GRASAS Y ACEITES, 59 (2),
ABRIL-JUNIO, 160-165, 2008,
ISSN: 0017-3495

Sterculia striata seed kernel oil: characterization and thermal stability

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RESUMEN

Aceite de almendra de la semilla de *Sterculia striata*: Caracterización y estabilidad térmica.

El objetivo de este trabajo fue la caracterización del aceite de almendra de la semilla de Sterculia striata. Se determinaron la composición química, las propiedades químico-físicas, composición en ácidos grasos, así como la estabilidad térmica de la harina (polvo) de almendra. La composición química de la harina contiene cerca de 25,8% de lípidos, los parámetros químico-físicos, tales como: índice de acidez, yodo, peróxido y saponificación fueron 0,82% (ácido oleico), 69,2 (g yodo/100 g de aceite), 4,20 m.eq./kg de aceite y 136,1 (mg KOH/g de aceite), respectivamente. Con relación a la composición en ácidos grasos, el aceite contiene 36,2; 43,7 y 10,9% de ácidos grasos saturados, monoinsaturados y polyinsaturados, respectivamente. El ácido palmítico (31,9%), oleico (41,7) y linoleico (10,73%), fueron los principales ácidos grasos saturados, monoinsaturados y polyinsaturados, respectivamente. Los ácidos grasos ciclopropenoides, esto es, ácidos estercúlico y malválico fueron identificados y cuantificados en la proporción de 5,3 y 2,3%, respectivamente. Con respecto a la estabilidad térmica del aceite, el análisis termogravimétrico (TGA) demostró que el aceite es estable hasta la temperatura de 284 °C, a partir de este valor el aceite comenzó a perder masa. El análisis termogravimétrico diferencial (DTGA) indicó la existencia de tres etapas de degradación con el aumento de la temperatura del aceite. Estas etapas significan la degradación de los aceites saturados, monoinsaturados y polyinsaturados. El análisis mediante calorimetría diferencial de barriido (DSC) detectó dos zonas de transición de energía exotérmica, una debida a la reacción de oxidación y la otra a la descomposición de los ácidos grasos. La primera transición exotérmica del aceite comenzó a la temperatura (Ti) de 287,79 °C con una variación de entalpía de 11,69 J/g y, la segunda con la temperatura inicial (Ti) de 384,87 °C, una temperatura de pico (Tp) 415,71 °C y una temperatura final (Tf) de 448,9 °C con una variación de entalpía de 200,83 J/g.

PALABRAS-CLAVE: *Composición en ácidos grasos – Estabilidad térmica – Propiedades químico-físicas – Sterculia striata (aceite de almendra).*

SUMMARY

***Sterculia striata* seed kernel oil: Characterization and thermal stability**

The objective of the present work was to characterize sterculia seed kernel oil. The chemical composition of the seeds, physicochemical properties as well as the fatty acid composition of the kernel oil was determined. The chemical composition of kernel flour presented about 25.8% lipid content. The physicochemical parameters such as acid, iodine, peroxide and saponification values were 0.82 (% as oleic acid), 69.2 (g iodine/100 g oil), 4.20 (m eq./kg) and 136.1 (mg. KOH/g oil), respectively. With respect to fatty acid composition, the oil contained 36.2, 43.7 and 10.9% saturated, monounsaturated and polyunsaturated fatty acids, respectively. Palmitic acid (31.9%), oleic acid (41.7%) and linoleic acid (10.73%) were the principal saturated, monounsaturated and polyunsaturated fatty acids. Two cyclopropanoid fatty acids i.e. sterculic and malvalic acid were identified at a concentration of 5.3 and 2.3%, respectively. With regards to the thermal stability of the oil, a thermogravimetric analysis (TGA) has shown that the oil was stable until about 284 °C, above that the oil started loosing mass, while a differential thermogravimetric analysis (DTGA) revealed three stages of degradation with an increase in temperature. These stages corresponded to the degradation of polyunsaturated, monounsaturated and saturated fatty aids. The Differential Scanning Calorimetric (DSC) analysis showed the existence of two exothermic events of energy transition, one of which is related to the oxidation reactions and another to the decomposition of the oil. Exothermic transitions in the oil were initiated at a temperature (Ti) of 287.79 °C, and terminated at 347.81 °C, with an enthalpy variation of 11.69 joules.g^{-1} and at initial temperature (Ti) of 384.87 °C, peak temperature (Tp) 415.71 °C, final temperature (Tf) 448.9 °C and an enthalpy of 200.83 Joules. g^{-1}.

