

Linseed oil: Characterization and study of its oxidative degradation

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Submitted: 26 October 2018; Accepted: 20 February 2019; Published online: 13 January 2020

SUMMARY: This paper proposes to characterize and monitor the degradation of linseed oil under two oxidation conditions using some traditional oxidative and quality parameters. The experimental section of this study was divided into 2 stages. In the first one, three commercial linseed oil samples (OL1, OL2, and OL3) were characterized according to oxidative stability (90 °C) and fatty acid composition. In the second stage, the OL1 sample, selected due to its availability, was subjected to the following oxidation procedures: storage at room temperature conditions with exposure to light and air (temperature ranging from 7 to 35 °C) for 140 days and accelerated oxidation at 100 °C for 7h. Samples were collected at different time intervals and analyzed for oxidative stability (90 °C), peroxide value, and acid value. The results showed that all the samples presented a similar fatty acid profile and that the OL3 sample showed a higher induction period ($p < 0.05$). Regarding the oxidative degradation, the induction period of the OL1 sample reduced from 9.7 to 5.7 and 9.7 to 6.3 during 140 days of storage under room temperature and 7 h of accelerated oxidation, respectively. The end of induction period of the OL1 sample is expected to occur within 229 days according to an exponential mathematical model fitted to the induction period values at different temperatures. In addition, the OL1 sample met the limits proposed by Codex and Brazilian regulations for peroxide and acid values during the oxidation time intervals.

KEYWORDS: Accelerated oxidation; Linseed oil; Oxidation; Room temperature

RESUMEN: *Aceite de linaza: Caracterización y estudio de su degradación oxidativa.* Este trabajo propone caracterizar y monitorear la degradación del aceite de linaza en dos condiciones de oxidación utilizando algunos parámetros oxidativos y de calidad tradicionales. La sección experimental de este estudio se dividió en 2 etapas. En la primera, se caracterizaron tres muestras comerciales de aceite de linaza (OL1, OL2 y OL3) a través de la estabilidad oxidativa (90 °C) y la composición de ácidos grasos. En la segunda etapa, la muestra OL1 se seleccionó por su disponibilidad y se sometió a los siguientes procedimientos de oxidación: almacenamiento en condiciones ambientales con exposición a la luz y al aire (temperatura que varía de 7 a 35 °C) durante 140 días y oxidación acelerada a 100 °C durante 7 h. Las muestras se recogieron a diferentes intervalos de tiempo y se analizaron mediante estabilidad oxidativa (90 °C), índice de peróxido e índice de acidez. Los resultados mostraron que todas las muestras presentaron un perfil de ácidos grasos similar y también que la muestra OL3 mostró un período de inducción más alto ($p < 0.05$). En relación con la degradación oxidativa, el período de inducción de la muestra OL1 se redujo de 9,7 a 5,7 y de 9,7 a 6,3 durante los 140 días de almacenamiento en condiciones ambientales y 7 h de oxidación acelerada, respectivamente. Se espera que el final del período de inducción de la muestra OL1 ocurra dentro de 229 días de acuerdo con un modelo matemático exponencial ajustado a los valores del período de inducción a diferentes temperaturas. Además, la muestra OL1 cumplió con los límites propuestos por el Codex y las regulaciones brasileñas para los valores de peróxido y ácido durante los intervalos de tiempo de oxidación.

PALABRAS CLAVE: Aceite de linaza; Oxidación; Oxidación acelerada; Temperatura ambiente

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Citation/Cómo citar este artículo: Berto BM, Garcia RKA, Fernandes GD, Barrera-Arellano D, Pereira GG. 2020. Linseed oil: Characterization and study of its oxidative degradation. *Grasas Aceites* 71 (1), e337. <https://doi.org/10.3989/gya.1059182>

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1. INTRODUCTION

Edible vegetable oils are widely used in food, whether in their direct form as frying or salad oils or used indirectly in the formulation of various food products, such as bread, cakes, and margarine, among others. Although there are several sources of oils available, the market is practically dominated by palm, soybean, rapeseed, and sunflower oils (Statistica, 2018). In recent years, the demand for unconventional oils has been increasing as a result of their health benefits (Ixtaina *et al.*, 2011). In this context, linseed presents great potential for the production of oil with functional properties due to its high levels of omega 3 fatty acids (linolenic acid) and high oil content (Bayrak *et al.*, 2010).

