

## Changes in physical and chemical properties of palm olein during heating.

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### RESUMEN

#### Cambios en las propiedades físicas y químicas de oleína de palma durante el calentamiento.

Este estudio evaluó algunos cambios ocurridos en oleína de palma doblemente fraccionada (DF) durante el calentamiento.

La oleína de palma se calentó a 180°C durante 60 horas (4 horas/día) sin adición de agua. Las muestras de aceite calentado se analizaron por: índice de refracción (IR), viscosidad, cambio de color, cambio en la constante dieléctrica (DCC), índice de acidez (AV), índice de peróxido (PV), índice de iodo (IV) y materia insaponificable (UM). Los resultados indicaron que, viscosidad, color, DCC y AV experimentan un incremento gradual, mientras que el IV disminuye con el aumento del tiempo de calentamiento. El estudio mostró también que los tiempos de calentamiento se correlacionan con los aumentos en DCC, viscosidad y AV. El índice de peróxido aumentó aproximadamente 17 veces, mostrando un máximo a las 16 horas, disminuyendo luego gradualmente y permaneciendo relativamente constante durante las últimas 20 horas de calentamiento.

El análisis cromatográfico ilustró que los componentes UM cambiaron cualitativamente y cuantitativamente debido al tratamiento por calor.

**PALABRAS-CLAVE:** *Calentamiento — Oleína de palma — Propiedades físicas — Propiedades químicas.*

### SUMMARY

#### Changes in physical and chemical properties of palm olein during heating.

This study evaluated some changes occurring in double fractionated (DF) palm olein during heating. DF palm olein was heated at 180°C for 60 hr (4hr/day) with no added source of water. Heated oil samples were analyzed for refractive index (RI), viscosity, colour change, dielectric constant change (DCC), acid value (AV), peroxide value (PV), iodine value (IV) and unsaponifiable matter (UM) constituents. The results indicated that, viscosity, colour, DCC and AV gradually increased, while IV gradually decreased with increasing heating time. The study showed also that heating times correlated with increases in DCC, viscosity and AV. PV increased ca 17-fold, reached a maximum at 16 hr, then gradually decreased to remain relatively constant through the last 20 hr of heating. Chromatographic analysis illustrated that UM components changed qualitatively and quantitatively due to the heating treatment.

**KEY-WORDS:** *Chemical properties — Heating — Palm olein — Physical properties.*

## 1. INTRODUCTION

In recent years, production of palm oil has increased up to a total world production of 11.88 million metric tons (1992) (1). Palm oil has become one of the leading oils for

edible purposes in Egypt and has been accepted by Egyptian consumers. Liquid fraction of palm oil is being used increasingly in frying operations in Egypt. Deep-fat frying is an important processing procedure used worldwide for preparation of foods. During deep-fat frying processing, oil is continuously subjected to high temperature in the presence of air and moisture. Under these conditions a variety of degradation reactions can occur such as autoxidation, isomer cyclization, thermal oxidation, thermal polymerization and hydrolysis (2)(3). Although a few papers have reported use of palm oil (4)(5)(6), data about the characteristics of palm Olein during heating and frying are not readily available in Egypt.

Therefore, this study was conducted to characterize DF palm Olein during heating by analyzing refractive index, viscosity, colour development, dielectric constant change, acid value, peroxide value, iodine value and unsaponifiable matter constituents.

## 2. MATERIALS AND METHODS

### 2.1. Materials

DF palm Olein was obtained from Misr Gulf Oil Processing Company (MIGOP), Suez, Egypt.

### 2.2. Heating Process

DF palm Olein was heated at 180 ± 20°C for 4 hr/day for fifteen consecutive days with no added source of water and oil samples were removed from fryer at the end of alternate days and kept for analysis.

### 2.3. Analytical Methods

Refractive Index, specific gravity, melting point, acid value, peroxide value, iodine value, and unsaponifiable matter were determined using AOCS Official methods (7).

