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Study of the physical and chemical properties and essential fatty acids of sunflower oil used for frying for a period of (1-5) hours

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ABSTRACT

A portion of edible sunflower oil obtained from the local market was obtained and split into six segments. One segment served as the control sample, while the other five segments were used for frying for a duration of five hours. At each hour, a sample was extracted for inspection. The study included conducting chemical and physical testing on all samples. Additionally, gas chromatography (GC) analysis was performed to track the concentrations of essential fatty acids (namely linoleic and linolenic acids) in four oil samples. The obtained findings are as follows: Following a duration of five hours of frying, the melting point experienced a reduction of 2°C, the specific weight underwent a change from 0.9178 to 0.9255, the viscosity exhibited a rise from 40.82 to 48.70 CBP, and the refractive index had a modest alteration, transitioning from 1.4722 to 1.4735. The chemical tests yielded the following results: the free fatty acid content did not exceed 0.6%, the smoking point was reduced from 285°C to 245°C, the peroxide number remained within the permitted range without any modification, and the acidity level met the required specifications. The levels of unsaturated fatty acids in the control sample were as follows: oleic acid decreased from 263 ppm to 0.173 ppm, linoleic acid decreased from 102 ppm to 8.784 ppm, and linolenic acid decreased from 339 ppm to 17.27 ppm. Unsaturated acids exhibit less stability throughout the course of frying. The stability of the oil is directly correlated with the duration of frying. In general, sunflower oil is considered one of the most suitable oils for frying and remains stable for up to 5 hours of frying. The oil's essential acid concentration drops as the frying time increases, in addition to its physical qualities.

1. INTRODUCTION

Sunflower oil output is globally listed as the third highest among oilseeds and the fourth highest among vegetable oils, trailing behind palm oil and soybeans. Sunflower oil maintains its position as one of the top oils derived from various crops due to its significant nutritional and industrial value. It is widely used in the food and confectionery sectors, as well as in the manufacture of biofuels and biomaterials (18, 33). The oil business produces by-products that include a significant quantity of proteins that are appropriate for use in food, animal feed, and aquaculture (33).

Sunflower oil is rich in vitamins, cholesterol-free, and has a delightful taste. Sunflower fields are viable sources of sustainable oil. Each year, agricultural land has the capacity to provide hundreds of metric tonnes of sunflower seeds that are rich in high-quality oil (1). Sunflower oil is widely regarded as one of the top choices for cooking due to its high content of essential fatty acids that promote bodily growth, as well as its abundance of fat-soluble vitamins E, D, A, and K, which act as natural antioxidants. Fats, comprising up to 40% of the total energy in food, are a significant source of energy (32).

Frying is a very prevalent cooking technique used in both households and enterprises due to its many benefits that enhance the palatability and desirability of fried meals (27). The process of frying involves heating and dehydrating food by immersing it in hot oil, resulting in a more enjoyable and flavorful culinary experience. Simultaneously, the high temperature during frying ensures food safety by eliminating microorganisms and enzymes while also reducing water activity on the food's surface. The shelf life of fried products is determined by assessing the moisture content after frying (35).

While frying, the oil undergoes chemical processes such as oxidation, hydrolysis, and polymerization due to exposure to moisture in food and oxygen at high temperatures (36). The process of over-frying causes the degradation of natural antioxidants in sunflower oil, such as tocopherols, and leads to the destruction of oil molecules. Decomposition, oxidation, and polymerization processes contribute to the formation of acrylamide in food. The presence of double bonds is directly

linked to the quantity of acrylamide. Oils with larger amounts of saturated acids tend to be more stable. Additionally, the level of acrylamide rises with repeated frying cycles, as shown in sweet potatoes (13).

