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Physicochemical Characterization of Samples of Vegetable Oils Traded in Local Markets

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دراسة الخصائص الفيزيوكيميائية لعينات من الزيوت النباتية المتداولة في الأسواق المحلية

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Abstract:
In this study, several physical and chemical properties were estimated (density, viscosity, moisture content, acidity, saponification number, and ester number) for various vegetable oil samples available in the Libyan local market. The results indicated no differences in the density and viscosity values of the corn oil samples, although differences were observed among the sunflower oil samples. Regarding moisture content, the highest value was found in corn oil (Al-Safwa) at 8.2%, while the lowest was in corn oil (Alhadayiq) at 30.18%. As for the chemical properties assessed, the acidity and saponification values of all samples did not meet standard specifications; the standard acidity is 0.6 mg/g, and the standard saponification number ranges from 187 to 194 mg/g. The acidity values varied from 2.805 mg/g for corn oil (Alhadayiq) to 3.703 mg/g for sunflower oil. The saponification number was lowest in the sunflower oil sample at 31.76 mg/g and highest in the corn oil sample at 68.80 mg/g. Additionally, the ester number values were also out of specification, with the lowest value for sunflower oil (Alzuhra) at 28.22 mg/g and the highest value for corn oil (Almarwa) at 65.83 mg/g.

Keywords: Sunflower Oil; Corn Oil; Physicochemical Analysis.

الملخص
في هذه الدراسة، تم تقدير بعض الخواص الطبيعية والكيميائية (الكثافة، اللزوجة، نسبة الرطوبة، رقم الحموضة، رقم التصبن، ورقم الاستر) لبعض عينات الزيوت النباتية المتوفرة في السوق المحلي الليبي. وبيّنت النتائج المتحصل عليها أنه لا يوجد اختلاف في قيم الكثافة واللزوجة لعينات زيت الذرة، مع وجود بعض الاختلافات في عينات زيت دوار الشمس. أما بالنسبة لمتغير نسبة الرطوبة، فكانت النتائج متقاربة، حيث كانت أعلى قيمة لزيت الذرة (الصفوة) 8.2%، وأقل قيمة لزيت الذرة (الحدائق) 30.18%. فيما يخص بعض الخواص الكيميائية التي تم تقديرها، فقد كانت قيم رقم الحموضة ورقم التصبن لجميع العينات غير مطابقة للمواصفات القياسية، حيث إن رقم الحموضة القياسي هو 0.6 *mg/g* ورقم التصبن القياسي يتراوح بين 187-194 *mg/g*. وقد تراوحت قيم رقم الحموضة بين 2.805 *mg/g* لزيت الذرة (الحدائق) وأعلى قيمة لزيت دوار الشمس (3.703 *mg/g*). أما أقل قيمة لرقم التصبن فقد كانت 31.76 *mg/g* في عينة زيت دوار الشمس (الزهرة)، في حين كانت أعلى قيمة له في عينة زيت الذرة (المروة) بقيمة 68.80 *mg/g*. كما كانت قيم رقم الاستر غير مطابقة للمواصفات، حيث كانت أقل قيمة لزيت دوار الشمس (الزهرة) 28.22 *mg/g* وأعلى قيمة لزيت الذرة (المروة) 65.83 *mg/g*.

الكلمات المفتاحية: زيت دوار الشمس، زيت الذرة، التحاليل الفيزيوكيميائية.

Introduction

Oils and fats have numerous applications in the food industry and food product manufacturing. Due to their economic significance, some individuals may resort to dishonest practices involving these products [1]. Vegetable oils are extracted through mechanical expulsion or solvent extraction from oleaginous seeds (such as soybeans, corn, and sunflower) or fruits (such as palm and olive) [2]. These oils typically consist of approximately 98g of triglycerides per 100g [3]. Sunflower seed oil is among the richest vegetable oils, with triglycerides accounting for 98–99% of its composition. It also contains small amounts of phospholipids, tocopherols, sterols, waxes, and palmitic acid. Sunflower oil is abundant in unsaturated fatty acids, contributing to energy efficiency, and it contains lecithin, which may help lower cholesterol levels [4,5] . Nutritionally, sunflower oil is significant because it provides essential fatty acids, particularly linoleic acid, and has a lower palmitic acid content compared to other oils. Triglycerides are triesters formed by the interaction of glycerol and fatty acids, along with other minor components.[6] Some of these compounds, including diglycerides, vitamins, phytosterols, tocopherols, and polyphenols, offer notable health benefits for humans [7,8].

