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Quality of *Glycine max* (soybean) and *Helianthus annuus* (sunflower) oils stored in different environments and their suitability for consumption

Qualidade dos óleos de *Glycine max* (soja) e de *Helianthus annuus* (girasol) estocados em diferentes ambientes e sua adequação ao consumo

Calidad de los aceites de *Glycine max* (soja) y *Helianthus annuus* (girasol) almacenados en diferentes ambientes y su idoneidad para el consumo

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Abstract
Soybean and sunflower oils contribute to the diet in many countries, serving as a source of protein, lipids and fatty acids, and repair worn-out tissues and are a useful source of energy. Their excessive or inadequate consumption may provide increased risk of developing various diseases. Sunflower seed provides 20-40% of oil and soybeans provide oil with immense advantages due to their high content of essential fatty acids. This work aimed to evaluate the physicochemical quality of soybean and sunflower oils and their effects on health. 5 samples of oils: A, sealed sunflower oil exposed to ambient light; B, sealed sunflower oil without exposure to sunlight; C, sealed soybean oil exposed to ambient light; D, unsealed sunflower oil exposed to ambient light and E, sealed soybean oil without exposure to light were evaluated by determining the acidity index, through titration with NaOH 0. 1N, peroxide index, by titration with $Na_2S_2O_3$ 0.01N, moisture, by dissection at 105ºC, rancidity, by qualitative method and vitamin A by iCheck Chroma. The NM 425:2012 legislation was used to verify the suitability or otherwise of the samples. Moisture ranged from 0.1 to 0.92 w/w, acidity index from 0.17 to 0.34mgKOH, peroxide index from 4 to 15.2 meq.O_2/Kg, vitamin A between 13.27 and 29.95mgRe/Kg and positive rancidity. The results showed that the acidity index and vitamin A provided adequacy and the other parameters showed inadequacy for consumption.
Keywords: Parameters of quality; Food legislation; Safe food.

Resumo
Os óleos de soja e girassol contribuem para a dieta em muitos países, servindo como fonte de proteínas, lipídios e ácidos graxos e fonte útil de energia. O seu consumo excessivo ou inadequado pode proporcionar maior risco de desenvolvimento de várias doenças. A semente de girassol fornece 20 a 40% do óleo e os grãos da soja fornecem 18% a 20% de óleo com imensas vantagens devido ao seu alto teor de ácidos graxos essenciais. Este trabalho objectivou avaliar a qualidade físico-química de 5 amostras de óleos de soja e girassol e seus efeitos na saúde, sendo: A, óleo de girassol selado exposto a luz ambiente; B, óleo de girassol selado sem exposição a luz solar; C, óleo de soja selado exposto a luz ambiente; D, óleo de girassol não selado e exposto a luz ambiente e E, óleo de soja selado sem exposição a luz. Determinou-se o índice de acidez, através da titulação com NaOH 0,1N, índice de peróxido, por titulação com $Na_2S_2O_3$ 0,01N, umidade, por dessecação a 105ºC, rancidez, pelo método qualitativo e vitamina A por iCheck Chroma. A legislação NM 425:2012 foi usada para verificar a adequação ou não das amostras. A umidade variou de 0,1 a 0,92 w/w, índice de acidez de 0,17 a 0,34mgKOH, índice de peróxidos de 4 a 15,2 meq.O_2/Kg, vitamina A entre 13,27 e 29,95mgRe/Kg e rancidez positiva. Os resultados mostraram que o índice de acidez e vitamina A propiciaram a adequação sendo que os outros parâmetros evidenciaram a inadequação ao consumo.
Palavras-chave: Parâmetros de qualidade; Legislação para alimentos; Alimentos seguros.