Regarding the fatty acid composition, linseed oil has approximately 59% linolenic acid. In addition, it presents more than 90% unsaturated fatty acids in its constitution (Przybylski, 2005). Several studies have demonstrated the beneficial effects of linseed oil consumption, such as a reduction in the incidence of renal injury and some cancers, reduction in blood pressure, and increase in EPA and DPA concentrations in plasma (Ogborn *et al.*, 2002; Dwivedi *et al.*, 2005; Harper *et al.*, 2006; Paschos *et al.*, 2007; Yamaguchi *et al.*, 2015). These beneficial effects, related to the consumption of linseed oil, are mainly associated with its high levels of omega-3 fatty acids.

The high content of linolenic acid is an important nutritional claim for linseed oil; however, this tri-unsaturated fatty acid is highly susceptible to the oxidation reaction, which substantially reduces the shelf-life of this oil (Choo *et al.*, 2007). Oxidation, also denominated autoxidation, is an autocatalytic chain reaction which creates undesirable new compounds. Some of them originate unpleasant flavors which characterize oxidative rancidity. In the initial stages of oxidation, the primary oxidation products (hydroperoxides) are formed from the bonding of oxygen with the alkyl radical. In advanced stages of oxidation, the hydroperoxides which were accumulated in the previous step tend to rearrange, condensing or cleaving. The products originated from the cleavage, such as alcohols, aldehydes, ketones, and esters are responsible for oxidative rancidity, which hinders the sensorial quality and indicates the end of the shelf-life of the oil (Frankel, 1984;

Frankel, 2005; Márquez-Ruiz *et al.*, 2013). The oxidation rate depends on several factors, such as fatty acids composition, temperature, and exposed to light, oxygen, and metals (Choe and Min, 2006; Shahidi and Zhong, 2010).

Some previous studies have evaluated the oxidation of linseed oil (Douny *et al.*, 2016; Prescha *et al.*, 2014; Rudnik *et al.*, 2001; Szumala and Wysocka, 2018; Wagner and Elmadfa, 2000; Zhang *et al.*, 2013). However, there is a lack of studies which evaluate the loss in the oxidative quality of this oil under conditions that simulate its home storage and food service. Therefore, the purpose of this paper was to characterize and monitor the oxidative degradation of commercial linseed oil during long-term storage under room temperature and exposure to air and light using some traditional quality and oxidative parameters. Additionally, an accelerated oxidation procedure at 100 °C was performed for comparative purposes.

2. MATERIALS AND METHODS

2.1. Materials

Three commercial crude linseed oil samples were used in this work. Here, the samples are described as OL1, OL2, and OL3. OL1 was supplied by a Brazilian producer. OL2 and OL3 were purchased from local grocery stores in Campinas-SP (Brazil). The solvents and chemicals used were at least of analytical grade and were obtained from national suppliers.

2.2. Linseed oil sample characterization

The OL1, OL2, and OL3 samples were characterized according to fatty acid composition and oxidative stability. The fatty acid composition analysis was based on the AOCS Ce 1h-5 standard method (AOCS, 2009) and the methyl esters were obtained according to the method proposed by Hartman and Lago (1973). The chromatography analysis was performed using an Agilent 6850 Series GC System (Agilent, United States) equipped with a DB-23 Agilent capillary column (50% cyanopropyl-methylpolysiloxane, 60 m in length, 0.25 mm internal diameter, and 0.25 µm film thickness) and a flame