Therefore, they should not be removed during processing. In contrast, components that negatively impact oil quality and stability include free fatty acids, unsaponifiable materials, waxes, colors, solid impurities (primarily fibers), and oxidation products (such as peroxides, aldehydes, ketones, alcohols, and oxidized fatty acids).[9] While these molecules are not toxic, their presence in oils is undesirable, as they affect both stability and consumer sensory acceptance. These compounds can impart off-flavors and odors to the oil, compromising its beneficial qualities [10-12]. Currently, there is global pressure from consumers and the food industry regarding the quality of refined oil to meet established food safety standards [13,14]. The oil must be odorless, neutral in taste, clear, and colorless, and it must be free of contaminants.[11] The process that removes undesirable and toxic components from crude oils is known as refining. Refining is often necessary for crude oils that cannot be consumed as virgin oils, as it results in a product with an appealing appearance, a neutral taste, and increased resistance to oxidation. Furthermore, refining produces oils that are more suitable for various industrial applications and eliminates unwanted substances such as pesticide residues, metal traces, polycyclic aromatic hydrocarbons, dioxins, and degradation products, while minimizing oil loss during processing.[12] Although refining extends the shelf life of oil, it has several disadvantages. One major drawback is the loss of beneficial substances that contribute to the oil's healthy and pharmaceutical properties, as well as their technological appeal. These substances include tocopherols, phospholipids, squalene, polyphenols, and phytosterols [7, 15]. Additionally, refining can lead to the formation of undesirable compounds such as glycidyl esters, 3-MCPD esters [16], harmful trans fatty acids [17], and polymeric triacylglycerols [18]. These compounds can directly impact the safety of refined oils.

To assess the quality and safety of vegetable oils sold in local markets, physicochemical characterization is essential. Numerous studies have focused on evaluating the physicochemical characteristics of edible oils available in nearby marketplaces. For instance, a study in Ghana highlighted the importance of trace metal analysis and physicochemical parameters in assessing the quality of edible vegetable oils [19]. Similarly, research conducted in Bangladesh examined the physicochemical qualities of cooking oils sold in local markets [20]. These studies underscore the critical need to analyze the chemical composition of vegetable oils to ensure consumer safety. An additional crucial aspect of determining the quality of vegetable oils is the assessment of trace metals. A study in Dedougou found that cottonseed oil samples tested positive for mineral oil [21].

Moreover, research in Nigeria emphasized the significance of microbiological quality evaluations by focusing on the mean aerobic plate count of palm oil sold in various markets [22]. These results highlight the necessity underscore the importance of thoroughly examining the physicochemical and microbiological properties of vegetable oils sold in regional marketplaces. Another key factor in determining the quality of vegetable oil is consumer perception. A study on customer perception and the physicochemical characteristics of crude oils highlights the need to dispel common misconceptions about various oil brands available on the market [23]. Vegetable oils sold in local markets can achieve higher overall quality if consumer tastes and concerns are better understood. Several studies have examined the quality of vegetable oils. Agaev et al. (2020) studied the dielectric spectroscopy of vegetable oils [24]. Sampson (2020) assessed the quality attributes of commonly consumed oils in Ghana, noting the high degree of unsaturation indicated by elevated iodine values. Beneito-Cambra et al. (2020) reviewed the direct analysis of olive oil and other vegetable oils by mass spectrometry, emphasizing the importance of analytical methods in determining oil quality.[25] Aidarkhanova et al. (2021) assessed the quality and food safety of vegetable oils produced in Kazakhstan, focusing on sunflower seed and flax oils obtained through cold pressing [26] . Zhang et al. (2020) discussed the significance of aroma in unrefined vegetable oils, particularly condiment oils, and the modulation of aroma compounds to improve acceptability [27]. To ensure consumer safety and uphold quality standards, it is essential to physiochemically characterize samples of vegetable oils traded in local marketplaces. By examining physicochemical parameters, researchers can provide valuable insights into the quality of edible oils sold locally [28]. To continue improving the standard and safety of the vegetable oils consumed by the public, further research in this field is necessary.