KEY-WORDS: *Fatty acid composition – Physico-chemical properties – Sterculia striata seed kernel oil – Thermal stability.*

1. INTRODUCTION

The Brazilian fauna is rich in a variety of exotic and native plants with a high potential for exploration either due to the genetic pattern of the specie or the diversification of its culture or due to the fruits (Carvalho, 1996). Braga (1976) emphasized the importance of some of these fruits such as cashew nuts, brazil nuts etc. and suggested that if explored rationally, they would constitute an excellent option as a food source. Among these species the seed kernels of sterculia striata St. Hill locally known as “chicha”, possess an elevated nutritional potential

due to their high lipid and protein content (Araújo, 1997).

The Chichazeiro belong to the family of *sterculiaceas* to which cocoa (*Teobromo cacao* L.), and cupuaço (*Theobromo grandiflorum* L.) also belong. This family is composed of a vast number of the specie of the genre sterculia which includes, among others, the *Sterculia apétala, Sterculia foetida, Sterculia speciosa,* and *Sterculia striata* (Lorenzi, 1992). The chichá has its origin in India and Malaysia where the seeds are consumed in dried and roasted form as well as for the extraction of oil. This specie is also very common in the Northeast of Brazil. The Empresa Brasileira de Pesquisa Agropecuária (EMBRAPA, 2002) studied the propagation and seed yield of this plant. The plant starts to produce fruits within 18 to 24 months after plantation and an adult tree with adequate agricultural practices can produce approximately about 40kg of seeds per year. The seeds contain about 60% kernels that contain lipids at concentrations varying approximately from 28 to 32% (Araújo, 1997; Oliveira *et al.,* 2000).

Thermo-analytical techniques are commonly used to evaluate the stability of vegetable oils (Kowalski, 1989; Wesolowski, 1993) and have the advantage of providing information that conventional methods are incapable of (Santos *et al*., 2004). Besides, these techniques require smaller quantities of samples, shorter periods of analysis, and offer smaller chances of error. The curves of Differential Thermogravimetry (DTG) provide detailed information such as initial and final temperatures of a particular process. The thermogravimetric profiles for sunflower oil showed a level stretch indicative of stability up to about 200 °C. Thermal decomposition of the oil occurred in three stages, related to the decomposition of polyunsaturated, monounsaturated and saturated fatty acids, respectively (Souza *et al*., 2004).

Very little information on the lipid fraction of *sterculia striata* (chichá) seed kernel is available except that of Miralles *et al.,* (1993) on cyclopropenoid fatty acids in sterculia oils from Senegal and Aued-Pimentel *et al.* (2004) on the evaluation of the formation of methyl esters procedure to determine these fatty acids. No report is so far available on the thermal stability of this oil. Therefore, the present work was undertaken to determine the physico-chemical properties, fatty acid composition and thermal stability of the *sterculia striata* seed kernel oil.

2. MATERIAL AND METHODS

2.1. Material

The fruits of *Sterculia striata* were collected at the experimental station of Empresa Brasileira de Pesquisa agropecuária (EMBRAPA-meio norte) located in Teresina - Piaui state, Brazil. The kernels were separated from the seeds, dried at 45-50 °C for about 24 hr and the pellicles were removed. The de-pellicled kernels were triturated in a mill and passed through the screen of 25 mesh size. The kernel powder was packed in polyethylene bags and stored in the refrigerator (5 °C) until use.

2.2. Proximate analysis of kernel powder and extraction of the oil

Moisture, protein, lipid and ash contents were determined following standard AOAC (1990) methods. Total carbohydrate content was calculated from the difference. Five different samples were analyzed in duplicate. The oils were obtained by extraction of the dried kernel powder with solvent hexane. Three lots of oil were prepared.

2.3. Physical and physicochemical properties of oil

The specific gravity and refractive index were determined at a temperature of 25 °C using a specific density bottle and a refractometer, respectively. For the determination of acid, peroxide, iodine and saponification values standard AOAC (1990) methods were used. Three different samples of oil were analyzed in duplicate.