ionization detector. The oven temperature program was as follows: 5 min at 110 °C; raised from 110 to 215 °C at a rate of 5 °C min⁻¹; held for 24 min at 215 °C. The detector and injector temperatures were 280 and 250 °C, respectively. Helium was used as carrier gas at 1 mL min⁻¹ at a split ratio of 1:50. The identification of fatty acids was made by comparing the retention times of the peaks with those of the respective fatty acid standards. Oxidative stability, expressed by the induction period, was determined according to AOCS Cd 12b-92 method (AOCS, 2009) using an 893 Biodiesel Rancimat (Metrohm, Switzerland). The experimental conditions were as follows: 5 g of sample, heat block adjusted at 90 °C; and air flow rate of 9 L·h⁻¹.

Among the three samples, OL1 was available in the greatest quantity; therefore, it was selected for other characterization analyses. Peroxide value, acid value, and Lovibond color were measured according to AOCS Cd 8b-90, AOCS Ca 5a-40, and AOCS Cc 13j-97 methods, respectively (AOCS, 2009). Tocopherol content was obtained by high performance liquid chromatography (HPLC) using normal phase and fluorescence detector (excitation at 290 nm and emission at 330 nm) according to the IUPAC method 2432 (IUPAC, 1992). The characterization analyses were performed in triplicate.

2.3. Oxidation procedures of linseed oil

The OL1 sample was submitted to two different oxidation procedures: storage under room temperature conditions and accelerated oxidation at 100 °C. In the first one, the sample was kept in an open glass container with a surface-to-volume ratio of 0.2 cm⁻¹ and stored in the laboratory with exposure to indoor air and light. Samples were collected for analysis each 20 days during 140 days of storage. Ambient temperature ranged from 7 to 35 °C during the storage period. The OL1 sample was also submitted to an accelerated oxidation procedure at 100 °C using a surface-to-volume ratio of 0.4 cm⁻¹. Samples were collected at different time intervals: 0, 1, 3, 5, and 7h. The experiments were performed in duplicate.

2.4. Monitoring of linseed oil oxidation

The OL1 samples submitted to oxidation procedures were analyzed using the oxidative and quality parameters frequently adopted in technical regulations, such as peroxide value (AOCS Cd 8b-90), acid value (AOCS Ca 5a-40), and oxidative stability at 90 °C (AOCS Cd 12b-92). The shelf-life or the end of the induction period of the OL1 sample under storage conditions (7 to 35 °C) was predicted by fitting an exponential model to the induction period data obtained at different temperatures

(70, 80, 90, and 110 °C) using an 893 Biodiesel Rancimat (Metrohm, Switzerland) according to AOCS Cd 12b-92 (AOCS, 2009).

2.5. Statistical analysis

Analysis of variance and the means comparison using Tukey's test at the 5% level were performed by Statistica 7 software (StatSoft, USA). Regression analyses were performed using the Excel 2007 software (Microsoft Co., USA).

3. RESULTS AND DISCUSSION

Table 1 shows that the induction period of the OL1, OL2, and OL3 samples ranged from 9.30 to 10.27 h at 90 °C and also that OL3 was the most stable sample ($p < 0.05$). Symoniuk *et al.*, (2016) reported values of induction period ranging from 7.47 to 9.20 h at 90 °C for five linseed oil samples; therefore, their results corroborate with those obtained in this paper. Similarly, Raczyk *et al.*, (2016) observed that the induction period of four linseed oil samples ranged from 3.47 to 5.63 h at 100 °C. Considering the Van't Hoff's rule, which states that the reaction rate practically doubles with each 10 °C increase in temperature, the results found by the authors are similar to those obtained in this paper. For comparison purposes, Castelo-Branco *et al.*, (2016) found that canola, corn, soybean, and sunflower oils presented induction periods of 8.63, 9.96, 12.00, and 4.91, respectively, at 110 °C.

