

**Chemical prospection of moringa oil and bromatological quality of the pie from
different types of grain processing**

**Prospecção química do óleo de moringa e qualidade bromatológica da torta,
provenientes de diferentes tipos de processamentos dos grãos**

**Prospección química de aceite de moringa y calidad bromatológica de la empanada, de
diferentes tipos de procesamiento de granos**

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Bárbara Lemes Outeiro Araújo

ORCID: <https://orcid.org/0000-0001-5350-5174>

Universidade Federal de Lavras, Brazil

E-mail: barbara@oleo.ufla.br

Ednilton Tavares de Andrade

ORCID: <https://orcid.org/0000-0002-8448-8781>

Universidade Federal de Lavras, Brazil

E-mail: ednilton@deg.ufla.br

Jaqueline Damiany Portela

ORCID: <https://orcid.org/0000-0003-3944-3928>

Universidade Federal de Lavras, Brazil

E-mail: jaqueline-portela@outlook.com

Rafael Peron Castro

ORCID: <https://orcid.org/0000-0002-0063-2302>

Universidade Federal de Lavras, Brazil

E-mail: peron@oleo.ufla.br

Pedro Castro Neto

ORCID: <https://orcid.org/0000-0002-3836-5553>

Universidade Federal de Lavras, Brazil

E-mail: oleo@deg.ufla.br

Abstract

Moringa oleifera Lam. is a drought-resistant plant and able to survive in poor soils, obtaining up to three harvests per year. The objective of this work was to study the chemical

prospecting of the oil and the bromatological quality of the moringa cake among different temperatures of drying (40, 55, and 70 °C) as well as from the oil chemical and mechanical extraction methods. The extracted oils were qualitatively evaluated for acidity, peroxide and iodine levels, as well as the chemical composition of fatty acids by gas chromatography, of samples dried at different drying air temperatures. The pies from mechanical extractions were evaluated for water content, ether extract, crude protein, ash and fibers in neutral detergent. The drying air temperatures of 40, 55, and 70 °C significantly affected the physical-chemical quality of the oil and the moringa cake, with the best result being the samples from the dry grains at 40 °C. The composition of the main fatty acids was not altered according to the statistical method applied, these being oleic fatty acid (73.60 to 77.07%), erucic (5.65 to 6.67%) and palmitoleic (4.90 to 5.72%). The chemical extraction of oil, although more efficient than the mechanical one, presented higher levels of acidity and peroxide. The content of fibers in neutral detergent and crude protein of the pie decreased significantly for dried grains with drying air temperature above 40 °C.

Keywords: Drying; Oil extraction; Fatty acids.

Resumo

A *Moringa oleifera* Lam. é uma planta resistente a seca e capaz de sobreviver em solos pobres, obtendo até três colheitas por ano. Esta cultura possui ampla empregabilidade, seus grãos possuem alto teor lipídico e proteico. Assim, o objetivo deste trabalho foi estudar a prospecção química do óleo e qualidade bromatológica da torta de moringa, provenientes da secagem dos grãos nas temperaturas do ar de 40 °C, 55 °C, 70 °C, como também dos métodos de extração de óleo, químico e mecânico. Foram utilizados grãos de *Moringa oleifera* Lam. pré-secos, provenientes da cidade de Barreirinhas (MA), da safra do segundo semestre de 2018, e o experimento foi conduzido no Departamento de Engenharia Agrícola da Universidade Federal de Lavras (MG). A secagem artificial foi feita até a massa constante dos grãos, por meio da utilização de um secador mecânico de laboratório em camada fixa com convecção forçada, na velocidade do ar de 0,33 m.s⁻¹, com temperaturas controladas do ar de secagem de 40 °C, 55 °C, e 70 °C. A extração do óleo foi feita pelo método mecânico, utilizando prensa, tipo expeller e, químico com solvente orgânico hexano, com o equipamento tipo soxhlet. O rendimento e a eficiência da prensa foram calculados a partir da diferença do teor lipídico obtido pela extração química inicial do grão e residual da torta. Os óleos extraídos foram avaliados qualitativamente quanto aos índices de acidez, peróxido e iodo, como também foi feita a composição química dos ácidos graxos por cromatografia gasosa,