2.4. Fatty acid composition of the oil

Approximately 10g chichá kernel powder was suspended in 50 ml ethyl ether for about 24 hr at room temperature. The suspension was filtered through a Whattman no. 32 filter paper containing anhydrous sodium sulfate. The ethereal extract was concentrated in a rotary evaporator at 40 °C to obtain the oil. The fatty acids were transformed into their methyl esters (FAME) following the method recommended by IUPAC (1987). One hundred milligrams of oil was weighed in 20 ml centrifuge tubes. 2 ml of hexane and 0.2 ml of 2M methanolic solution of potassium hydroxide were added to the centrifuge tube. The mixture was agitated for about 60 sec. A 2 ml portion of saturated sodium chloride was added to the mixture to accomplish phase separation. The phase containing the oil was separated. The fatty acids were identified and quantified by using a gas chromatograph HP 5890 Series II (Hewlett Packard, Palo Alto, CA, USA) equipped with a flame ionization detector. A 1.5 µl FAME sample was injected and GC separation was carried out on an HP-INNOwax capillary column (Hewlett Packard; 30 m length, 0.25 mm id. and 0.25mm film thickness). The carrier gas (helium) head pressure was maintained at 11.5 psi and the column flow rate was 1ml/min. The oven temperature was held initially at 120 °C for 1 min, later increased at a rate of 8 °C/min to 210 °C and maintained at 210 °C for 45 min. The temperatures of the injection port and of the detector were 250 °C and 280 °C, respectively. FAME were identified by matching their retention time data with those of the

authentic standards obtained from various firms (Sigma; Nu-Chek-Prep, USA), which were also run under identical analytical conditions. The peaks of Cyclopropenoid fatty acids were tentatively identified by matching their spectra with that of the spectra contained in GC/MS software provided by Varian.

2.5. Identification of Cyclopropenoid fatty acids by Nuclear Magnetic Resonance

The NMR ^{1}H and NMR ^{13}C spectra were taken. Fifty mg samples of cold extracted oil were dissolved in CDCl3 in a proportion of 1:20 and analyzed in a Magnetic Nuclear Resonance spectrophotometer (Varian, mod. Mercury 200). The frequency of 50 and 200 MHz was used for ^{13}C NMR and ^{1}H NMR, respectively.

2.6. Evaluation of the Thermal Stability of Oil

The evaluation of the thermal stability of *sterculia striata* kernel oil was determined through the thermogravimetric (TG) and Differential thermogravimetric (DTG) curves in a thermal analyzer Shimadzu model TGA-50 in the atmosphere of air with a flux of 50 ml/ min and a heating rate of 10 °C/min. Ten milligrams of oil sample were subjected to a temperature range of 30 to 600 °C. The thermal stability was determined by the analysis of thermograms that registered a loss in mass of oil during the heating period.

For differential calorimetric analysis about 10 milligrams of oil sample were subjected to heating at a rate of 10 °C/min in a Shimadzu Thermal differential calorimeter DSC-50 in an atmosphere of nitrogen. The sample was heated to 500 °C. The nitrogen atmosphere was maintained with a nitrogen flux of 50 ml/min.

2.7. Statistical Analysis

For the statistical analysis, the Statistical Analysis system (SAS) version 6.12 (SAS Institute, 1996) was used.

3. RESULTS AND DISCUSSION

A proximate analysis is of utmost importance to provide information on the basic constituents of a particular raw material. The results of the analysis of the dried kernel powder of *sterculia striata* are shown in Table 1. These results show that dried kernel powder is a good source of lipid (25.8%) and proteins (17.9%) similar to other oleaginous seed kernels that are rich in these constituents. In earlier works, Araújo (1997) and Oliveira *et al.* (2000) reported the lipid content of 30.2 and 28.2% respectively, and protein content of 21.3 and 22.5%, respectively for the kernel powder of *sterculia striata*. However, this small difference in the protein and lipid contents between our work and that of the authors cited may be explained by the difference in the moisture content of the kernel powder, agricultural practices and geographical region.

The oil extracted from the kernel powder was odorless and light yellow in color. The physicochemical properties of the oil are shown in Table 2. The acid value of the crude oil was 0.82% as oleic acid that is within the range 0.3 to 2.0% established by the Ministry of Health (Brazil, 1998). Though the acid value is caused by the hydrolytic rancidity of the seed, it could be easily minimized during refining of the oil. The saponification value of the oil was about 136 which is indicative that the oil contained fatty acids of higher molecular weight on average. The oil, for this reason, is not appropriate for soap manufacturing. The oil also presented a low iodine (69.2) value suggestive of the presence of significant amounts of saturated fatty acids.