Sunflower oil has a longer durability for pressure frying compared to air frying (30). Frying oil serves as both a medium for transferring heat and a generator of flavor, while also enhancing the nutritional value and texture. Fryers has desirable aesthetic, tactile, and sensory attributes that appeal to consumers. Nevertheless, the fried goods assimilate the oil used for frying. Hence, it is recommended to avoid reusing fried oil several times owing to changes in its composition. Edible oils play a crucial role in fried food preparation. Their significance lies in their impact on human health and nutrition. These oils serve as primary sources of essential fatty acids, including linoleic and linolenic acids, as well as fat-soluble vitamins (24). Therefore, it is essential to closely monitor and investigate their usage.

Frying leads to the degradation of vitamins and the suppression of enzyme activity, which may result in discomfort in the digestive system. To ensure that frying oil remains stable at high heat and oxidation, it should have a high smoke point and excellent heat conduction (15). The pace of degradation is influenced by the chemical composition of the oil, specifically the levels of saturated and unsaturated fatty acids, as well as the frying temperature and time. Additionally, the kind of frying process (continuous or intermittent) and the depth of the frying (deep or shallow) have a role, along with the quality of the food being fried (20). Among the significant chemicals that form during the degradation of oil are fatty acids, mono- and diglycerides, and polymers. These molecules are present in fried oil and are referred to as polar substances (21). The primary role of frying oil is to transfer heat to the food product, resulting in flavour enhancement and improved appearance and texture. Additionally, frying oil adds calories to the food due to its absorption. However, it is important to note that the frying process alters the chemical and physical composition of the oil, which limits its reusability. Prior to oil replacement, it is necessary to establish the frying cycles, as is done in commercial labs. This practice directly impacts the quality of the fried food that is preserved. The higher the quality of the oil, the safer it is to store fried food. During storage, the oil undergoes oxidation, resulting in the development of unwanted odours and a loss of flavour. This change is also accompanied by an imbalance in The oil undergoes chemical and physical changes with time, leading to a deterioration in its features. Consequently, the quality of the meal is adversely affected.

The quality of oils is assessed by their content of unsaturated acids. Oils with higher levels of unsaturated fatty acids are more stable. However, oils with low levels of unsaturated fatty acids have lower quality. Therefore, the quality of oils and fats varies depending on the quality of the fatty acids they contain. When oils break down, they create free fatty acids, di- and monoglycerides, polymers, and free radicals. The free radicals pose a risk to the consumer's health. Improperly produced oils include a proportion of free fatty acids that might lead to rancidity during storage (2).

The frying method involves submerging the meal in heated oil at a temperature ranging from 150 to 200 degrees Celsius in order to cook it thoroughly without causing it to become rigid or burnt. Additionally, individuals with hypertension are advised to decrease the oil level in their diet. One factor that decreases the quantity of oil in food is the frying technique, the quality of the fried items, their moisture level, and the quality of the oil used (14).

Fats are highly significant in nutrition as they supply the body with the majority of its energy, equivalent to that obtained from proteins and sugars. Additionally, fats transport fat-soluble vitamins and other essential compounds, playing a crucial role in human development. Hence, fats and oils used for consumption must adhere to specified criteria, including being devoid of any peculiar odours or rancidity and being pure and unadulterated with other oils. Adding ingredients to them other than those permitted by the Committee on Food, Agriculture, and World Health is strictly prohibited. Additionally, any additives used must comply with the specified guidelines. It is crucial that these additives are free from hydrogenation aids, as their presence can have detrimental effects on health (26).