Dielectric constant change was measured with food oil-sensor (FOS) model NI-20. Calibration with fresh oil and analysis were the same as in the manufactures operation manual.

Viscosity as a relative flow time (RFT) at 60°C was determined using Ostwald Viscometer (8).

Colour was measured by Lovibond Tintometer (model E) using a 5.25" cel. the absorbance as an index of colour development was also determined at 420 nm of a 50% (v/v) solution of the oil in isooctane using a Beckman spectrophotometer (2).

Unsaponifiable matter of fresh and heated oil were separated (7) and analyzed by a Hewlett-Packard gas Chromatograph model 5890 using a 25m x 0.2 mm I.D. fused Silica capillary column coated with dimethyl silicon fluid. The temperature programme was 100-280°C at 5°C/min, then oven temperature was held at the maximum temperature for 20 min. Peak area and retention times were determined using a Hewlett-packard 3392 A integrator. The identification of UM components were performed by comparing the relative retention time of each compound with those of authentic compounds.

Fatty acid methyl esters prepared (9) and analyzed by PYE Unicam gas chromatograph model 4550. The methyl esters were fractionated using coiled glass column (1.6 m x 4 mm I.D) packed with Chromosorb C and coated with 10% PEGA under the conditions mentioned by Rady et al., (1987) (10).

### 3. RESULTS AND DISCUSSION

The characteristics of fresh DF palm Olein used in this study are given in Table I.

Table I  
Characteristics of fresh DF Palm Olein used in Experiment

Characteristics of the Oil	
Refractive index 40°C	1.4587
Specific gravity 25°C	0.9248
Melting Point	22-24°C
Acid Value	0.06
Peroxide value (meq/Kg)	1.9
Iodine value	56.1
Saponification value	187.3
Unsaponifiable matter	0.49%
Fatty acids composition:	
C14:0	1.1%
C16:0	35.7%
C18:0	3.9%
C18:1	45.8%
C18:2	11.7%
Others	1.8%

Colour is an important physical characteristic of an oil. Table II and Fig. 1 shows the changes in the colour of DF Palm Olein during heating. It has been reported that heating and frying oils become darker as heating procedure (1). The results indicated that red colour gradually increased with heating time. The increases in red colour are characterized by three stages, the first one showed a slight gradual

increase and rose from 3.6 in fresh oil to 6.8 after 24 hr of heating the oil, followed by a noticeable gradual increase, and reached 22.4 after 40 hr of heating Olein. The final stage showed a sharp gradual increase and the red colour was 49.1 at the end of heating process. In addition, blue colour started to appear only after 44 hr of heating the oil and reached a value of 2.0 after 60 hr of heating DF Palm Olein. Furthermore, the absorbance as an index of colour development was also followed up during heating DF palm Olein Fig. 1. The absorbance at 420 nm increased from 0.15 in fresh oil to 0.35, 0.35, 0.5, 0.75, 1.6 and 3.0 after heating the oil for 4, 8, 16, 20, 40 and 60 hr, respectively.

