \pm 1.20 ^d	129.53 \pm 2.32 ^c	218.09 \pm 2.25 ^b	215.92 \pm 3.77 ^b	225.43 \pm 3.68 ^a
Turbidity	1.82 \pm 0.23 ^d	4.77 \pm 1.44 ^{dc}	17.14 \pm 2.50 ^{bcd}	26.55 \pm 2.35 ^{bc}	33.04 \pm 19.07 ^b	67.55 \pm 4.65 ^a
Contact angle	62.22 \pm 0.35 ^a	60.38 \pm 1.23 ^{ab}	57.85 \pm 0.89 ^{bc}	55.39 \pm 1.17 ^{dc}	54.70 \pm 1.39 ^{dc}	51.70 \pm 1.12 ^d
TPM	6.50 \pm 0.07 ^c	6.50 \pm 0.03 ^c	8.50 \pm 0.49 ^{bc}	9.50 \pm 0.07 ^b	8.50 \pm 0.64 ^{bc}	10.50 \pm 0.71 ^a

FFA: (%), PV: meqO₂/kg. Contact Angle: $^{\circ}$. Turbidity: NTU, TPM: %.

Mean \pm SD (n = 3). Means within a row with the same letter are not significantly different as indicated by Tukey's HSD test (P < 0.05).

Table II - Physicochemical properties of sunflower oils used in fish frying

	Control	30 min.	90 min.	150 min.	210 min.	270 min.
FFA	0.29±0.01 ^e	0.30±0.01 ^e	0.33±0.01 ^d	0.41±0.01 ^c	0.51±0.01 ^b	0.58±0.01 ^a
K232	2.55±0.03 ^c	2.77±0.04 ^b	2.74±0.06 ^{bc}	2.89±0.04 ^{ab}	2.99±0.04 ^a	3.04±0.05 ^a
K270	2.31±0.04 ^a	2.42±0.08 ^a	2.39±0.13 ^a	2.47±0.15 ^a	3.01±0.18 ^a	3.29±0.16 ^a
p-AV	1.31±0.05 ^f	1.96±0.07 ^e	3.76±0.08 ^d	5.14±0.08 ^c	7.19±0.10 ^b	9.52±0.12 ^a
PV	3.53±0.80 ^b	3.46±0.57 ^{ab}	3.04±0.30 ^a	2.99±0.21 ^a	2.51±0.25 ^a	1.61±0.30 ^a
L	133.20±2.15 ^a	121.12±2.48 ^b	99.40±3.78 ^c	84.46±2.80 ^d	76.00±0.73 ^e	61.43±1.15 ^f
c	254.14±32.97 ^a	175.55±32.32 ^b	59.13±25.05 ^c	30.99±19.69 ^c	46.63±19.01 ^c	46.29±14.87 ^c
ΔE	0.00±0.00 ^e	82.41±0.83 ^d	221.56±2.08 ^c	287.72±2.48 ^b	301.29±2.82 ^a	302.73±1.99 ^a
Turbidity	0.03±0.03 ^f	7.13±0.73 ^e	13.92±2.04 ^d	24.88±1.50 ^c	45.70±1.50 ^b	57.90±1.65 ^a
Contact angle	64.18±0.51 ^a	64.00±1.58 ^{ab}	59.45±1.02 ^{bc}	57.88±1.19 ^{cd}	54.47±2.52 ^{cd}	53.56±0.71 ^d
TPM	7.50±0.42 ^d	7.13±0.02 ^d	7.76±0.16 ^{cd}	8.77±0.28 ^{bc}	9.76±0.51 ^b	12.00±0.21 ^a

FFA: (%), PV: meqO₂/kg. Contact Angle: °. Turbidity: NTU, TPM: %.

Mean ± SD (n = 3). Means within a row with the same letter are not significantly different as indicated by Tukey's HSD test (P < 0.05).