Material and methods

In this study, ten different samples of cooking vegetable oils (5 sunflower oil, and 5 corn oil) were randomly collected from markets in Tarhouna- Libya. Physical and chemical properties (specific gravity, moisture content, viscosity, pH, saponification number, ester number) were determined. The following figure (1) shows the samples of vegetable oils studied:

Figure 1: Studied Samples of Vegetable Oils.

Table 1: Studied Samples of Vegetable Oils.

Key No.	Sample	Manufacturer	Shelf life
1	Alhadayiq - Sunflower Oil	Tripoli - Libya	F:28/11/2023 E:28/11/2025
2	Prima - Sunflower Oil	Tobruk, Libya	F:4/1/2024 E:3/1/2025
3	Alzuhra - Sunflower Oil	Turkey	F:1/3/2024 E:1/3/2026
4	alkhalij- Sunflower Oil	Turkey	F:24/2/2024 E:24/2/2026
5	Carthage-Sunflower Oil	Turkey	F:1/3/2024 E:1/3/2026
6	Al-Safwa - Corn Oil	Turkey	-
7	Al-Baraka - Corn Oil	Tripoli - Libya	F:14/11/2023 E:13/11/2025
8	Alzuhra - Corn Oil	Turkey	F:24/8/2023 E:24/8/2025
9	Almarwa - Corn Oil	Turkey	F:26/1/2024 E:26/1/2026
10	Alhadayiq - Corn Oil	Tripoli - Libya	F:27/08/2023 E:27/08/2025

Physical Measurements:

physical constants determine the type and purity of the oil, and since oils are not naturally homogeneous substances because they contain many fatty acids and triglycerides, they are always within certain limits, not a fixed number, but are called oil constants.

Measurement of Relative Density:

Relative density is determined practically using a density bottle, by taking a certain volume of oil until the bottle is full, weighing it, and recording the density. The density is calculated by the following equation:

$$d_{40}^{20} = d_{40}^{t} + (t - 20) \times 0.00068 \qquad (1)$$

Estimation of moisture and volatiles in oil:

The percentage of moisture and volatiles is calculated from the following equation (2):

$$\frac{w3 - w2}{w1} \times 100 \qquad (2)$$

Wherein:

W1: weight of crucible, w2: weight of crucible and sample before drying, w3: weight of crucible and sample after drying.

Measurement of Viscosity

Viscosity is the resistance encountered by a layer of liquid as it flows against another layer (thus a measure of how fast a liquid flows under certain forces), as all liquids exhibit a certain resistance to flow. The viscosity is calculated from the following equation (3):

$$n_1 = \frac{n_2 \times d_1 \times t_1}{d_2 \times t_2} \qquad (3)$$

where:

n_1: viscosity of the oil in Poise at lab temperature, n_2: viscosity of distilled water at lab temperature.

t_1: Oil flow time from point A to point B, t_2: Distilled water flow time from point A to point B.

d_1: Oil density, d_2: Density of distilled water.

The viscosity of five samples of sunflower oil was measured at 25Cº and the viscosity of five other samples of corn oil was measured at 27Cº on another day.

Chemical Measurements:

Estimation of Acidity Number (IA)

The number of milligrams of potassium hydroxide required to neutralize the free fatty acids present in one gram of oil or fat. The Acidity number increases with the degree of vaporization, which increases the proportion of free fatty acids in the fatty substance, so the (IA) depends on the efficiency of the preservation methods to which the oil or fat is exposed during various manufacturing processes. Principle of action is neutralizing free fatty acids only with a solution of KOH dissolved in ethanol in the presence of phenolphthalein reagent characterized by a change from pink to transparent.

$$R\text{-}COOH + KOH \rightarrow R\text{-}COOK + H_2O$$

The acidity number is calculated according to the following equation (4):

$$IA = \frac{V \times N \times 56.1}{m} \qquad (4)$$

Where:

IA: Acidity number, V: Volume of KOH solution needed for titration in milliliters ml

N: Normality of the KOH solution (mol/) , m: Mass of the oil sample in grams (g)

56.1: Molar mass of potassium hydroxide (g/mol).