The low oxidative stability of commercial linseed oil samples can be explained by their fatty acid compositions. Table 2 shows that the content of unsaturated fatty acid in the commercial samples ranged from 87.33 to 89.17%. Among these, linolenic (46.57 to 54.24%) was the major fatty acid, followed by oleic (20.46 to 24.00%) and linoleic (14.06 to 18.21%) fatty acids. It is noteworthy that the oxidation rate of esters from linolenic acid is 96 times higher than that of oleic acid because linolenic acid has two bis-allylic carbons in its molecule (Frankel, 2005). Thus, the OL1, OL2, and OL3 samples were prone to oxidative degradation.

The OL1 sample was also characterized for other oxidative and quality parameters normally applied

TABLE 1. Induction period of the commercial linseed oil samples

Sample	Induction period at 90 °C (h)
OL1	9.71 ± 0.02 ^a
OL2	9.30 ± 0.33 ^a
OL3	10.27 ± 0.04 ^b

The results are given as mean ± SD (n = 3). Values with the same superscripts do not differ significantly from one another ($p > 0.05$) according Tukey's test.

TABLE 2. Fatty acid composition of the commercial linseed oil samples

Fatty acids	OL1 (%)	OL2 (%)	OL3 (%)
Myristic (C14:0)	0.07 ± 0.01	0.10 ± 0.00	0.09 ± 0.01
Palmitic (C16:0)	5.72 ± 0.02	6.87 ± 0.15	6.62 ± 0.03
Palmitoleic (C16:1)	0.09 ± 0.01	0.09 ± 0.01	0.29 ± 0.01
Margaric (C17:0)	0.10 ± 0.03	0.08 ± 0.02	0.07 ± 0.00
Stearic (C18:0)	4.49 ± 0.05	5.00 ± 0.00	5.00 ± 0.00
Oleic (C18:1)	20.46 ± 0.16	22.10 ± 0.00	24.00 ± 0.00
Linoleic (C18:2)	14.06 ± 0.03	18.21 ± 0.02	15.33 ± 0.02
Linolenic (C18:3)	54.24 ± 0.20	46.57 ± 0.09	47.42 ± 0.02
<i>Trans</i> linolenic (C18:3 t)	0.24 ± 0.00	0.20 ± 0.00	0.19 ± 0.00
Arachidic (C20:0)	0.17 ± 0.01	0.25 ± 0.01	0.31 ± 0.01
Gondoic (C20:1)	0.10 ± 0.01	0.19 ± 0.02	0.23 ± 0.00
Behenic (C22:0)	0.17 ± 0.01	0.24 ± 0.01	0.35 ± 0.01
Lignoceric (C24:0)	0.13 ± 0.02	0.14 ± 0.00	0.22 ± 0.02
Saturated	10.84 ± 0.09	12.63 ± 0.15	12.66 ± 0.01
Unsaturated	89.17 ± 0.08	87.33 ± 0.08	87.49 ± 0.01
<i>Trans</i>	0.24 ± 0.00	0.20 ± 0.00	0.19 ± 0.01

The results are given as mean ± standard deviation (n= 3).

TABLE 3. Oxidative and quality parameters of the OL1 sample

Parameter		
Peroxide value (meq O ₂ ·Kg ⁻¹)		3.00 ± 0.50
Acid value (mg KOH·g ⁻¹)		2.14 ± 0.05
Tocopherol (mg·100g ⁻¹)	α	1.17 ± 0.23
	β	-
	γ	43.53 ± 0.82
	δ	0.86 ± 0.11
	Total	45.56 ± 1.16
Lovibond color	Red	9.20 ± 0.10
	Yellow	70.00 ± 0.00

The results are given as mean ± standard deviation (n= 3).