Table III - Correlation between contact angle and other physicochemical properties of chicken frying oils

	Contact angle	TPM	FFA	K232	K270	p-AV	PV	Totox	Turbidity
Contact angle	1.000								
TPM	-0.945	1.000							
FFA	-0.959	0.977	1.000						
K232	-0.968	0.888	0.912	1.000					
K270	-0.842	0.648	0.713	0.921	1.000				
p-AV	-0.976	0.874	0.897	0.991	0.931	1.000			
PV	-0.949	0.870	0.938	0.904	0.812	0.925	1.000		
Totox	-0.981	0.889	0.936	0.966	0.888	0.981	0.981	1.000	
Turbidity	-0.949	0.902	0.881	0.969	0.853	0.954	0.813	0.901	1.000

Table IV - Correlation between contact angle and other physicochemical properties of fish frying oils

	Contact angle	TPM	FFA	K232	K270	p-AV	PV	Totox	Turbidity
Contact angle	1.000								
TPM	-0.874	1.000							
FFA	-0.920	0.973	1.000						
K232	-0.889	0.836	0.917	1.000					
K270	-0.820	0.952	0.964	0.863	1.000				
p-AV	-0.960	0.965	0.987	0.927	0.939	1.000			
PV	0.900	-0.982	-0.958	-0.854	-0.950	-0.974	1.000		
Totox	-0.973	0.917	0.974	0.951	0.894	0.984	-0.917	1.000	
Turbidity	-0.934	0.959	0.995	0.939	0.965	0.992	-0.962	0.980	1.000

were found in both fish and chicken frying oils. Free fatty acidity values increased by almost twice for both oils and rose from 0.21±0.00 to 0.41±0.01 and from 0.29±0.01 to 0.58±0.01 for oils used in chicken and fish frying, respectively. The peroxide values prior frying (time zero) were 1.18±0.17 and 3.53±0.80, respectively for sunflower oils used for chicken breast and fish fillets. While the peroxide value of chicken frying oil increased until 210 min and decreased afterwards, it decreased linearly in fish frying oil through the entire frying process. It can be explained by the changes during oxidation, the formation of secondary products was higher in fish frying oil than in the chicken frying oil.

The L value decreased linearly in oil used in fish frying

until the 270th minute as previous studies [2], [18]. However, it decreased to 150 minutes of chicken frying process and increased between 150 and 270 minutes of frying. As the frying time went up, ΔE increased and the c value decreased for both oils.

The increase in the turbidity of oils with the frying time can be caused by high-molecular-weight compounds, including saturated triglycerides, sterols, waxes, fatty alcohols, free fatty acids and minor amounts of hydrocarbons, sterols or their esters [19]. Turbidity of sunflower oils used in both frying procedures linearly increased as the frying time rose, the turbidity of oils used in chicken and fish frying reached to 67.55±4.65 and 57.90±1.65, respectively at 270 minutes frying cycle.

Table V - Percentage fatty acid composition of sunflower oils

Fatty Acid	CoF	UoF	CoC	UoC
C16:0	7.14	7.98	6.10	7.59
C18:0	3.61	3.77	3.66	3.93
C18:1	38.23	39.02	38.67	37.04
C18:2	47.24	47.07	49.23	49.03
C18:3c	0.01	0.45	0.01	0.11
C20:0	0.28	0.29	0.08	0.03
C20:4c	0.61	0.69	0.11	0.35
COX	5.25	5.34	5.46	5.44
18:2/16:0	6.62	5.90	8.07	6.46
Totox Value	8.37	12.74	3.51	22.94

CoF: Control sunflower oil before fish frying. UoF: Used sunflower oil for fish frying (270 min). CoC: Control sunflower oil before chicken frying. UoC: Used sunflower oil for chicken frying (270 min).

FATTY ACID COMPOSITION AND COX VALUE

Change in the main fatty acid percentages and COX values between control oils and oils subjected to 270 minutes of frying are reported in Table V. The COX value of 270 minutes of fish fried oil increased by 0.95% and decreased by 0.36% in 270 minutes of chicken fried oils compared to unused oils. The decrease in the linoleic acid percentage of fish frying oil was lower than chicken frying oil. On the other hand, the increase in linolenic acid percentage was higher in fish fried oil. The fatty acid composition and the presence of phenolic compounds significantly affect the oxidative stability of oils [16]. The major fatty acids found in all oils were linoleic and oleic acids. The major fatty acids ranges were 47.07-49.23 and 37.04-39.02 for linoleic and oleic acids, respectively. The oleic acid (39.02%) was determined the highest in oil used for fish frying for 270 minutes that can be explained by a higher oleic acid content of fish. The increase in palmitic acid was observed slightly higher in chicken fried oil for 270 minutes than fish fried oil for 270 minutes. The % increase in palmitic acid in both oils can be explained by the relative decrease in unsaturated fats.