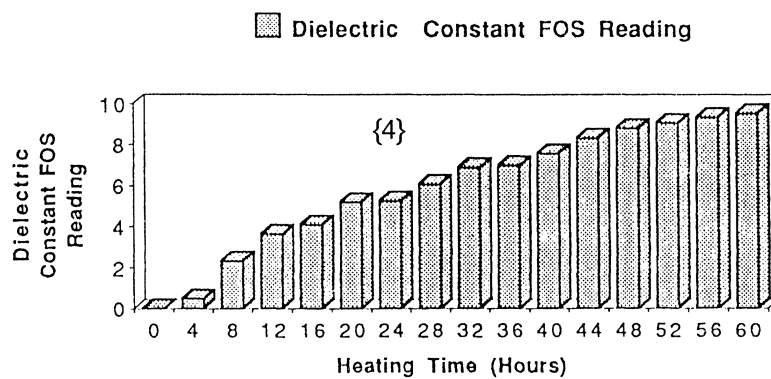
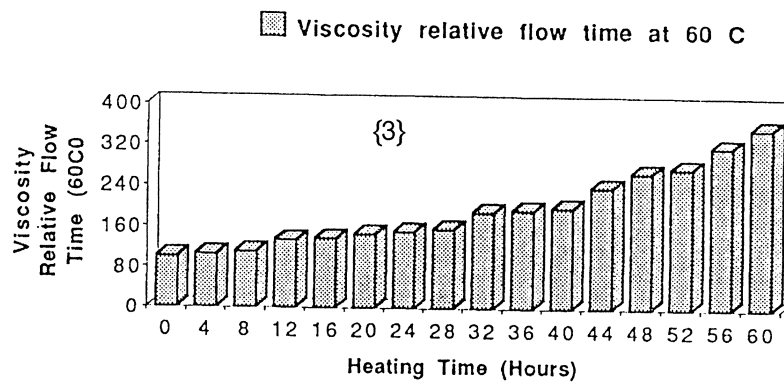
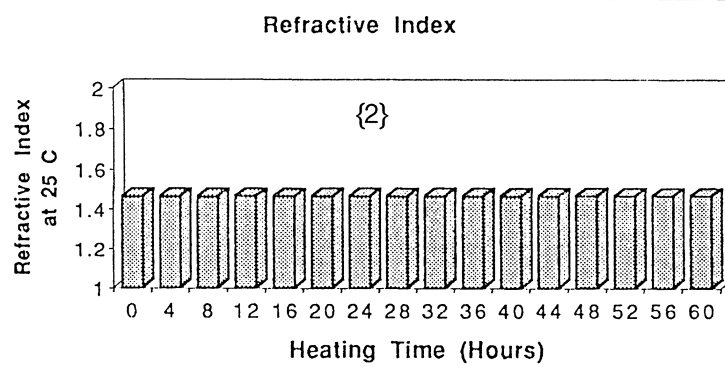
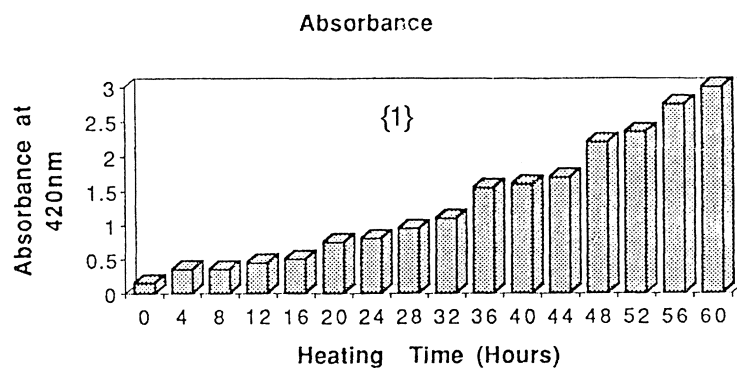
Table II  
Effect of Heating Time on the colour of DF Palm Olein

Heating Time (hr)	Yellow	Red	Blue	Absorbance at 420 nm
0	35	3.6	-	0.15
4	35	3.8	-	0.35
8	35	4.1	-	0.35
12	35	4.4	-	0.45
16	35	4.8	-	0.50
20	35	5.6	-	0.75
24	35	6.8	-	0.80
28	35	11.8	-	0.95
32	35	13.3	-	1.10
36	35	20.1	-	1.55
40	35	22.4	-	1.60
44	35	29.0	0.7	1.70
48	35	39.0	0.8	2.20
52	35	43.0	0.9	2.35
56	35	47.1	1.9	2.75
60	35	49.1	2.0	3.0