When food is submerged in heated oil, the heat is delivered to the food by convection currents created by the surrounding oil. The oil then carries the heat to all portions of the food. The water undergoes evaporation and subsequently exits from the food's surface, while some solid food constituents dissolve and disperse inside the oil. As water evaporates from the food's surface, the oil seeps into the food, causing more water to evaporate and creating a crispy crust. This process also leads to various chemical changes in both the food and the oil, including sugar caramelization, starch gelatinization, protein denaturation, hydrolysis reactions, browning reactions, and alterations in physical properties like viscosity. Food colour and solubility, as well as chemical processes like oxidation, polymerization, and hydrolysis, may cause changes in the food system that ultimately alter the physical and chemical characteristics of lipids. Hence, a range of secondary products, including free fatty acids, alcohols, cyclic compounds, dimers, and polymers, are generated (25). Viscosity refers to a liquid's ability to resist spilling and is quantified in units of poise, which are equal to force divided by area multiplied by time (newtons per square metre times a second). The viscosity decreases as the amount of unsaturated fatty acids increases, and it increases through the process of hydrogenation. Additionally, viscosity increases with prolonged exposure to high temperatures (30 hours). The viscosity of oil is a measure of the internal friction of its particles, indicating the oil's reluctance to flow. The viscosity of fatty acids and triglycerides is contingent upon their quality, particularly the length of the chain in fatty acids and the level of unsaturation present (31).

Acidity is determined by titrating fatty acids with a 0.1-standard solution of potassium hydroxide. It represents the quantity

of milligrammemes of potassium hydroxide required to neutralise the fatty acids present in one gramme of oil. In purified and refined edible oil, the acidity should not exceed 0.6 mg of potassium hydroxide per gramme of oil. On the other hand, the peroxide number is a measure of the milliequivalents of peroxide per kilogramme of fat. This number represents the level of oxidation that the oil or fat is subjected to. Additionally, it serves as a rancidity test to assess the quality of the fat or oil (27).

The melting point refers to the temperature at which the fat transitions from a solid to a liquid form. It is observed that the melting point tends to rise as the length of the fatty acid chain grows, while it decreases with an increase in the degree of unsaturation (5). The smoking point refers to the minimum temperature at which visible smoke may be seen rising from the oil, given the suitable circumstances. The smoke is generated by the oxidation or decomposition of several substances, including free fatty acids, short-chain fatty acids, and reactions arising from oxidation and other processes. The smoke point of oils is strongly correlated with the amount of saturated fatty acids they contain. Covalent bonds need greater temperatures for their rupture in comparison to the temperatures required for the rupture of double bonds (22). Index of refraction: The refractive index of oils is the ratio of the speed of light in air to its speed in the medium (oil) at a certain temperature. The refractive index is a crucial physical property in the analysis of oils. The refractive index of oils is an indicator of their purity and quality. Specifically, it is observed that the refractive index rises as the temperature falls.

The objective of this research was to investigate the impact of frying on various chemical and physical characteristics of sunflower oil, as well as track the levels of essential fatty acids during a five-hour frying period.

2. METHODS AND MATERIALS

The materials and equipment utilised in the experiment include sodium thiosulfate, saturated potassium iodide, starch, chloroform, glacial acetic acid, sodium hydroxide, phenonphthalene, iodine, ethanol, dimethyl ether, gas chromatography (GC), Ostwald viscometer, refractometer, class (0-100°C and 300°C) thermometers, burette, flasks, a specific gravity measuring flask, and a sensitive balance. Collect and organize the samples.

A portion of sunflower oil (obtained from a local market) was used to fry the potatoes. The oil was divided into six segments for frying; the first segment served as the control sample (F0) and was not fried. The second segment (F1) was fried for one hour, the third segment (F2) was fried for two hours, the fourth segment (F3) was fried for three hours, the fifth segment (F4) was fried for four hours, and the sixth segment (F5) was fried for five hours. Subsequently, all segments were stored in the freezer until the tests were conducted.
Melting point determination: The melting point was determined by immersing a capillary tube in the oil until the oil column reached a height of 1 cm (±0.2). The oil column was then solidified in the tube for a minimum of 16 hours. A rubber belt was used to connect the tube to the thermometer, ensuring that the tube was aligned parallel to the mercury deposit. Subsequently, the thermometer was suspended in the centre of a beaker holding 350 ml of water, which had a temperature of around zero degrees Celsius. The bottom of the thermometer was positioned 30 mm above the water's surface. Gradually increase the temperature of the cup while stirring, ensuring that the temperature rises at a pace of 2°C per minute. Observe the temperature at which the oil column starts to climb in the capillary tube. The temperature was measured as the melting point, which was reported as 22 degrees.