Resumen
Los aceites de soja y girasol contribuyen a la dieta en muchos países, sirviendo como fuente de proteínas, lípidos y ácidos grasos, y reparan los tejidos desgastados y son una fuente útil de energía. El consumo excesivo o inadecuado de semillas de girasol puede aumentar el riesgo de desarrollar diversas enfermedades. Las semillas de girasol aportan un 20-40% de aceite y las de soja, inmensas ventajas por su alto contenido en ácidos grasos esenciales. El objetivo de este trabajo era evaluar la calidad fisicoquímica de los aceites de soja y girasol y sus efectos sobre la salud. Se evaluaron 5 muestras de aceite: A, aceite de girasol sellado expuesto a la luz ambiente; B, aceite de girasol sellado sin exposición a la luz solar; C, aceite de soja sellado expuesto a la luz ambiente; D, aceite de girasol sin sellar expuesto a la luz ambiente y E, aceite de soja sellado sin exposición a la luz, mediante la determinación del índice de acidez, por titulación con NaOH 0. 1N, índice de peróxido mediante valoración con $Na_2S_2O_3$ 0,01N, humedad mediante disección a 105°C, rancidez mediante método cualitativo y vitamina A mediante iCheck Chroma. Para comprobar la idoneidad o no de las muestras se utilizó la normativa NM 425:2012. La humedad osciló entre 0,1 y 0,92 p/p, el índice de acidez entre 0,17 y 0,34mgKOH, el índice de peróxidos entre 4 y 15,2 meq.O_2/Kg, la vitamina A entre 13,27 y 29,95mgRe/Kg y el enranciamiento positivo. El índice de acidez y la vitamina A favorecieron la adecuación, mientras que los demás parámetros mostraron inadecuación para el consumo.
Palabras clave: Parámetros de calidad; Legislación alimentaria; Alimentos seguros.

1. Introduction

The physical and chemical properties of an oil are mainly related to its composition in fatty acids, the degree of unsaturation and position of these in the glycerol molecule and the length of their carbon chain. The difference between a fat and an oil is in the physical state at ambient temperature, that is, a fat is a solid and an oil is a liquid generally, solid fats are indicated by a higher saturated fatty acid content, and liquids by a higher high level of unsaturated fatty acids (O'Brien et al., 2000).

Oil seeds are well differentiated as they provide protein and energy in the human diet (Khan et al., 2015). Sunflower is a significant crop that has 15-21(%v) protein and 50% oil content, ranking second in the world in edible oil manufacturing after soybean oil and grouped among the prominent vegetable oils for human diet. due to its nutritional value (Skorić et al., 2008; Nandha, 2014). Sunflower oil is obtained from sunflower seeds, with a variation between 20 and 40% of the oil obtained. This oil consists of 4 fatty acids, in particular oleic acid, linoleic acid, palmitic acid and a smaller portion of stearic acid (Ventura, 2014).

On the other hand, Soybean oil is obtained from soy beans and its use has immense advantages due to its high content of essential fatty acids, with linoleic acids (18:2 n^{-6}) and α-linoleic acid (18:3 n^{-3}); formation of crystals, which are easily filterable when the oil is hydrogenated and fractionated; high iodine index, which allows its hydrogenation producing a wide variety of plastic fats and refining with low losses (Santos et al., 2017).

Possible physicochemical alterations in oils, such as oxidation and hydrolysis, changes in acidity index, peroxide index and refractive index can lead to the production of toxic compounds such as peroxides, aldehydes, ketones, free radicals, trans fatty acids and others, harmful to human health, in addition to changes in taste, color and odor. Such toxic compounds can develop cardiovascular diseases, in addition to cancer, arthritis and premature aging. (Mendonça et al., 2008).

The acid number characterizes the hydrolytic rancidity which is the hydrolysis of the ester bond by lipase and moisture, this index reveals the state of conservation of oils in which the decomposition of glycerides is accelerated by heating and light, with rancidity almost always accompanied by the formation of free fatty acids (Tofanini, 2004).

The peroxide value characterizes oxidative rancidity. It is a classic and sensitive method for the determination of hydro peroxides, primary products of oxidation. The presence of these compounds is an indication of the deterioration of oil and fat samples. Therefore, it is an indicator of the initial stage of oxidative changes (Monteiro, 2014).