in the vegetable oil analysis routine. Table 3 shows that the peroxide and acid values of the OL1 sample were 3 meq O₂·Kg⁻¹ and 2.14 mg KOH·g⁻¹, respectively. Thus, the OL1 sample met the limits proposed by Codex Standard 19-1999 and Brazilian regulation RDC 270 from September 22, 2005 for virgin and cold pressed oils, whose limits are 15 meq O₂·Kg⁻¹ for peroxide value and 4 mg KOH·g⁻¹ for acid value. Regarding the natural antioxidant content, the OL1 sample presented 45.56 mg·100 g⁻¹ of total tocopherols (Table 3). γ-tocopherol was the most abundant isomer present in the OL1 sample, representing approximately 95% of the tocopherol content. This result corroborated those found by Wagner and Elmadfa (2000), who observed

that the total tocopherol content in linseed oil was 68.1 mg·100 g⁻¹, of which 8.2, 58.9, and 1.0 mg·100 g⁻¹ were represented by α, γ, and δ-tocopherols, respectively. The tocopherol content is a very important parameter in the quality control of linseed oil because together with phenolic compounds, it represents the defense mechanism of the oil against oxidative degradation (Khattab and Zeitoun, 2013). Tocopherols are known to block the formation of free radicals in the initiation and propagation steps (Steel *et al.*, 2013). Thus, the higher the tocopherol content, the higher the oxidative stability, and consequently, the longer the shelf-life of linseed oil. However, it is important to note that other minor compounds present in the oil, as well as processing and storage conditions, also influence the shelf-life of the oil. Table 3 shows that the OL1 sample presented Lovibond color of 9.20 and 70.00 for red and yellow, respectively. For comparison purposes, refined soybean oil can be obtained with Lovibond color values lower than 1 for red and 20 for yellow (Erickson, 1995). The OL1 sample showed a more intense Lovibond color because it was not submitted to refining, especially to the bleaching step. However, unrefined oils retain a greater amount of functional compounds, such as tocopherols, and also have a differentiated coloration, which refer to a “less processed product”, like olive and avocado oils for example, which have been gaining prominence among consumers.

Figure 1 shows that the induction period of the OL1 sample reduced from 9.7 to 5.7 h (approximately 41%) and 9.7 to 6.3 h (approximately 35%)

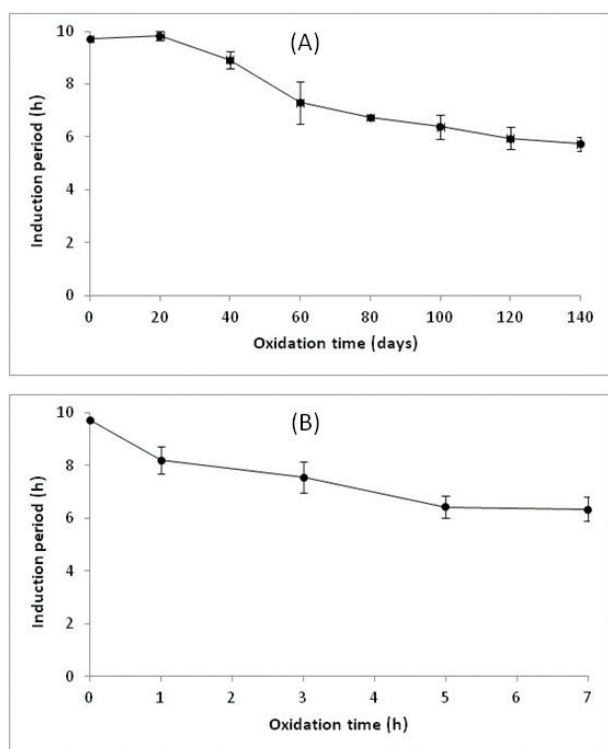


FIGURE 1. Induction period of the OL1 sample during storage under room temperature conditions (A) and accelerated oxidation at 100 °C (B).