Density relative to the density of a reference substance: The specific weight of the oils was determined using the technique described in reference (22). This included measuring the weight of a specified amount of oil (25 ml) in a pycnometer at a temperature of 25 °C. The weight was then divided by the weight of the same volume of distilled water at the same temperature, as shown in the equation:

$$\text{Specific weight of oil} = \frac{wt\ (25ml) of\ oil}{wt\ (25ml)\ of\ water}$$

Viscosity estimation: The viscosity was calculated by measuring the time it took for the fluid to flow through a capillary tube over a certain distance. The duration for the liquid to travel between two specified points on the Ostold device was approximated. Subsequently, the device was thoroughly cleaned and filled with a comparable volume of water. The time it took for the water to descend between the same two points in the device was measured. Finally, using equation (9), the viscosity of the liquid was determined at the identical temperature.

$V = \frac{d1 * t1}{d2 * t2}$: Where: V = viscosity , d_1 = density of the liquid whose viscosity is to be tested.

d_2 = density of water , t_2 = time of flow of water between the two points in the viscometer.

The refractive index of oil was determined at a temperature of 25°C using a refractometer. The prisms were cleaned with cotton soaked in ether. Drops of oil were then placed on the prisms, and the gate of the refractometer was closed. The device was positioned towards a light source, and the refractive index was read through the eyepiece. The refractive index was identified as the line separating the shadow area and light, measuring (9).

The peroxide value (PV) of oil samples was determined using the method proposed in (11). Firstly, 5 g of oil samples were

dissolved in a mixture of solvents consisting of 60% acetic acid and 40% chloroform, with a volume of 30 ml. Next, 0.5 ml of saturated potassium iodide solution was added, followed by continuous stirring for exactly two minutes. Then, 30 ml of distilled water and 0.5 ml of a 1% starch solution were added. The resulting mixture was crushed and vigorously shaken with a 0.01 N sodium thiosulfate solution until the yellow colour disappeared. Finally, the peroxide value was calculated based on the equivalents. For every 1000 grammes of oil, according to the above equation:

PV(mEq / kg of oil)= $\frac{V*N*1000}{Wt\ of\ sample}$, whereas: V = volume (ml) of sodium thiosulfate consumed per flush , N = normality of sodium thiosulfate solution .

Quantification of the proportion of unbound fatty acids (FFA): 2 g of oil (11) was weighed in a clean flask, then 20 ml of a 1:1 solvent combination of ethanol and diethyl ether was put into it, then 4:3 drops of phenolphthalein proof were added and titrated with alcoholic KOH until a faint pink hue showed. (FFA%) = $\frac{100*282*N*(KOH)V}{W*1000}$

Acidity % = $\frac{100*N*(KOH)V}{W}$, Where: N = base normality , V = base volume , W = sample weight.

Smoking point temperature: A volume of 25 ml of oil was added to a 100-ml beaker and heated slowly while monitoring the thermometer until the presence of smoke-like threads became visible. The smoking point temperature was determined as the point at which the oil started to degrade and smoke was continuously emitted (3).

Estimation of essential fatty acids: Samples To, T3, T4 and T5 were used in the GC device to calculate the area (A) of the samples based on standard samples. The concentration for each of the fatty acids was deduced as in the equation below:

In the GC device (DANI-MASTER GC) use column DN10, column length (500) mm and mobile phase (ACN/FORMIC 50:50 with 0.1% phosphoric acid, flow rate 1 ml/min and (FID: detector) and the temperature (150°C-220°C at a rate of 1.5 minutes/sec and a pressure of 4psi. The acid concentration is calculated according to the following equation: $C_{sample} = \frac{A\ sample * C\ stander}{A\ stander}$ whereas : C=concentration, A=area, C stander=10ppm.