Estimation of Saponification Number (IS):

The number of milligrams of potassium hydroxide required to saponify one gram of oil or fat. The saponification number is calculated from the following equation (5):

$$IS = \frac{(V° - V) \times N \times 56.1}{m} \qquad (5)$$

Where:

IS: Saponification number.

V°: volume of HCl used in the comparison experiment in milliliters (without oil).

V: Volume of HCl in milliliters needed to modify the soap solution ml.

N: Normality of the HCl solution (mol/L), m: Mass of the oil sample in grams (g).

56.1: Molar mass of potassium hydroxide (g/ml).

Estimation of Ester Number (IE):

the number of milligrams of potassium hydroxide needed to saponify one gram of neutral oil (i.e., triglyceride) free of fatty acids.

The ester number is calculated from the following equation (6):

$$IE = IS - IA \qquad (6)$$

Where: IE: Esther number, IA: Acidity number, IS: saponification number.

Results and discussion

Sensory Properties of the studied vegetable oils:

Sensory properties vary from one oil to another, each oil has its own characteristics. These characteristics are color, taste and odor, and these characteristics were studied through sensory observation and recorded in the following table:

Table 2. Sensory properties of the studied vegetable oils: -

Key No.	Sample	Taste	Color	Odor
1	Alhadayiq - Sunflower Oil	Neutral	Golden yellow	Neutral
2	Prima - Sunflower Oil	Neutral	Light yellow	light
3	Alzuhra - Sunflower Oil	Neutral	Golden yellow	Neutral
4	alkhalij- Sunflower Oil	Neutral	Light yellow	Neutral
5	Carthage-Sunflower Oil	Neutral	Light yellow	light
6	Al-Safwa - Corn Oil	Delicate	Dark yellow	Pale
7	Al-Baraka - Corn Oil	Delicate	Dark yellow	light
8	Alzuhra - Corn Oil	Delicate	Dark yellow	light
9	Almarwa - Corn Oil	Delicate	Dark yellow	light
10	Alhadayiq - Corn Oil	Delicate	Dark yellow	light

The table shows that the vegetable oils (sunflower oil, corn oil) were studied in liquid form at laboratory temperature. The texture of the studied oils was liquid at room temperature due to the presence of unsaturated fatty acids. The color of vegetable oil is an important criterion for determining its quality. The color of the studied oils is limited to yellow, light yellow, and dark yellow; these colors indicate the presence of colored pigments (green plant pigment xanthophyll and carotenoids).

Physical properties

The physical properties of the vegetable oil samples, including relative density, moisture content, volatile substances, and viscosity, were studied. The results are presented in Table 3 below.

Table 3. Physical properties of the studied vegetable oils:

No.	Sample	Relative Density	Moisture (%)	Viscosity (Poise)
Sunflower Oils				
1	Alhadayiq	0.911	28.6	34.628
2	Prima	0.900	29.5	36.364
3	Alzuhra	0.905	27.1	33.757
4	alkhalij	0.902	23.6	30.703
5	Carthage	0.898	17.7	34.039
Corn Oils				
6	Al-Safwa	0.897	30.18	32.174
7	Al-Baraka	0.898	25.9	33.425
8	Alzuhra	0.899	25.2	29.535
9	Almarwa	0.896	10.1	32.287

10	Alhadayiq	0.894	8.2	32.892

Relative Density:

Relative density in vegetable oils refers to the ratio of the density of a specific oil to the density of water. This measurement is crucial for various applications in food processing, such as assessing the purity and quality of oils. Understanding relative density ensures consistency in recipes and product formulations and aids in identifying potential adulteration or contamination in vegetable oils. In our study, we measured this criterion at a temperature of 25°C, with the density values of the oils examined presented in figure 2 below:

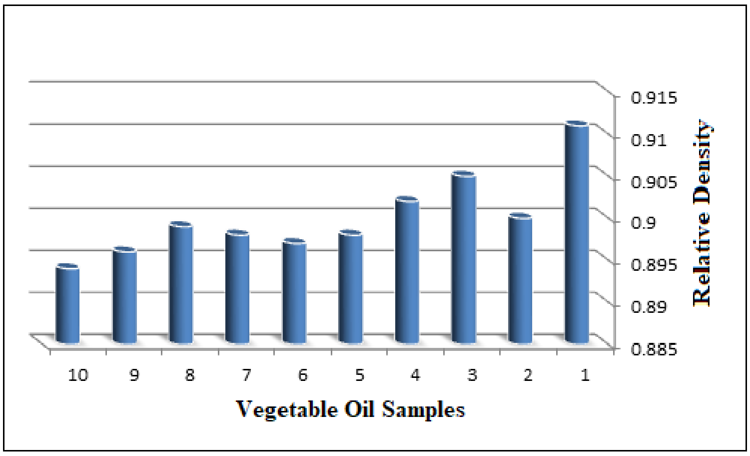

Figure 2. Relative density results for the studied oil samples

From Table 3, and figure 2 the relative density values for all samples ranged between (0.894 - 0.911), The results of the samples are in accordance with Codex standards, through these values the degree of purity of the oils can be predicted.

Moisture and Volatiles

Moisture in vegetable oils refers to the water content present in the oil, which, if not properly controlled, can lead to oxidation and rancidity. Volatiles are compounds that can evaporate at room temperature and significantly contribute to the aroma and flavor of the oil. The content of volatiles can impact both the quality and shelf life of vegetable oils. Understanding the moisture and volatile content in vegetable oils is crucial for maintaining product quality, stability, and consumer satisfaction. Monitoring these levels enables producers to make informed decisions regarding processing, storage, and packaging, ensuring optimal freshness and flavor retention.

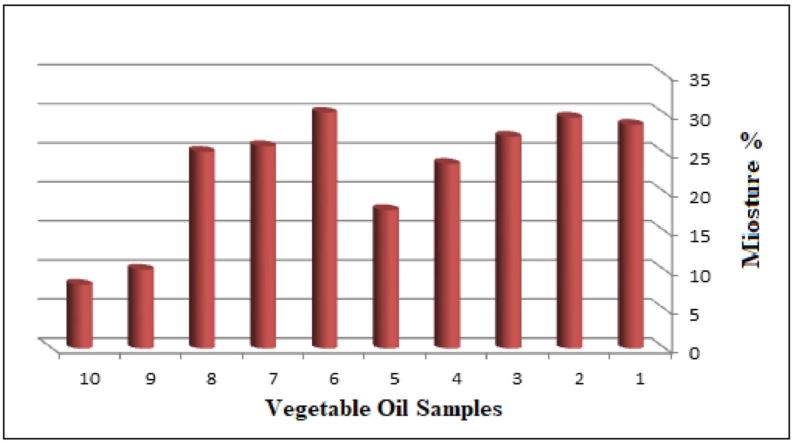

Figure 3. Moisture content and volatiles results for the studied oils

From figure 3 above, we can see that the moisture content values of the studied oils ranged between (8.2 - 30.18%), where the highest value was for Al-Safwa oil (30.18%) and the lowest value was for Alhadayiq oil (8.2%).

Viscosity

Viscosity in vegetable oils is influenced by factors such as temperature, fatty acid composition, and the presence of impurities. When selecting a vegetable oil for a specific culinary or industrial application, it is important to consider viscosity, as it can impact the texture and performance of the final product. For instance, oils with higher viscosity may be better suited for deep frying, while those with lower viscosity are often preferred for salad dressings. Furthermore, understanding the factors that influence viscosity can aid in making informed decisions about the storage and handling of vegetable oils to maintain their quality.