Since fried foods are highly consumed by the population and due to the fact that some substances present in oils are related to a series of diseases in men, companies must, during manufacturing, respect some physicochemical parameters, such as refractive index, peroxide value and acid value established (Freire et al., 2013). The present study aimed to evaluate the quality of glycine max (soybean) and helianthus annuus (sunflower) oils stored in different environments and their suitability for consumption.

2. Methodology

2.1 Sample collection

Sunflower and soybean oil samples were randomly collected from different shopping centers in the Municipal Market of Maputo City and taken to the LINHAA laboratory, for analysis purposes.

2.2 Sample preparation

In triplicate and preceded by a manual homogenization of the oil, 100mL were filtered through filter paper and sodium sulfate anhydride (Na_2SO_4) and placed in a 250ml Erlenmeyer flask.

2.3 Determination of physicochemical parameters

2.3.1 Moisture content

It was determined through desiccation in an oven. For this purpose, 5g of the sample was weighed in a petri dish and placed in an oven with circulating air at 105ºC for 2 hours until the constant weight, after which they were removed with tweezers, allowed to cool to ambient temperature and weighed again. The moisture content was obtained by formula 1.

$$\textit{Moisture content } (\%)\frac{w}{w} = \frac{m - m1}{m} * 100 \quad (1)$$

Where:

w/w – Weight per weight;

M – Final mass of the sample.

M1- initial mass of the sample.

2.3.2 Acidity index

It was measured by titration according to Machado et al. (2006), 10g of sample were placed in a 100mL Erlenmeyer, followed by addition of 25mL of neutral ether-alcohol solution (1:1), the content being homogenized using a magnetic stirrer. Then, 3 drops of phenolphthalein solution were added, followed by titration with sodium hydroxide solution (0.1N NaOH) in a 25mL burette until reaching the turning point of the indicator (pink color). The percent acidity was determined using formula 2.

$$\textit{Acidity index } (\%)\frac{w}{w} = \frac{V * N * Pm}{m} * 100 \quad (2)$$

Where:

V – Volume of spent titrant;

N – Normality of the titrant solution;

Pm - Molecular Weight of the characteristic acid.

2.3.3 Peroxide Index

Using the method proposed by IAL (2008), 5g of the sample were weighed in a 100 mL Erlenmeyer, with subsequent addition of 30 mL of acetic acid-chloroform ($C_2H_4O_2$) 3:2 solutions, then 0.5 mL of potassium iodide solution was incorporated and the contents were allowed to stand in the dark for 1 minute, after which 25 ml of distilled water were added. Then the solution was titrated with sodium thiosulphate ($Na_2S_2O_3$ 0.01N) until reaching a yellow color, at which point 0.5ml of the starch indicator solution was added and the titration continued until reaching a colorless blue color. Formula 3 was used to obtain the peroxide index.

$$N.P\ \%\frac{w}{w} = \frac{V*N*100}{m} * 100 \quad (3)$$

Where:

V- Volume of spent titrant;

N – Normality of the titrant solution;

M – Mass of the sample;

NP – Peroxides.

2.3.4 Kreiss reaction

The determination of rancid state in oils samples was based on the qualitative method suggested by IAL (2008), where 2 ml of the sample was transferred to 15 ml test tubes, with subsequent addition of 2 ml of HCl (36%) and 2 ml of ethyl ether $(C_2H_5)_2O$ and stirred for 30 seconds, followed by addition of 2 mL of 0.1% phloroglucin indicator and stirred again for the same period, leaving it to rest for 10 minutes. The presence of pink or red coloring (+) in the lower portion indicated the presence of rancidity and clear solution (-), non-rancidity.

2.3.5 Vitamin A

The quantification of vitamin A proceeded using the iCheck Chroma 3 method, a portable photometer to measure the concentration of vitamin A in oil, according to Huey et al. (2022). For that purpose, 0.1mL of sample was injected into a vial and the concentration of vitamin A in mgRE/kg.