As for Wang et al. [34], it was based on the use of improved (HPLC) technology and the use of standard samples of essential fatty acids in the measurement of essential fatty acids in the oil.

Results and discussion

Physical tests of sunflower oil

The physical tests of sunflower oil used for frying for five hours at a temperature of 180°C are shown in Table 1. Compared to the control sample, it was seen that the melting point declined after the completion of the first and second hours of frying. The melting point remained steady thereafter but decreased to 6°C after the third hour. Subsequently, it dropped to 5 p.m., and during the fourth and fifth hours of frying, it further lowered to 4 °C.

Table (1) Results of physical tests for sunflower oil.

Samples	Melting Point	Specific Weight	Weight Viscosity	Refractive Index
Control	6°C	0.9178	40.82	1.4722
fry for an hour	6°C	0.9163	43.90	1.4734
Fry for 2 hours	6°C	0.9209	45.71	1.4735
Fry for 3 hours	5°C	0.9221	46.62	1.4733
Fry for 4 hours	5°C	0.9241	47.61	1.4735
Fry for 5 hours	5°C	0.9255	48.70	1.4735

The reduction in the melting point suggests a change in the molecular structure of the oil. According to White (38), the melting point of short-chain fatty acids decreases and they become less hardening as their fat content increases and the number of unsaturated bonds in them increases. The melting point serves as an indicator of purity, and it decreases as the duration of heating increases. Additionally, the melting point of oil is greater when it is more pure, but it falls if a mix contains many types of oil. [19,27].

Regarding the specific weight, we see a distinct rise in the value of the specific weight of the samples subjected to frying, which continuously increases with the duration of frying. The initial specific weight of the oil in the control sample was 0.9178. After the first hour of frying, it increased to 0.9163. Subsequently, the specific weight of the oil rose to 0.9209 after the second hour, 0.9221 after the third hour, 0.9241 after the fourth hour, and 0.9255 after the fifth hour. This increase in

specific weight may be attributed to bond breakage, hydrogen saturation, or oxygen linkage. Hassoun (16) claimed that the oil's density rises as the frying time lengthens. The oil's standard specification indicates that the specific gravity should range from 0.918 to 0.923 at a temperature of 20 degrees Celsius. [16,27].

Additionally, the viscosity prior to the commencement of frying was approximately 40.82 centipoise (CBP). Subsequently, it escalated to 43.90 CBP after the initial hour of frying, further increasing to 45.71 CBP after the second hour, then to 46.61 CBP after the third hour, followed by 47.61 CBP after the fourth hour, and finally reaching 48.70 centipoise after the fifth hour. It is evident that the viscosity increased by 7.54% after the first hour of frying. Subsequently, the viscosity continued to rise significantly with each subsequent hour of frying, particularly after the second, third, fourth, and fifth hours. Overall, after the fifth hour, the viscosity increased by 19.30% compared to the control sample. This indicates Nevertheless, the process of frying has adversely affected the chemical structure of the oil. Al-Ali and Al-Halfi (4) have observed a substantial rise in the viscosity of the oil as the frying duration progresses. Additionally, the viscosity increases by 12% when including additional oils, such as ginger oil, in the frying oil.

Refractive index: The results showed in Table (1) that the refractive index increased with the increase in heating time, as the refractive index was 1.4722, and it maintained its stability after the first hour, then increased after the second, third, fourth and fifth hours to reach 1.4735 after five hours of frying. This rise may be due to the oxidation of the oil and the saturation of the unsaturated bonds in the fatty acids or the occurrence of polymerization of the oil and an increase in the specific weight, as mentioned earlier in the discussion of the specific weight. The Gulf specification for edible oil has specified that the refractive index is between 1.461-1.468. The refractive index of oils is used as a criterion for identifying the quality of oils. The refractive index is related to the degree of unsaturation, and the low refractive index of the oil is evidence of a decrease in the amount of unsaturated fatty acids [3,10]. In general, these results indicated that the refractive index was affected by an increase in the frying period. And that the oil is still within the validity of use.