The fatty acids composition of the sterculia striata kernel oil showed the presence of 16 to 24 carbon-containing fatty acids. The saturated fatty acids (Table 3) constituted 36.2% of the total fatty acids among which palmitic acid was the dominant one (31.9%) followed by stearic (4.34%) acid. Aued-Pimental *et al.* (2004) reported palmitic and stearic acids at concentrations of 20.8 and 2.8% of the total fatty acids, respectively, while Miralles *et al.* (1993) observed these two saturated fatty acids at concentrations of 20-24 and 3.8- 6.0%, respectively.

Monounsaturated fatty acids were present in the highest concentration (43.7%) composed of oleic

Table 1
Proximate analysis of Chichá (*Sterculia striata*) seed kernel [a]

Constituents	(%)
Moisture	11.02 ± 0.04
Protein	17.98 ± 2.11
Lipids	25.82 ± 1.44
Ash	3.42 ± 0.02
Carbohydrates (by diff.)	41.71 ± 0.12

[a] The results show the media and standard deviation of the analysis of three lots of dried and powdered *Sterculia striata* seed kernels.

Table 2
Physicochemical properties of Chichá (*Sterculia striata*) seed kernel [a]

Property	Value
Specific density (25°C)	0.8532 ± 0.0002
Refractive index (25°C)	1.4625 ± 0.0007
Acid value (oleic acid %)	0.82 ± 0.04
Iodine value (g of I_2/100g oil)	69.1 ± 0.1
Peroxide value (mEq.g/kg oil)	4.2 ± 0.2
Saponification value (mg KOH/g oil)	136.0 ± 1.0

[a] The results show the media and standard deviation of the analysis of three lots of *Sterculia striata* seed kernel oil.

Table 3
Fatty acid composition of Chichá (*Sterculia striata*) seed kernel oil [a]

Fatty acid	(%)
Saturated fatty acids	36.24
Palmitic acid	31.90 ± 0.07
Stearic acid	4.34 ± 1.04
Monounsaturated fatty acids	43.73
Palmitoleic acid	2.00 ± 1.02
Oleic acid	41.73 ± 2.15
Cyclopropenoid fatty acids	7.66
Sterculic acid	5.32 ± 1.08
Malvalic acid	2.34 ± 0.32
Polyunsaturated fatty acids	12.36
Linoleic acid	10.73 ± 0.49
Linolenic acid	0.89 ± 0.12
Arachidonic acid	0.74 ± 0.04

[a] The results show the average value and standard deviation of the analysis of three oil samples.

(41.7%) and palmitoleic (2.0%) acid. Aued-Pimental *et al.* (2004) reported 30.2 and 2.1% oleic and palmitoleic acid in the *sterculia striata* oil they have analyzed but Miralles *et al.* (1993) reported a low value (15-21%) for oleic acid. In our study, linoleic, linolenic and arachidonic acids were the polyunsaturated fatty acids present at concentrations of 10.73, 0.89 and 0.74% respectively. Miralles *et al.* (1993) reported higher linoleic acid concentrations (16-30%)

The cyclopropenoid fatty acids (CPFA) were tentatively identified by comparing their spectra with that of the software provided together with GC/MS (Varian). However, the presence of these CPFA was confirmed by Nuclear Magnetic Resonance. In ^{1}H NMR, the scanning region varied between δ 0.5 to 1.5 of the spectra. The spectra initially exhibited a singlet in δ 0.78 indicating two hydrogen of the cyclopropenoic (CH_2) ring; two duplets at δ 0.84 attributed to the two hydrogen of the terminal methyl group (CH_3-CH_2) and a triplet at δ 0.87, corresponding to the proton of the methyl group of the methyl esters of the fatty acids. The duplet at δ 5.3 is suggestive of the presence of a proton of the hydroxyl (-O-H) group. In ^{13}C NMR, the oil revealed the presence of a mixture of steroids. Among other signals, the spectra also signalled a double bond of CH_2=CH_2 at δ 107.9 of the cyclopropene ring. In a total of 7.65% concentration of cyclopropenoid fatty acids, two acids were identified in the oil- sterculic (5.32%) and malvalic (2.33) acid. An almost similar concentration (8.1%) of cyclopropenoid fatty acids in sterculia striata oil was also reported by Aued-Pimental *et al.* (2004). Earlier Miralles *et al.* (1993) observed a concentration of 11.3 to 30.2% sterculic acid and 0.9 to 5.0% dihydrosterculic acid in the oil of the seeds of sterculeaceas. These authors detected the presence of two more cyclopropenoid acids that were not identified.