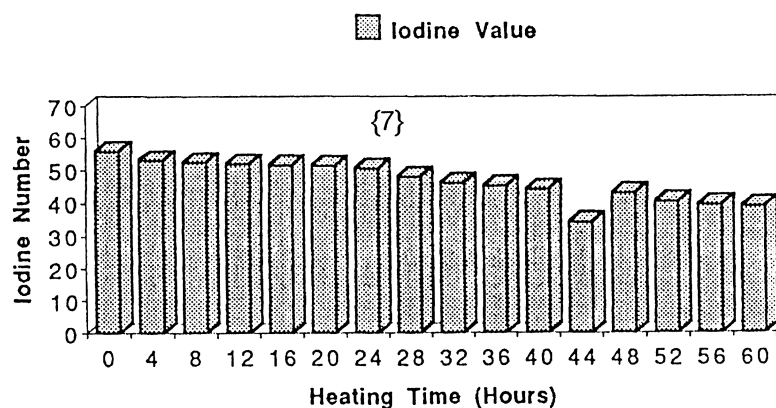
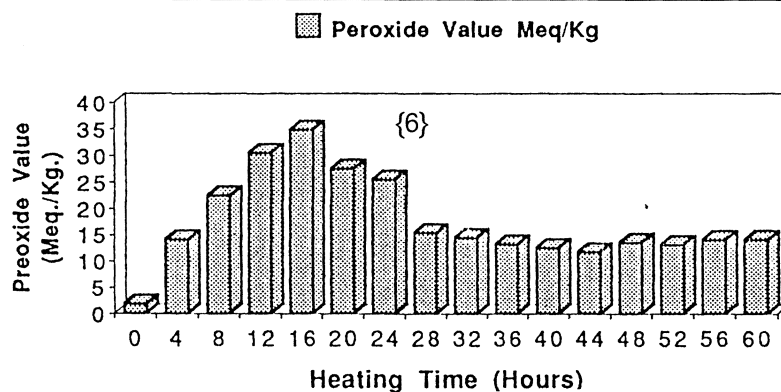
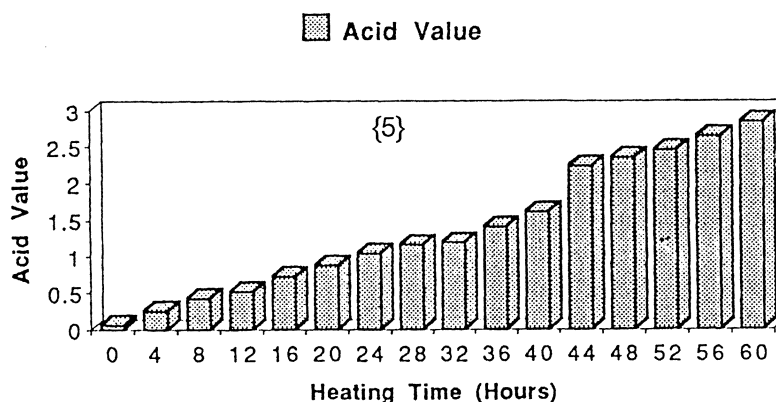
The changes in physical and chemical properties of DF palm Olein during heating are shown in Table III and Figures 2-7. It is obvious from these results that the refractive index cannot be used to follow up heating process of DF Palm Olein easily, as heating process induced a slight increase in the refractive index of the oil Fig. 2.

Table III and Fig. 3 reveal that, viscosity as a relative flow time at 60°C showed a gradual increase with heating time. The relative flow time increased to 105, 110, 136, 145, 198, and 354 after heating the oil for 4, 8, 16, 20, 40 and 60 hr, respectively.

A similar study has been done in Egypt on the effect of heating cottonseed oil on its relative flow time which reached 111, 124, 173, 187.2, 497 and 1272 after heating cottonseed oil at 180°C for 4, 8, 16, 20, 40 and 60 hr, respectively (12). It is obvious that the rate of increase in viscosity was much higher in cottonseed oil than DF palm Olein during heating process. The larger increase in viscosity of cottonseed oil compared to Olein is thought to be because more double bonds are present in cottonseed oil than Olein.