2.4 Adequacy for consumption

Using the Mozambican legislation NM 425:2012 for fortified vegetable cooking oils, INNOQ (2014), the levels of moisture, acidity, peroxides and vitamin A in the studied samples were compared with those recommended for decision-making purposes.

2.5 Statistical analysis

Collected in a DBC based on a factorial scheme, the data were submitted to analysis of variance (ANOVA) through the general linear model (GLM) using the statistical package R version 4.1.2, and for the comparison of means it was based on the test of Tukey at a significance level of 5%.

3 Results and Discussion

3.1 Physicochemical analysis

The physicochemical constituents of vegetable oil samples are shown in Table 1.

Table 1 - Quality parameters of vegetable oil samples.

Parameters	Samples					NM 425: 2012	
	A	B	C	D	E	Min.	Max.
Moisture (%m/m)	0.10±0.05[c]	0.35±0.01[b]	0.46±0.01[ab]	0.92±0.01[a]	0.89±0.01[a]	-	0.2
Acidity index (mg KOH, m/m)	0.26±0.03[a]	0.26±0.1[a]	0.20±0.09[b]	0.17±0.06[b]	0.34±0.06[a]	-	0.6
Peroxide Index (millieq. O_2 /Kg)	15.2±0.9[a]	13.6±1.3[a]	13.40±1.9[a]	13.0±1.3[a]	4.0±1.00[b]	-	10
Vitamin A(mgRe/kg)	25.14±1.0[a]	29.9±1.6[a]	15.15±1.0[b]	13.27±0.85[b]	15.92±0.51[b]	15	43
Rancidity	+	+	+	+	-	-	-

Means ± standard deviation followed by the same letters on the same line do not show significant differences between them (*P >0.05*). A, sealed sunflower oil exposed to ambient light; B, Sunflower oil sealed without exposure to sunlight; C, soybean oil sealed exposed to ambient light; D, unsealed sunflower oil exposed to ambient light; and is, Sealed soybean oil without exposure to light. (+) indicates rancidity and (-) not rancid. Source: Authors.

3.1.1 Moisture content

The evaluated samples indicated moisture content ranging from 0.1 to 0.92%. Complying with Mozambican legislation NM 425: 2012 on fortified cooking oils (INNOQ, 2014), the moisture of vegetable oils must be below 0.2% m/m, therefore the values found are above the established except for sample A which showed a value of 0.1±0.05% in line with the established criteria for quality of vegetable oils. The highest moisture content (0.92%) was observed in sample D and the lowest (0.35%) in sample B, with significant differences between them ($p< 0.05$). This difference it is possibly allied with the storage conditions of the oils, and their constituents.

In the assessment carried out by Barros et al. (2013) when analyzing the physicochemical properties of soybean oil, a moisture content of 0.1 to 0.2% was obtained. Similarly, Almeida et al. (2013) in their research entitled physical-chemical characterization of mixtures of vegetable oils for food purposes, they obtained moisture content around 0.2%, results consistent with those verified in sample A of the present study. Ahmed et al. (2019) developing their study on evaluation of the quality and level of vitamin A fortification of soybean oil available in Bangladesh, obtained, similarly, moisture content ranging from 0.35±0.02 to 0.46±0.04%, Gariso (2011) obtained a moisture content around 0.46%, when developing his study on the effect of successive reuses of frying oils on quality parameters, results allied to those obtained in samples B and C of the present study.

Lower results than those obtained in the present study were referenced by Correia et al. (2014) on the evaluation of the potentialities and physical-chemical characteristics of sunflower oil, where they found a moisture content around 0.002% and by Sources (2011) in his research evaluating the quality of soybean and sunflower oils, obtained moisture content around 0.05% for soybean oil and 0.03% for sunflower oil, as well as by Nhatave (2015) when researching about the oxidative and hydrolytic stability of vegetable oils types produced at FASOREL-SAR, found moisture around 0.02%. In turn, Negash et al. (2019), obtained a moisture content ranging from 0.089 to 0.33%, a result lower than those obtained in samples B, C and D of this research.