The presence of CPFA has also been detected in cottonseed oil that is used for human consumption. When these acids are present in low concentrations, they are eliminated during the refining process due to their sensibility to heat (Aued-Pimental *et al.,* 2004). However, these fatty acids are responsible for certain physiological disorders in animals, especially for their co-carcinogenic activity (Park and Rees, 1988). The fact that no ill effect has so far been reported in the local population of the region that consume the roasted seed kernel of *Sterculia striata*, may possibly be due to the destruction of cyclopropenoid fatty acids during roasting process.

The process of thermal stability in vegetable oils is characterized initially by the oxidation forming secondary products – peroxides. This behavior is characterized by the increase in mass. The following phase corresponds to the decomposition of monounsaturated fatty acids, mainly oleic acid and the formation of the polymerization of the substances remaining from the previous phase. The thermal stability in terms of thermogravimetry (TG) and differential thermogravimetry (DTG) with respect of the loss in mass of the *sterculia striata* oil is shown in Figure 1. The process started at 28 °C and finished at 600 °C at a rate of 10 °C per min. The curves of TG and DTG presented a typical profile with loss in mass occurring in three stages. The first one is considered more important and represents the initial phase of the degradation of triglycerides, mainly composed of the poly-unsaturated fatty acids. In this phase the oxidation of the poly-unsaturated fatty acids take place. According to the differential thermogram, the oxidation of poly-unsaturated fatty acids started at about 284 °C and reached its peak value at 392 °C. The second and third phases represent the decomposition of monounsaturated and saturated fatty acids, respectively. High temperatures catalyze the reactions of hydrolysis and oxidation of oils (Dobarganes & Perez-Camino, 1991). The products of these reactions react among themselves and produce cyclic monomers, dimers, and polymers.

The thermogravimetric profile (Fig. 1) of *Sterculia striata* kernel oil also shows that the oil was stable up to a temperature of 284.7 °C, above that, temperature started producing a loss in mass. The stability could be attributed to an expressive quantity of saturated fatty acids (37.2%) in the oil. The presence of antioxidants improves oil stability (Santos *et al.,* 2002). Normally, oil that contains high concentrations of unsaturated fatty acids is more susceptible to thermal deterioration. However, Warner *et al.* (1986), reported that several other vegetable oils that contain a high concentration of unsaturated fatty acids possess stability equivalent to other oils with lesser instauration showing that there are other factors which influence the thermal stability of vegetable oils.

The Differential Scanning Calorimetric (DSC) profile of the oil (Fig. 2) shows the existence of two events of exothermic energy transitions, one is related to the oxidation reactions and another to the decomposition of the oil. First energy transition was