Figures 1, 2, 3 and 4



Figures 5, 6 and 7

Table III and Fig. 4 illustrate that dielectric constant change (DCC) also increased gradually at three stages, the first one started by a sharp increase after 8 hr of heating the oil, followed by a noticeable gradual increase till 20 hr, as DCC increased to 0.47, 2.3, 4.1 and 5.16 after heating DF Olein for 4, 8, 16 and 20 hr, respectively.

After 20 hr of heating, DCC increased constantly until 44 hr, but the rate of increase was lower than in the first period of heating. During the last 16 hr of heating the increase in DCC was relatively slight.

It is clear from the results in Table II and Fig. 5 that acid value showed also a gradual increase with the

ascending time of heating, as it increased from 0.06 in fresh oil to 0.25, 0.42, 0.51, 0.87, 1.61, and 2.84 after heating the oil for 4, 8, 16, 20, 40 and 60 hr of heating DF palm Olein. The same observation was also noticed in other study in Korea during heating the Palm Olein (11).

Peroxide value behaved differently at three stages, the first one showed a sharp increase and it increased ca 17 times (1.82) and showed a maximum at 16 hr of heating (34.8), then started to decrease gradually and reached 12.4 after 40 hr and finally remained relatively constant through the last 20 hr of heating the DF Palm Olein Fig. 6.

Table III  
Changes in Physical and Chemical Properties of DF Palm Olein During Heating

Heating Time (hr)	Refractive Index at 25 °C	Viscosity relative Flow time at 60°C	Dielectric Constant FOS Reading	Acid Value	Peroxide Value Meq/Kg	Iodine Value
0	1.4630	100	0	0.06	1.82	55.85
4	1.4630	105	0.47	0.25	14.01	52.92
8	1.4638	110	2.32	0.42	22.41	52.25
12	1.4640	133	3.62	0.51	30.44	51.71
16	1.4640	136	4.07	0.72	34.81	51.53
20	1.4648	145	5.16	0.87	27.47	51.22
24	1.4652	150	5.22	1.04	25.40	50.47
28	1.4658	155	6.05	1.16	15.28	47.85
32	1.4658	189	6.85	1.19	14.34	46.03
36	1.4658	193	6.97	1.41	13.09	45.06
40	1.4660	198	7.54	1.61	12.43	44.01
44	1.4660	237	8.29	2.23	11.59	34.09
48	1.4668	266	8.78	2.34	13.40	42.85
52	1.4670	275	9.04	2.45	13.019	40.29
56	1.4670	317	9.32	2.64	14.0	39.33
60	1.4670	354	9.52	2.84	13.97	38.72

It is well known that iodine value of oil decreases as heating time increases (2)(11) and the same trend was also observed during heating DF Palm Olein. It decreased from 55.8 in the untreated oil to 52.2, 51.5, 51.2, 44.0 and 38.7 after heating the oil for 8, 16, 20, 40, and 60 hr respectively Fig. 7.

Table IV Fig. 8 illustrate the unsaponifiable matter (UM) components of DF palm Olein before and after heating the oil at 180°C for 60 hr. These results indicated that heating process induced a dramatic changes in the unsaponifiable matter constituents of DF palm Olein.