3.1.2 Acidity level

The results showed that the acid index ranged from 0.2 to 0.34 mg/KOH. This range of values can be considered acceptable assuming that acid level for vegetable oil should be around 0.6 mg/KOH, according to INNOQ (2014). Higher acidity level was observed in sample E around 0.34 mg/KOH and samples A and B with 0.26 mg/KOH, respectively.

Statistically, samples E, A and B showed significant differences (p< 0.05) in relation to samples C and D. These variations may be associated with exposure to light causing photo oxidation.

Results allied to those obtained in the present research were reported in the study conducted by Silva et al. (2012) with the aim of characterizing the physical-chemical and dielectric constituents of biodegradable oils that obtained acid level around 0.14 mg/KOH for sunflower oil and 0.2 mg/KOH for soybean oil. Similarly, Almeida et al. (2013) when evaluating the physical-chemical characterization of mixtures vegetable oils for food purposes, obtained acid index of 0.17mg/KOH. In agreement with the results of the present study, Filho et al. (2014), in their study on the deterioration of vegetable oils exposed to different storage conditions, observed that soybean oil had acidity around 0.20KOH m/m to 0.26KOH m/m.

Higher results than those obtained in the present study were reported by Correia et al. (2014) when carrying their research on evaluation of potentialities and physical-chemical characteristics of Sunflower oil produced in Brazilian Northeast, which obtained an acidity index of 3.09 mg/KOH, and by Barros et al. (2013) when analyzing the physicochemical properties of soybean oil, who obtained acidity index ranging from 2.20 to 2.24 KOH/kg.

On the other hand, Neves et al. (2021) when studying physical-chemical characterization of the sunflower produced in cerrado south of Mato Grosso, reported acidity levels of 0.62mg/KOH and Laillou et al. (2012), in their research entitled low-quality vegetable oil is the limit for successful vitamin A fortification in Egypt, obtained acidity from 0.45 to 0.90mg/KOH and Castro et al. (2021) when evaluating the oil chemical quality of sunflower seeds, they found acidity index of 0.79 g/KOH.

Lower acidity levels in soybean and sunflower oil (0.08mg/KOH) than those obtained in the present study were reported by Sources (2011) in his research with the aim of evaluating the quality of soybean and sunflower oils, and by Nhatave (2015) who obtained acidity index of 0.06mg/KOH, in his reasech entitled study of the oxidative and hydrolytic stability of vegetable oils types produced at FASOREL – SAR, and also by Pigeon (2015) in his study related to chemical degradation of sunflower oil during the frying process, who obtained acidity around 0.06 and 0.08 mg/KOH m/m, as well as in the study carried by (Pinto et al., 2016) on the evaluation of the quality of frying oils in the preparation of foods sold at Universities in Belém, who found acidity between 0.13 and 0.14 mg/KOH.

3.1.3 Peroxide index

The results showed that the peroxide content ranged from 4 to 15.2 meq/kg and statistical differences were evident in this regard. Attending to INNOQ (2014), in vegetable oils the maximum limit is 10 meq/kg, in which only sample E was inserted. The highest peroxide content (15 meq/kg) was observed in sample A, and the lowest (4.0±1.0meq/kg) in E, being significantly different (p< 0.05). This higher value found may be related to the degradation of fat-soluble vitamins and essential fatty acids present in the sample.

Results in agreement with those obtained in the present study were found by Castro et al., (2021) when evaluating the oil chemical quality of sunflower seeds, indicating the peroxide index around 15.89meq/Kg. Masuchi et al. (2008) also evaluated the oxidative stability of commercial sunflower oil, and obtained a peroxide index of 13.3 meq/Kg, similar to the results obtained in samples C, B and D. In the evaluation made by Nhatave (2015), looking to study the oxidative and hydrolytic stability of several types of vegetable oils (Soybean and Sunflower) produced at FASOREL-SAR, obtained a peroxide index ranging from 0.38 to 10.17meq/kg for soybean oil.