The oleic acid percentage decreased when frying time

increased in chicken frying, whilst it increased by 0.79% in fish frying. The linoleic acid decrease in sunflower oil after 4.5 h frying was similar to what was found after by Liu et al. [1] The concentration of linoleic acid decreased from 47.24% to 47.07% for frying chicken oils and from 49.23% to 49.03% for oils during fish frying. The linolenic acids levels ranged between 0.01% and 0.11% for chicken frying oil samples and increased from 0.01% to 0.45%, explained by omega 3 fatty acids contained in fish oils.

THE CONTACT ANGLE AND TPM

Aydar et al studied the contact angle of olive and Canola oils on a Teflon surface at high temperatures (20-200°C) and reported that the contact angle decreased with the increase in temperature. They determined that when the temperature rose from room temperature to the smoke point temperature, contact angles of Canola and olive oil decreased significantly ($p < 0.001$). It was found that there was a strong linear correlation between the contact angles of Canola oil and a temperature with an oil-steam and oil-air interfaces ($R^2 > 0.98$) [16].

The static contact angle of fresh sunflower oils used for chicken and fish frying were $62.22 \pm 0.35^\circ$ and $64.18 \pm 0.51^\circ$, respectively. They decreased to $57.85 \pm 0.89^\circ$ and $59.45 \pm 1.02^\circ$ and after 90 minutes of frying of chicken and fish, respectively, and these values were similar to contact angle values of canola oil found by Sahasrabudhe et al., [6]. The contact angle of oils used in chicken frying decreased by 23.78% when the frying time increased from 30 min to 270 min (Fig. 1).

Figure 2 shows the change of TPM and contact angles of oils during frying. The contact angles of oils decreased while TPM values of oils increased. This can be explained by increased wettability with the utilisation of frying oil for longer times [6]. Since liquids with higher contact angles have a lower wetting ability on the surface based on the wetting phenomena, smaller

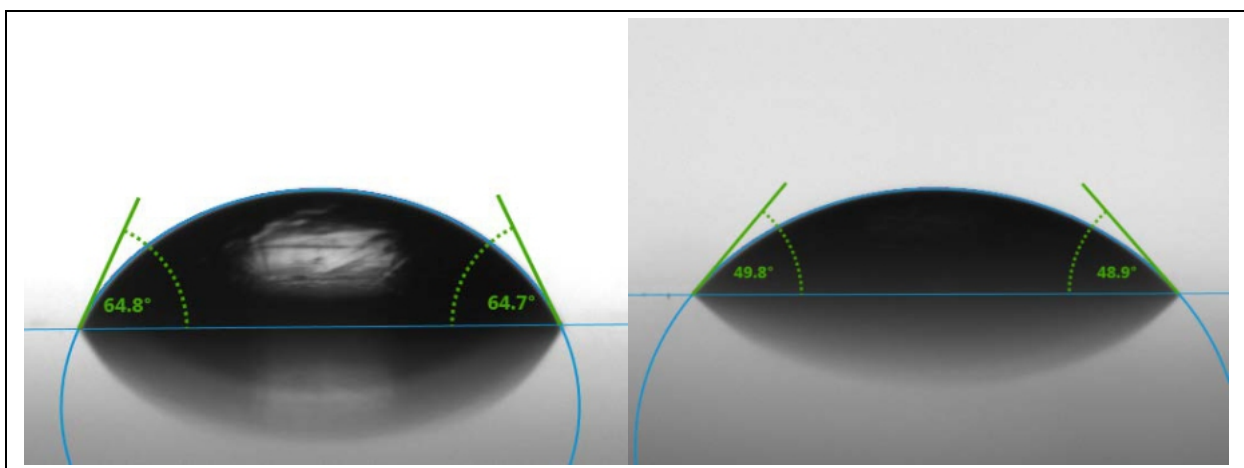


Figure 1 - Contact angles ($^\circ$) of oils used in chicken frying 30 minutes (a) and 270 minutes (b)