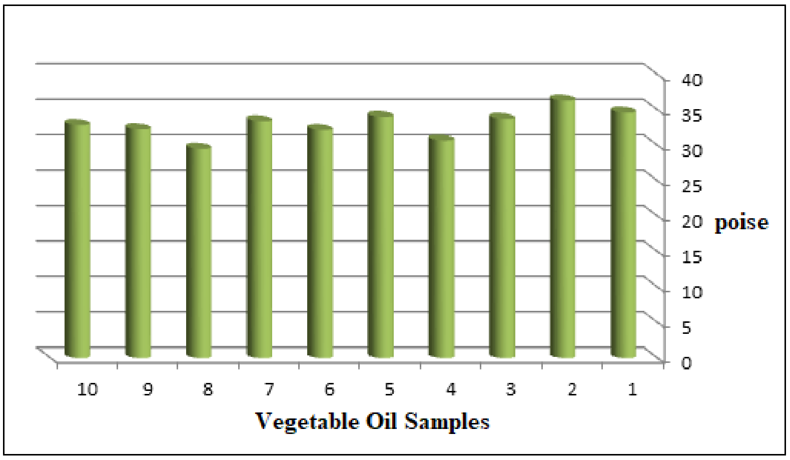

Figure 4. Viscosity results for the studied oil samples

We can see from the above figure that the viscosity values for all oil samples ranged between (36.364 - 29.535 poise), where the highest value was for Prima oil and the lowest value was for Alzuhra oil.

Chemical properties:

Table 4. Chemical properties of the studied vegetable oils:

No.	Sample	Acidity Number (IA)	Saponification Number (IS)	Ester Number (IE)
		Sunflower Oils		
1	Alhadayiq	3.1416	58.217	55.075
2	Prima	3.7026	58.217	54.514
3	Alzuhra	3.5343	31.755	28.220
4	alkhalij	3.1416	42.339	39.198
5	Carthage	2.806	63.509	60.704
		Corn Oils		
6	Al-Safwa	3.1416	47.632	44.490
7	Al-Baraka	2.805	42.339	39.535
8	Alzuhra	3.366	52.924	49.559
9	Almarwa	2.973	68.802	65.829
10	Alhadayiq	2.805	58.217	55.412

Acidity Number (IA)

The acid value measures the amount of free fatty acids resulting from triglyceride reactions, which can vary depending on the type of oil. It is important to note that a lower acidity value indicates a higher level of acidity, which can lead to rancidity, off-flavors, and decreased shelf life. It can also indicate poor quality or improper handling during processing and storage.

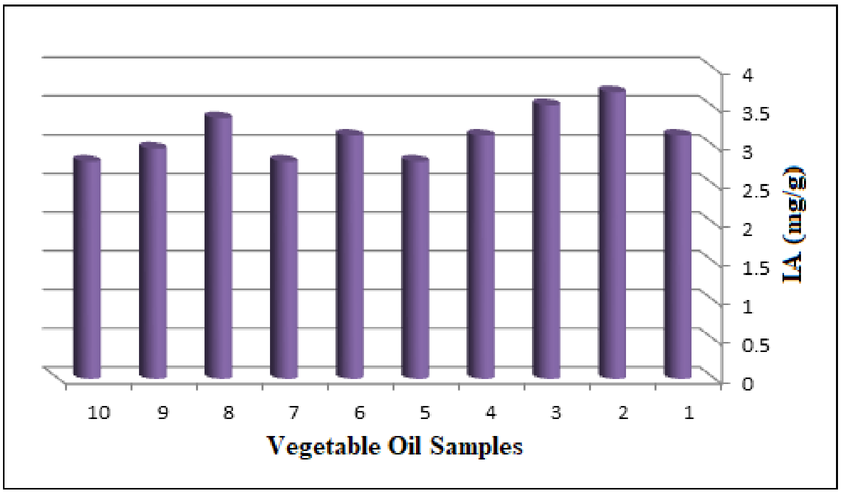

Figure 5. Results of acidity values for the studied oil samples

From the results shown in the table and figure (5), we can see that the acidity values of all studied samples ranged between (3.705 - 2.805 mg/g), where the highest value was for Prima oil, and the lowest value was for Alhadayiq and Al-Baraka oil.

Saponification Number (IS)

The saponification number, a measure of alkali needed to saponify fat or oil, is crucial for evaluating vegetable oils' quality, purity, and potential applications. Factors affecting this number include fatty acid types, unsaturation, impurities, processing methods, and storage conditions.