Table IV  
Effect of Heating Process on Unsaponifiable Matter Components of DF Palm Olein

Components	Fresh DF Palm Olein	Heated DF Palm Olein For 60 hr
Hydrocarbons < C <sub>20</sub>	2.60	92.02
Hydrocarbons C <sub>20</sub> -C <sub>32</sub>	19.97	5.88
Total Tocopherols + Tocotrienols	15.05	0.75
Brassicasterol	2.75	–
Campesterol	12.06	–
Stigmasterol	7.52	0.37
B-Sitosterol	37.11	0.98
Fucosterols	2.25	–
Total Hydrocarbons	22.57	97.90
Total Tocopherols + Tocotrienols	15.05	0.75
Total Sterols	62.38	1.35

Total hydrocarbons markedly increased from 22.5% in fresh oil to 97.9% of UM of heated oil. Among hydrocarbons, the hydrocarbons less than C<sub>20</sub> showed a sharp increase due to heating process, they increased from 2.6% in fresh oil to 92.0% of UM of heated palm Olein. On the other hand, C<sub>20</sub>-C<sub>32</sub> hydrocarbons markedly decreased from 19.97% in unheated oil 5.8% of UM of heated one. The increases in <C<sub>20</sub> hydrocarbons might to due to either formation of these hydrocarbons during heating process or the degradation of the existing C<sub>20</sub>-C<sub>32</sub> hydrocarbons of UM in fresh DF palm Olein. It has been reported in our previous study (5) that DF palm Olein contained 73.24 mg/100 oil total tocopherols and tocotrienols. It is well known that tocopherols and tocotrienols are very sensitive to any kind of heat. These results indicated that heating palm Olein for 60 hr at 180°C has almost destroyed tocopherols tocotrienols, as they sharply decreased from 15.05% in UM of fresh oil to only 0.75% in UM of heated one. Moreover, heating process also destroyed most the sterols in UM of palm Olein, as total sterols showed an acute drop from 62.30% in UM of fresh oil to 1.3% of UM of heated one. Among sterols compounds, brassicasterol, campesterol and fucoesterol were completely disappeared, while stigmasterol and B-sitosterol sharply dropped from 7.25%, 37.1% in unheated oil to 0.37, 0.98% of UM of heated oil. Similar studies has been done in Egypt and they found that heating and frying processing caused dramatic changes in the UM of other several oils (12).

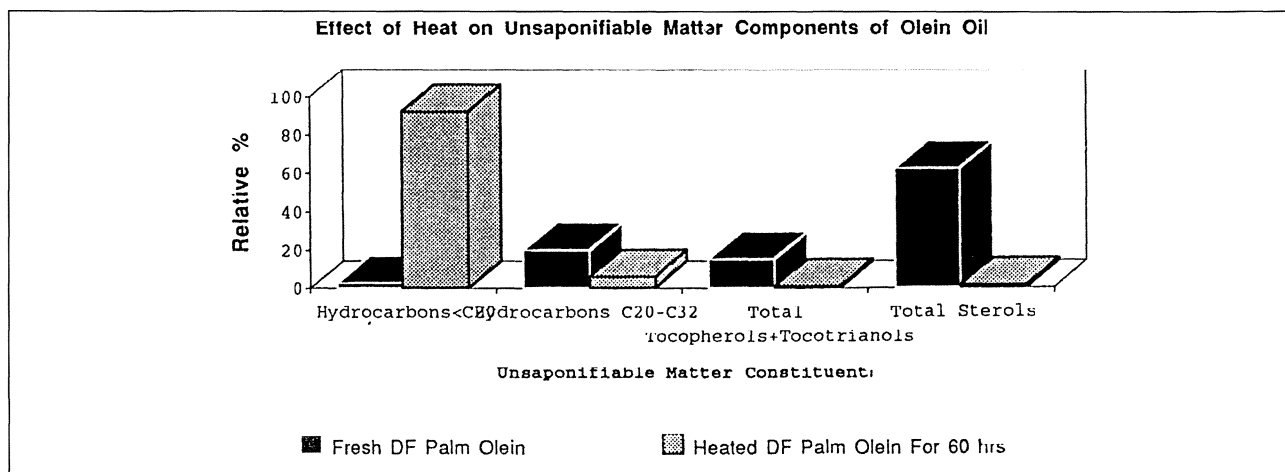


Figure 8

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