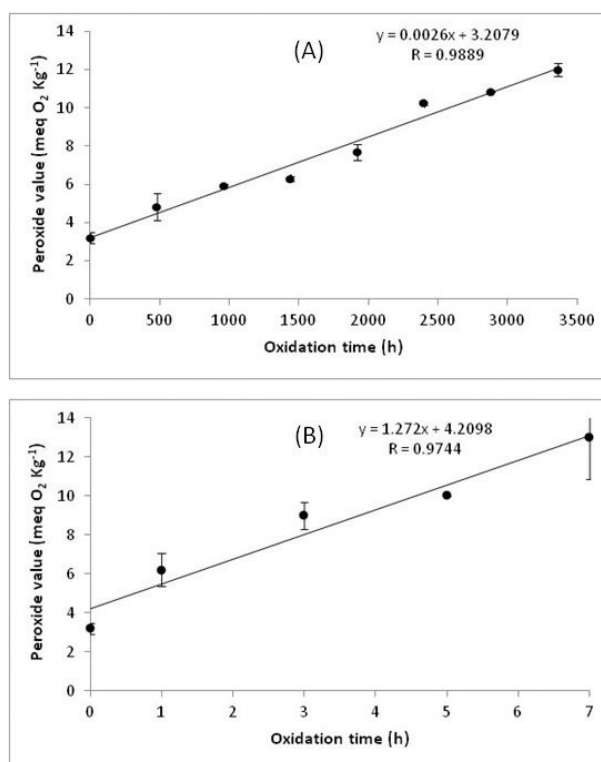


FIGURE 2. Peroxide values of the OL1 sample during storage under room temperature conditions (A) and accelerated oxidation at 100 °C (B).

after 140 days of storage under room temperature conditions and 7 h of oxidation under high temperature (100 °C), respectively. Therefore, these results suggest that the OL1 sample was in the early stages of oxidation even after storage and exposure to high temperatures, since its oxidation resistance was not overcome during the time intervals mentioned above.

The formation of the primary oxidation products, also called hydroperoxides, was evaluated by the peroxide value. Figure 2 shows that the peroxide value of the OL1 sample increased moderately under both oxidation conditions evaluated in this paper. The peroxide value of the OL1 sample was 12 and 13 meq O₂·kg⁻¹ in 140 days of storage under room temperature conditions and 7h of accelerated oxidation, respectively. Therefore, these results suggest that the OL1 sample was not rancid even in 140 days of storage and 7 h of at 100 °C because the peroxide values were under the limit proposed by the Codex Standard 19-1999 and Brazilian regulation RDC 270 from September 22, 2005 for virgin and cold pressed oils. Figure 2 also shows that the oxidation rates of sample OL1 were 0.0026 and 1.2720 meq O₂·kg⁻¹ h⁻¹ under room temperature storage conditions and accelerated oxidation, respectively. Therefore, the oxidative degradation of the OL1 sample at 100 °C was approximately 489-fold in comparison with the room temperature. The evolution

of the primary oxidation products measured by peroxide value was used to calculate the oxidation rate because hydroperoxides are recognized as the main product formed during the shelf-life of oil (Pereira *et al.*, 2013).

Regarding to the oxidative quality, the results shown in Figures 1 and 2 suggest that linseed oil can be used as salad oil for periods of more than 4 months under storage at room temperature (7 to 35 °C) and it can also be used directly or as an ingredient in food products under high temperatures provided that the processing time and the shelf-life of the product are short.

Figure 3 shows that the acid values for the OL1 sample ranged from 2.14 to 2.52 and 2.14 to 2.25 mg KOH·g⁻¹ in 140 days of storage under room temperature conditions and 7h of accelerated oxidation, respectively. Therefore, acid value presented a small variation, especially in the accelerated oxidation condition. This small variation occurred because acidity is a parameter which changes mainly in the advanced stages of oxidation, which is after the end of the induction period of the oil (Pereira *et al.*, 2013). In addition, the acid values for the OL1 sample in both oxidation procedures were under the limit proposed by the Codex Standard 19-1999 and Brazilian regulation RDC 270 from September 22, 2005 for virgin and cold pressed oil (4 mg KOH g⁻¹).