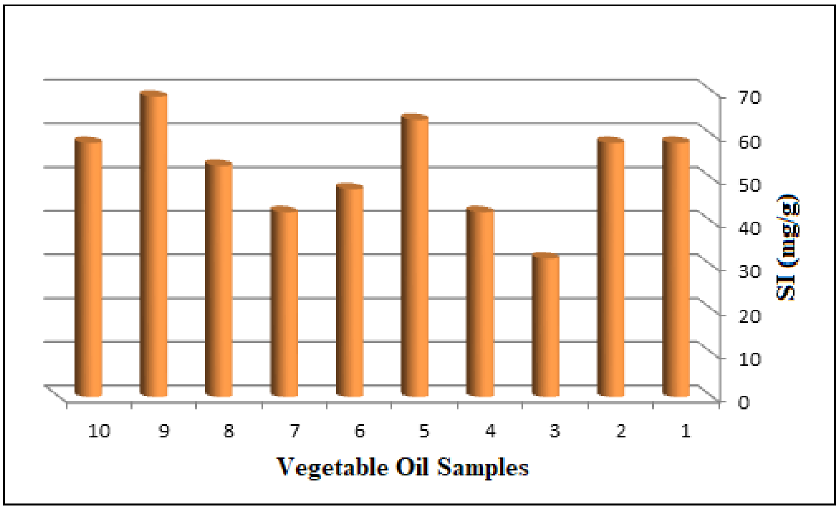

Figure 6. Saponification number results for the studied oil samples

From the table and figure (6) above, we notice that the saponification values of the studied oil samples ranged between (68.802 - 31.755mg/g), where the highest saponification value was for Almarwa oil, and the lowest value was for Alzuhra oil.

Ester Number (EI)

The ester number in vegetable oils is a key indicator of their chemical composition and reactivity, influencing factors such as viscosity and stability. Understanding the ester number is essential for ensuring the optimal performance of vegetable oils in various industrial and culinary applications.

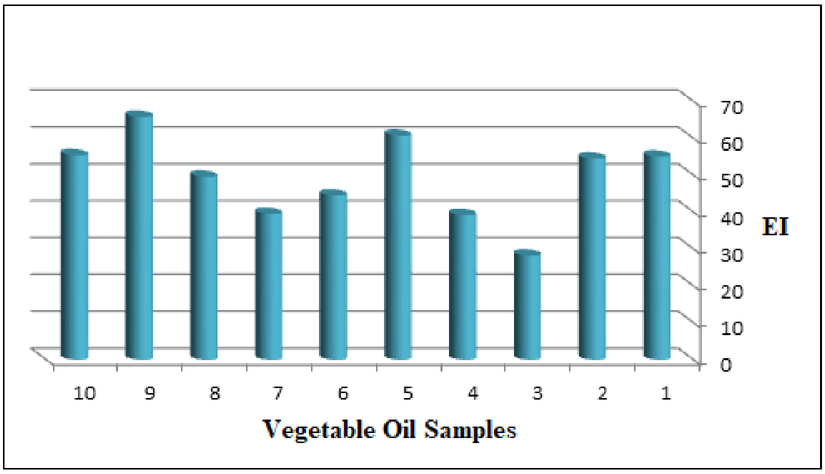

Figure 7. Ester number results for the studied oil samples

The results shown in Figure 7 indicate that the ester number values for all studied samples ranged from 28.220 to 65.829. The highest value was for corn oil (Almarwa), while the lowest value was for sunflower oil (Alzuhra).

Conclusion

10 samples of vegetable oils (5 from corn oil and 5 from sunflower oil) were analyzed by measuring physical and chemical constants (relative density, viscosity, humidity, pH number, saponification number, and ester number). Some of the results were consistent with standard standards such as humidity and density, while others did not correspond to standards such as pH, saponification number, and ester number, due to poor preservation and refining. The study also focused on determining the sensory characteristics of the oils (taste, color, and smell), noting that they had a liquid nature and a delicate and neutral taste. Their colors ranged from yellow to light yellow to dark yellow, and the smell

was described as mild and acceptable. The results of this study can take into account the validity date of these oils, their source, and the method of preservation.

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