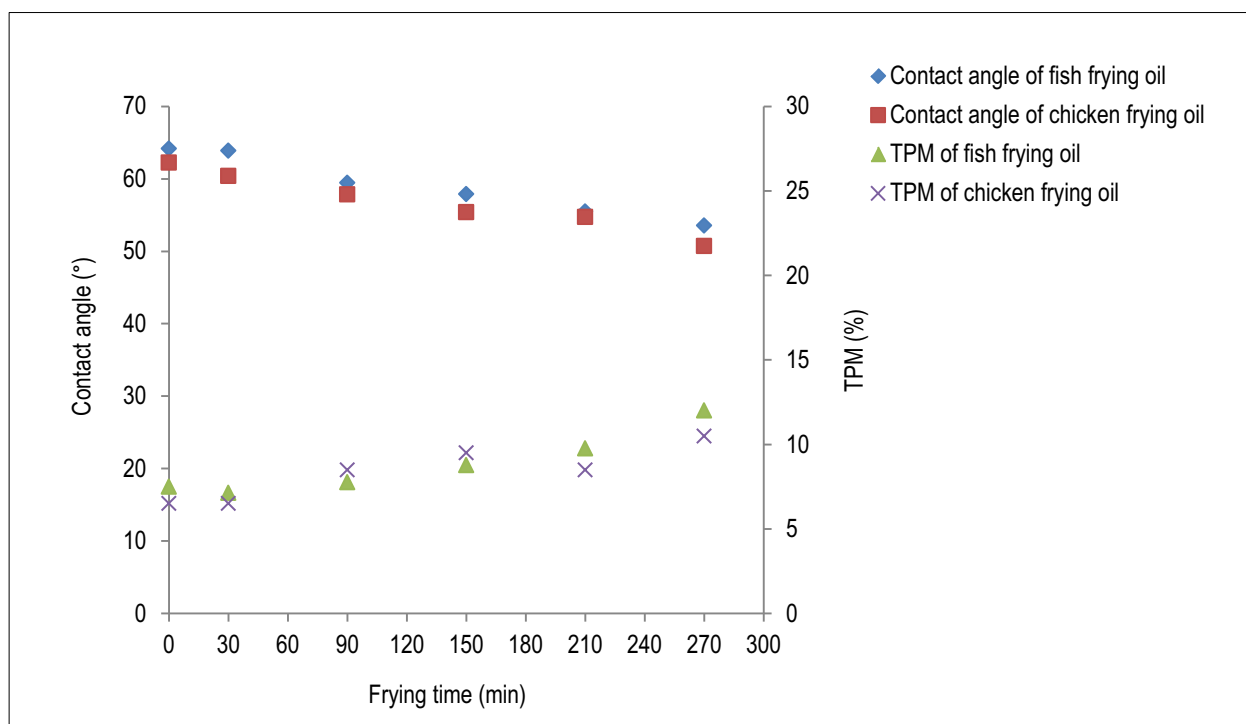


Figure 2 - Change in contact angles and TPM of oils during frying

contact angle values are important for the oil industry [3].

It was determined that there was a high negative correlation between the contact angle and all other physicochemical properties of chicken frying oil and shown in Table III. However, a positive linear relationship was found between peroxide value and the contact angle of chicken frying oils shown in Table IV. Pearson correlation coefficients were -0.842 and -0.981 for the contact angle and K_{270} and Totox values of chicken frying oil, respectively. They ranged from -0.820 to -0.973 for the contact angle and K_{270} and Totox values of fish frying oils. Correlation coefficients showed that the contact angle has a high relationship with other qualitative parameters of oils, thus it can be used as an oil degradation parameter.

CONCLUSION

The physicochemical (FFA, PV, p-AV, UV absorbance values, Turbidity, TPM etc.) and colour properties, fatty acid composition and contact angle changes of sunflower oil during different frying processes for 4.5 h were investigated. Utilisation of physicochemical properties of oils for oil degradation has been applied within the oil industry and by scientists for many years. In the following years, rapid tests are expected to continue to be implemented and improved, so that rapid detection of changes in the physical properties of oils during frying will be of interest. In this study, the correlation coefficients showed that the contact angle,

a quick and easy method, has a high relationship with other qualitative parameters of oils, thus it can be used as an oil degradation parameter.

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