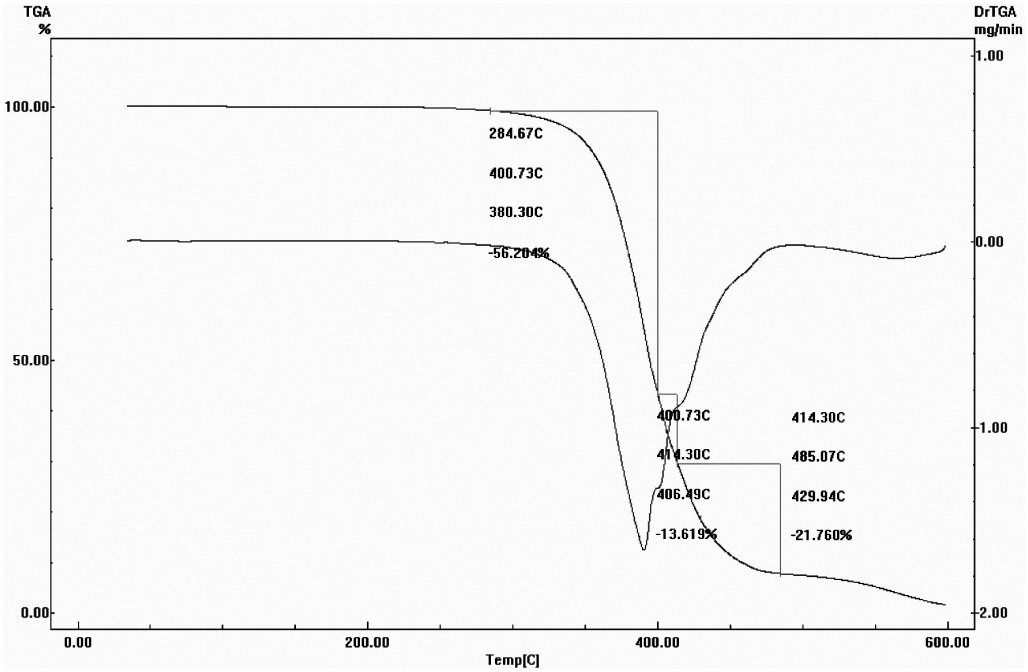

Figure 1
Thermogravimetric and differential thermogravimetric profile of *Sterculia striata* seed kernel oil.

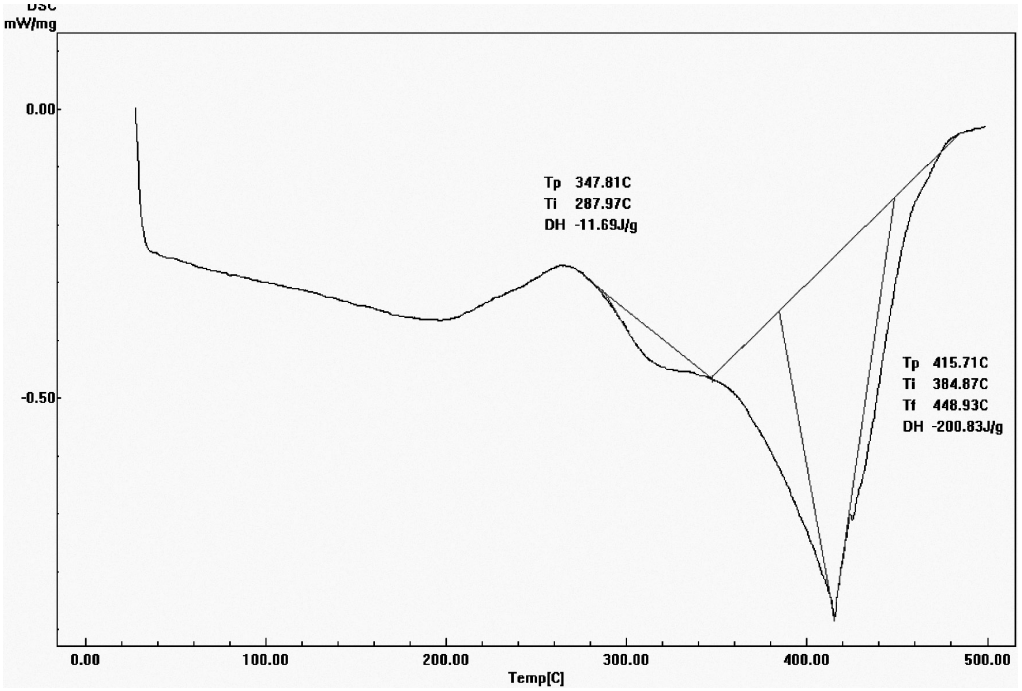

Figure 2
Differential Scanning Colorimetric profile of S*terculia striata* seed kernel oil.

initiated at a temperature (Ti) of 287.79 °C, and terminated at 347.81 °C with a variation in enthalpy of –11.69joules.g^{-1}. In the second transition, this manifestation was observed at an initial temperature (Ti) of 384.87 °C peak temperature 415.71 °C, final temperature 448.9 °C and an enthalpy of –200.83 Joules. g^{-1}. The values of the enthalpy of activation are negative because the oil is receiving heat for its oxidation and decomposition. In any case, the bigger the size of the hydrocarbon chain, the higher the enthalpy of activation is. The presence of antioxidants in the oil

also influences its energy of activation through the induction of enthalpy (Simon *et al.,* 2000). These authors observed that the enthalpy of oils containing antioxidants requires enthalpy of decomposition superior to oils that do not contain antioxidants. Kasprzycka-Guttman and Coziniak (1995) reported that the thermal decomposition of saturated fatty acids requires more energy than the unsaturated fatty acids. Thus the first event corresponds to the polymerization of a part of the unsaturated fatty acids and the second, to the polymerization of saturated fatty acids and the unsaturated fatty acids reminiscent of the first phase. Olive, canola and rice oils present only one endothermic event possibly due to high concentrations of monounsaturated fatty acids whose enthalpy was superior to other oils.

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Recibido: 19/11/07
Aceptado: 17/12/07