das amostras secas nas diferentes temperaturas do ar de secagem. As tortas provenientes das extrações mecânicas, foram avaliadas quanto ao teor de água, extrato étereo, proteína bruta, cinzas e fibras em detergente neutro. As temperaturas do ar de secagem de 40 °C, 55 °C, 70 °C afetaram significativamente a qualidade físico-química do óleo e da torta de moringa, tendo como melhor resultado as amostras provenientes dos grãos secos à 40 °C. A composição dos principais ácidos graxos não foi alterada de acordo com método estatístico aplicado, sendo estes o ácido graxo oléico (73,60 à 77,07%), erúico (5,65 à 6,67%) e palmitoléico (4,90 à 5,72%). A extração química de óleo, apesar de mais eficiente que a mecânica, apresentou maiores índices de acidez e peróxido. O teor de fibras em detergente neutro e proteína bruta da torta, diminuiu significativamente para os grãos secos com temperatura do ar de secagem acima de 40 °C.

Palavras-chave: Secagem; Extração de óleo; Ácidos graxos.

Resumen

Moringa oleifera Lam. es una planta resistente a la sequía y capaz de sobrevivir en suelos pobres, obteniendo hasta tres cosechas por año. El objetivo de este trabajo fue estudiar la prospección química del aceite y la calidad bromatológica de la torta de moringa a diferentes temperaturas de secado (40, 55 y 70°C) así como de los métodos de extracción química y mecánica del aceite. Los aceites extraídos se evaluaron cualitativamente para determinar los niveles de acidez, peróxido y yodo, así como la composición química de los ácidos grasos mediante cromatografía de gases, de muestras secadas a diferentes temperaturas del aire de secado. Se evaluó el contenido de agua, extracto etéreo, proteína cruda, cenizas y fibras en detergente neutro de los pasteles de extracciones mecánicas. Las temperaturas del aire de secado de 40, 55 y 70 °C afectaron significativamente la calidad físico-química del aceite y la torta de moringa, siendo el mejor resultado las muestras de los granos secos a 40 ° C. La composición de los principales ácidos grasos no se alteró según el método estadístico aplicado, siendo estos ácidos grasos oleico (73,60 a 77,07%), erúico (5,65 a 6,67%) y palmitoleico (4,90 a 5,72%). La extracción química del aceite, aunque más eficiente que la mecánica, presentó mayores niveles de acidez y peróxido. El contenido de fibras en detergente neutro y proteína cruda de la tarta disminuyó significativamente para los granos secos con temperatura del aire de secado superior a 40 ° C.

Palabras clave: Secado; Extracción de aceite; Ácidos grasos.

1. Introduction

Moringa oleifera Lam. is a plant, also known as white wattle, tolerant to a wide pH range (5 to 9), reaching up to 4 m in height in a year and, as an adult, obtains an annual production of 3 to 5 tons of seeds per hectare. It grows quickly, being able to survive in poor soils and quite resistant in long periods of drought (Gopalakrishnan, Doriva, & Kumar, 2016; Gandji, Salako, Fandohan, Assogbadjo, & Kakai, 2018)

Moringa grains have approximately 40% oil, with a high percentage of oleic acid, around 78% (Ojewumi, 2018; Oladipo & Betiku, 2019; Mat Yusoff, Niranjana, Mason, & Gordon, 2020). In addition, the oil has excellent oxidative stability, with the presence of δ -tocopherol, aiding in preservation during processing and storage. It is widely used in industry to lubricate watches, delicate machinery, in the manufacture of perfumes, biodiesel, as its antioxidant effect has also been studied when added to others oils (Fitriana, