CHEMICAL CHARACTERIZATION OF JATROPHA OIL STORED OVER A PERIOD OF TIME AND ANALYSIS OF ITS PRESSED CAKE

Caracterização química do óleo de pinhão manso (Jatropha curcas) após período de armazenamento e da sua torta

> Roberto Ananias Ribeiro¹ Maria das Graças Mota Nobre Queiroz² Vera Lúcia Alves³ Emille Rocha Bernardino de Almeida Prata⁴ Érica Soares Barbosa⁵

Abstract: **Objective**: In this paper, the chemical properties of Jatropha oil and cake were investigated in order to determine the characteristics this plant. **Methods**: A mechanical expeller was used to obtain oil which was filtered and degummed with phosphoric acid. Jatropha oil and cake were characterized by chemical analysis. **Results**: Initially, lower values in measurement of acid, iodine, saponification and peroxide values were obtained. After five months of storage, modifications occurred in the oil so that the values increased, demonstrating oil oxidation. Jatropha cake presented an amount of crude fiber and crude protein equals to 21.1 and 20.9 (%w/w), respectively, and mineral elements, Ca, Mg and P, in relatively high amounts, 0.42, 0.54 and 0.71 (%w/w), respectively. **Conclusions**: Crude Jatropha oil presented low acid and peroxide values.

Key words: Jatropha curcas. Oil. Cake. Chemical analysis.

Resumo: Objetivo: Nesse trabalho, as propriedades químicas do óleo e da torta foram investigadas a fim de determinar as características de uma planta cultivada em uma região brasileira. **Metodologia:** Uma prensa mecânica foi usada para obter o óleo que foi filtrado e degomado com ácido fosfórico. O óleo e a torta foram caracterizados por análise química. **Resultados:** Inicialmente, valores menores dos índices de iodo, saponificação e peróxido foram obtidos. Após cinco meses de armazenamento, ocorreram modificações no óleo de maneira que os valores aumentaram, demonstrando a oxidação do mesmo. A torta apresentou uma quantidade de fibra bruta e proteína bruta igual a 21,1 e 20,9 (%m/m), respectivamente, e os elementos minerais Ca, Mg e P, em quantidades relativamente altas, 0,42, 0,54 e 0,71 (%m/m), respectivamente. **Conclusões:** O óleo do Jatropha apresentou valores baixo de acidez e peróxido.

Palavras-chave: Pinhão-manso. Óleo. Torta. Análise química.

3 Professora da Unimontes.

¹ Doutor em Química pela Universidade Federal de Minas Gerais - UFMG. Professor da UFMG.

² Mestre em Educação com Ênfase em Química pelo Instituto Superior José Henrique Verona. Professor da Unimontes.

⁴ Doutoranda em Ciência e Tecnologia de Alimentos pela Universidade Federal de Viçosa - UFV.

⁵ Mestre em Ciências Agrárias pela Universidade Federal de Minas Gerais - UFMG.

Montes Claros, v. 15, n. 2 - jul. 2013. (ISSN 2236-5257)

INTRODUCTION

Conversion of biomass to biofuels supplies energy and such use of vegetable wastes is beneficial in terms of the environment¹. Biomass such as sugar, starch, vegetable oils and animal fats are raw materials used to produce biofuels. Some edible oils come from soybean, sunflower, canola and palm, whereas feedstock such as *Jatropha*. *curcas*, *Pongamia*. *pinnata*, *Madhuca*. *indica*, etc. are considered non-edible oils for biodiesel production². *Jatropha curcas* is an oilseed plant with potential for being used as raw material in the production of a kind of oil that yields high-quality biodiesel³⁻⁵.

Jatropha oil is obtained through hydraulic pressing of seeds⁶ or by extraction using solvents⁷. The extraction of the Jatropha oil was performed through enzymes, obtaining significant results with alcalase and viscozyme⁸. Nevertheless, lower yields were obtained through aqueous enzymatic oil extraction with Protizyme^{TM 9}.

Studies have verified many possibilities of use of Jatropha fruit; it's seed, cake, husk and shell¹⁰. The results showed that the blending of diesel and extracted oil seeds may be used in diesel engines and its cake is a good feedstock for biogas production and shell for combustion¹¹. In addition, Jatropha fruit can be used to obtain some other products, such as, charcoal from the wood and nutshell, fertilizer from the seed cake¹², soap, extraction of tannin, etc¹³. The oilcake is toxic to animals due to the presence of phorbol esters¹⁴ but treatment of the material at 260°C leads to its degradation¹⁵. The production of activated carbon from Jatropha oil waste showed results that suggest the sustainable usage of this plant as an alternative fuel¹⁶.

The quality of biodiesel depends on the oil used for transesterification since the properties of biodiesel are markedly influenced by properties of the fatty esters¹⁷. Hence, in this paper, Jatropha oil and cake chemical properties were described.

MATERIAL AND METHODS

Jatropha seeds were supplied by the company Biojan Agro Indústria Ltda, located in Janaúba city, Northern Minas Gerais State, Brazil. Jatropha oil was extracted using a mechanical expeller then filtered. The oil was degummed using 1% phosphoric acid and 3% distilled water (w/w) under agitation at 75-80 °C for 30 min. The mixture was then centrifuged, neutralized with sodium hydroxide and washed with water. After separation of the phases, the oil was dried in a stove at 80 °C for 48 h. The oil was stored in a dark glass bottle at room temperature (around 30 °C).

Chemical characterization was done according to the Adolfo Lutz Institute Analytical Norms¹⁸. The analyses realized considered acid, iodine, peroxide and saponification values for oil and mineral elements for the cake.

RESULTS AND DISCUSSION

The chemical properties of the oil are shown in Table 1.

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Table 1 Chemical characteristics of Jatroph	a oil
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Properties	Crude oil	Degummed oil
Acidity (mg KOH/g)	2.3±0.2	2.0±0.2
Iodine (g iodine/100 g)	63±3	57±5
Peroxide (meq/kg)	2.4±0.0	167±2
Saponification (mg KOH/g)	193±4	197±1

Means ± standard deviation for samples in triplicate

The results showed that the oil presented lower acid and iodine values whereas saponification values were according to the literature. Samples of oil from Ekiti State, Nigeria, extracted with n-hexane, presented acid, saponification and iodine values equal to 3.5 ± 0.1 mg KOH/g, 198.85 ± 1.40 mg KOH/g and 105.2 ± 0.7 mg iodine/g, respectively⁷. Saponification and iodine values obtained by Prasad et al. $(2012)^{19}$ were 189.2 mg KOH/g and 97 g iodine/100 g, respectively, whereas Patil and Deng $(2009)^{20}$ found 196 mg KOH/g for saponification values. Autoxidation reaction was confirmed through peroxide values as a result of the presence of unsaturated chains in fatty compounds⁴.

Comparison of chemical properties of crude Jatropha oil from different Brazilian regions have demonstrated that the oil extracted from seeds cultivated in Janaúba presented lower acid and peroxide values. Campos et al. (2010)²¹ obtained acid and peroxide values at 27 °C, respectively, equal to 5.18 mg KOH/g and 6.67 meq/kg. Four months of storage revealed higher values for these properties: acid and peroxide, with values that reached 6.41 ± 1.40 and 5.53 ± 1.99 , respectively. Different results of acid values were obtained of samples from Viçosa city²². Acid value equal to 3.9 mg KOH/g was found and after six months of storage this value increased to 5.0 mg KOH/g. Iodine value decreased from 110 to 80 g iodine/100 g in this period.

The results obtained in this paper for degummed Jatropha oil showed that the degumming process contributed to the high level of peroxide²³.

Modifications in Jatropha oil chemical properties were evaluated over five months and the results are presented in Figures 1 and 2.

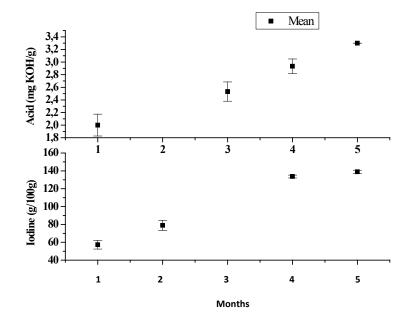


Figure 1- Modifications in the acid and iodine values of Jatropha oil over five months.

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Montes Claros, v. 15, n. 2 - jul. 2013. (ISSN 2236-5257)

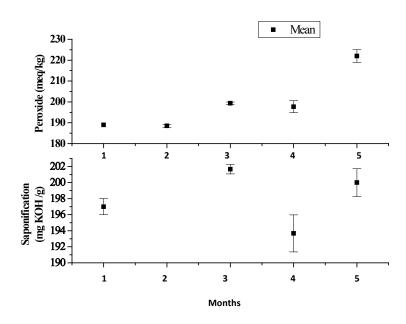


Figure 2- Modifications in the peroxide and saponification values of Jatropha oil over five months.

The acid values increased during the investigated period. Triglyceride molecule degradation releases fatty acid molecules which support the reaction between iodine and unsaturated chains; consequently, higher iodine values were verified in the samples.

According to Figure 2, no statistical difference (p < 0.05) was observed in the peroxide values between the first and the last two months. On the other hand, between the second and third or the fourth and six months, statistical differences

were verified. These results show that the oxidation process requires time to occur, reaching elevated values after a month. This may be connected with the autoxidation process, in which reactions are slow²³. Molecule degradation was observed on saponification values as they increased in the first month one and decreased in the next ones. At the 0.05 level of significance, the means were statistically different.

The analysis of Jatropha cake is presented in Table 2.

Chemical analysis	% w/w	Mineral elements	% w/w
Crude fibre	21.1±0.6	Р	0.71±0.06
Crude protein	20.9±1.4	Ca	0.42
Crude fat	15.8	Mg	0.54
Total humidity	8.3	Fe	0.0092
Ash	5.9	Cu	0.0026
-	-	Zn	0.0041

Table 2 Chemical characteristics of the Jatropha cake

Means ± standard deviation for samples in triplicate, other quantities for samples in duplicate

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Staubmann et al. (1997)¹¹ and Winkler et al. (1997)⁸ analyzed Jatropha cake (seed with shells) obtaining an amount (% w/w) of crude protein and crude fiber that was equals to 24.54 and 32.26 and 17.08 and 22.96, respectively. The value of 6.03 for ashes determined by Staubmann et al. (1997)¹¹ was also reached in our work. Since mechanical pressing was used to obtain oil, the amount of crude fat indicated that the extraction process needs to be improved in order to reach a higher yield. The measurement of mineral elements revealed elevated content of calcium, magnesium and phosphorus in Jatropha cake.

CONCLUSIONS

Over five months of storage Crude Jatropha oil presented low acid and peroxide values. Degummed oil showed low acid values, saponification values according to current literature, elevated level of peroxide values and higher iodine values. The chemical characterization of Jatropha cake revealed an amount of crude protein and crude fiber that accords to literature and a relatively higher amount of mineral elements Ca, Mg and P.

ACKNOWLEDGMENTS

We would like to thank Fundação de Amparo à Pesquisa do Estado de Minas Gerais (FAPEMIG) for their support and Conselho Nacional de Desenvolvimento Científico e Tecnológico (CNPq) for paying for this study.

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 Table 1- Chemical characteristics of Jatropha oil

 Properties
 Crude oil
 Degummed oil