3. CHEMICAL TESTS OF SUNFLOWER OIL

3.1 Free fatty acid results

Figure (1) displays the proportion of unbound fatty acids in sunflower oil used for frying during a period of (0-5) hours. The figure demonstrated that the oil saw a rise in the proportion of unbound fatty acids as the frying time increased. Specifically, in the fifth hour, the oil reached a level of 0.539 millilitres equivalent per kilogramme of oil. The control sample yielded a concentration of 0.310 milliequivalents per kilogramme of oil, which is the outcome of the oil undergoing thermal breakdown in the presence of water. The study (5) determined that prolonged frying duration and elevated temperatures are causative factors for fat cracking and unsaturated fatty acid oxidation. The guideline also mandates that the proportion of unbound fatty acids must not above 0.6 (15).

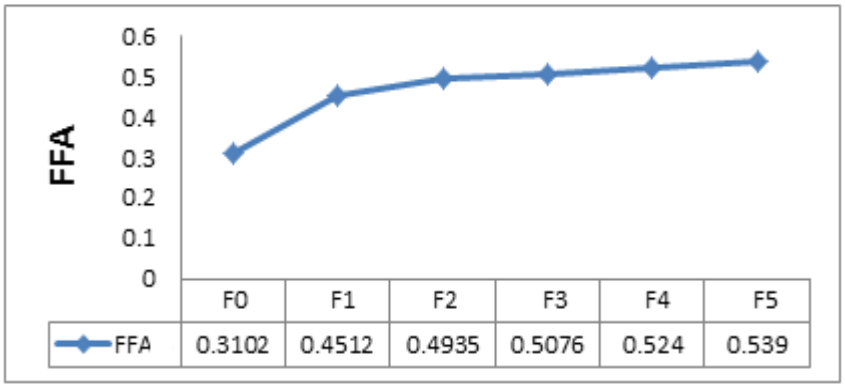

Figure (1) The percentage of free fatty acids during 5 hours of frying.

3.2 Smoking point

Figure (2) shows the smoking point temperatures for different oil samples. In the control sample, it was found that the smoking point was 285°C, which indicates that the high-quality oil, after an hour of frying, decreased to 280°C and after two hours it decreased to 260°C until it reached after the third hour to 245°C and the fourth hour 230°C. The fifth is 220°C and within the permissible limit, which is that the smoking degree of frying oils is not less than 185°C [15].

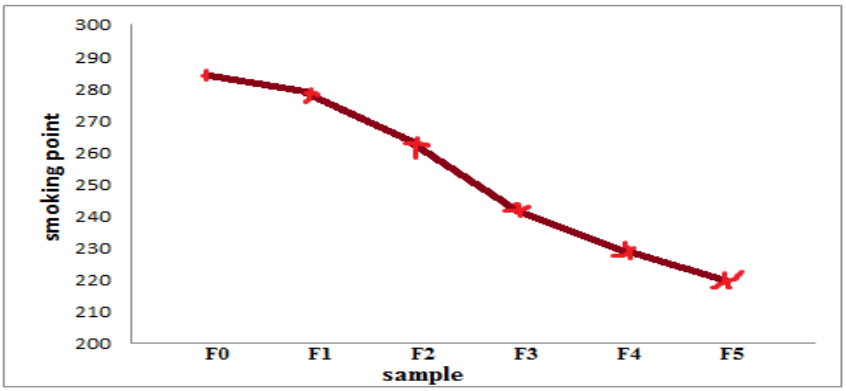

Figure (2) Smoking point of sunflower oil samples.

3.3 Peroxide value

The results showed that the peroxide value was 2.5 mg/kg for the control sample and for the flower oil samples used in frying, so the peroxide value did not change in all samples and was within the permissible limit, and that all the sunflower oil samples were stable during the frying period and did not differ from the control sample and thus this The samples were stable in terms of peroxide number and no oxidation occurred [(15)].