Lower results than those obtained in the present study were reported by Sources (2011) when evaluating the quality of soybean and sunflower oils, mentioning a peroxide index of 0.7 to 1.0meq/kg in soybean oil and 1.1mEq/kg for sunflower oil. Rios et al. (2013) in his research on the oxidation of edible oils in the frying process, reported values around 2.91 to 5.0meq/kg for sunflower oil and values around 1.38 to 9.95meq/kg for oil of soy. Pinheiro (2019), evaluating the physical-chemical quality of soybean and sunflower oils, found 1.04 and 1.40meq/kg of peroxide value for soybean and sunflower oils, respectively. Similarly, Jorge et al. (2005) obtained a peroxide index around 0.99meq/kg for sunflower oil and 3.21meq/kg for soybean oil, when developing their research about physicochemical alterations of sunflower and soybean oils, Laillou et al. (2012) on low quality vegetable oil the peroxide content ranged from 0.41 to 5.81meq/kg, Hasan et al. (2016) about physicochemical properties of edible oils available in the local market in Bangladesh, was fixed in the range of 1.17 meq/kg and by Ahmed et al. (2019), evaluating the quality of vegetable oils, got 0.8 to 8meq/kg peroxide value. The higher peroxide indices observed in the present study can be justified taking into account Ludwig (2019) and Pinheiro (2019) who state that the process of autoxidation and enzymatic oxidation increases peroxide levels in oils.

3.1.4 Vitamin A

Vitamin A contents ranged from 13.27 to 29.95 mgRe/kg. These values can be considered acceptable assuming the limits (15 to 43 mgRe/kg) established by INNOQ (2014), for vegetable oils. Exposure to ambient light did not cause significant differences ($p>0.05$) between oil samples, although Kakuda et al. (2001) revealed in their study on the effect of packaging and light exposure on the stability of vitamin A in fortified oil, that light is a factor that is directly correlated with the quickly degradation of vitamin A in oil.

Lower values than those obtained in the present study were reported by Renaud et al. (2013) when quantifying vitamin A in fortified soybean oil and ranged from 0.20 to 4mgRe/kg. Ahmed et al. (2019) found values of vitamin A ranging from 0.93 to 1.35mgRe/kg. Silalahi et al. (2017) obtained vitamin A concentrations ranging from 33.7 to 34.3mgRe/kg, values above those found in the present study.

3.1.5 Kreiss reaction

The results obtained in the Kreiss reaction indicated that sample E did not show rancidity (-), indicating good quality and good conservation status. On the other hand, the remaining samples were rancid (+). This finding is directly correlated with the formation of free fatty acids, since Pombo (2015) and Morais et al. (2020) reveal that the higher peroxide content, its greater fragility in relation to oxidation reactions, resulting from degradation due to exposure of the samples to the incidence of sunlight and, or due to the deficit of the storage condition.

In the evaluation carried by Pinheiro (2019) on the physicochemical quality of soybean and sunflower oils under high temperatures, he reported that soybean and sunflower oil reached rancidity over 2 hours and 30 minutes of heating. Results similar to those of the present study were reported by Nawaz et al. (2019), comparing the physical, physicochemical properties of edible oils, by announcing positive as well as negative rancidity in sunflower and soybean oils, and by Babatunde & Umoru (2022), in a similar study that indicated positive and negative rancidity in different vegetable oils even when the oils were kept in a cool place and protected from sunlight and air.

4 Conclusion

The quality of oils was influenced by exposure to solar radiation as well as by conditions of conservation and storage. Soybean oil not exposed to ambient light was suitable for consumption. Significant differences were observed between sunflower and soybean oil samples when exposed to the same environmental and conservation conditions. There was not enough evidence of compliance with the criteria and storage conditions of vegetable oils in commercial establishments. Useful information was produced for manufacturers and consumers about the conditions of exposure and/or sale and use of vegetable oils.

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