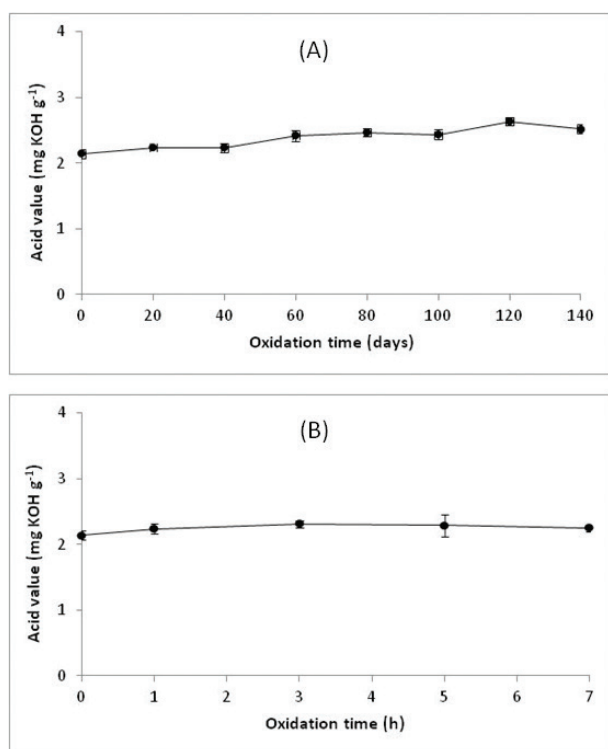


FIGURE 3. Acid values of the OL1 sample during storage under room temperature conditions (A) and accelerated oxidation at 100 °C (B).

As the OL1 sample still presented 5.7 h of induction period in 140 days of storage under room temperature conditions, it was proposed to infer when the end of the stability of this sample would occur. Thus, an exponential mathematical model with an excellent correlation coefficient ($r = 0.9987$) was obtained (Figure 4) from the induction period values achieved according to AOCS method Cd 12b-92 using different temperatures. The induction period extrapolated from this model, under storage conditions (7–35 °C), occurred between 27 and 229 days. Consequently, the end of the shelf-life of the OL1 sample is expected to occur within 229 days of storage. Although this result is useful for predicting the shelf-life of linseed oil, it is noteworthy that some factors, such as contact with light, oxygen, and metals can influence it.

4. CONCLUSIONS

Commercial linseed oil shows proper oxidative quality, measured by peroxide value, acid value, and induction period, even after 140 days of storage under room temperature (7 to 35 °C) and 7 h under accelerated oxidation at 100 °C. Thus, linseed oil, which is one of the main sources of omega-3 fatty acid from vegetables, has potential to be better exploited for food use, either for direct

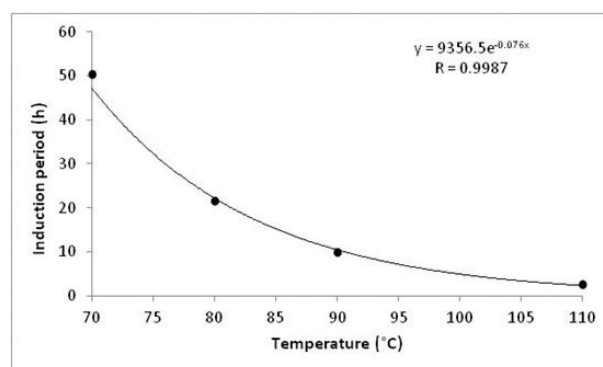


FIGURE 4. Exponential relationship between induction period and temperature obtained by Rancimat.

consumption as salad oil, or as an ingredient in other food products.

ACKNOWLEDGEMENTS

The authors are grateful to the National Council for Scientific and Technological Development (CNPq) for the scholarship (PIBIC).

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