3.4 Acidity

The results showed in Figure (3) that the acidity did not change much from the control sample, but there was a clear change during the first and second hour, and it increased more in the third, fourth and fifth hours, which are within the permissible limits [(15)]. Some of the fatty acids, due to the heat and water in the fried pieces, decomposed, which caused the acidity to rise.

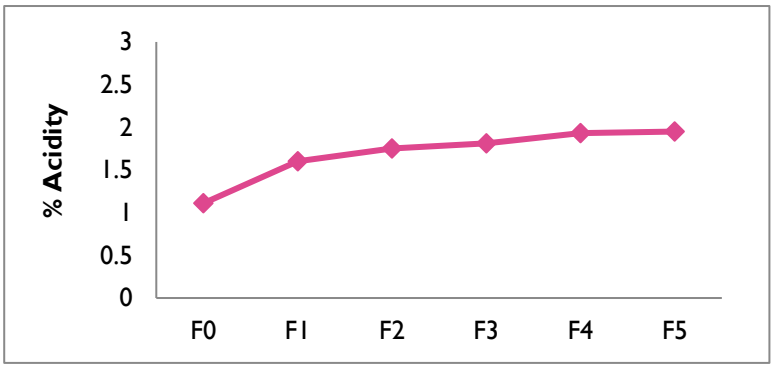

Figure (3) The development of acidity during five hours of frying.

3.5 HPLC test results for unsaturated fatty acids

The results showed in Table No. (3) that the proportion of essential fatty acids varied from one acid to another, as the proportion was high in the control sample and decreased in the research samples with the hours of frying for all essential acids, as in the above argument, and it was found that oleic acid had a concentration of 263 ppm in a sample. Control, after three hours of frying it became 71 ppm, after four hours it became 8.38 ppm, at the end of the fifth hour the concentration was 0.173 ppm, as for linoleic acid its concentration in the control sample was 102 ppm, after three hours the concentration became 81 ppm, then it decreased to 31.69 ppm, then At the end of the fifth hour of frying, the concentration was 8.784ppm, as well as the linolenic acid in the sample was 339ppm. After three hours of frying, its concentration became 187.73ppm, then it decreased to 85.69ppm after four hours and to 17.27ppm at the end of the fifth hour, and the latter is more.

Table No. (3) Results of HPLC analysis of essential fatty acids.

Sample	Aerea palmetic	Aerea Oleic	Aerea Linoleic	Aerea Linolenic
(Stander sample C 10ppm)	77.26	333.73	149.80	776.08

Ao	116.0	263.00	102.00	339.00
A4	72.96	71.00	81.00	187.73
A5	4.07	8.38	31.69	85.70
A6	0.28	0.17	8.784	17.27

The second option had a higher degree of stability compared to the first two. Typically, we see that unsaturated acids exhibit lower stability as the duration of frying increases, and stability is directly correlated with the frying time. Simultaneously, other unsaturated acids also degrade, such as palmiteic acid, whose content decreased from 116.00 ppm in the control sample to 0.277 ppm after five hours of frying.These findings suggest that the oil content of the essential fatty acids significantly decreases after 5 hours of frying. Numerous bioactive substances exist, such as prostaglandins and leukotrienes. Eicosanoids play a crucial role in the regular metabolic functioning of cells and tissues, and they also enhance the body's ability to fight several chronic illnesses, including infections and cancer (8). Insufficient linoleic acid in babies may result in the development of scaling, lesions, growth retardation, changed patterns of fatty acids in the blood, and thrombocytopenia (12).

4. CONCLUSIONS

Sunflower oil is one of the best oils used for frying and is suitable for frying within 5 hours of frying. The physical qualities are greatly affected with the hours of frying. Chemical properties were affected less than physical properties. The essential acid content of the oil decreased with the frying hours.

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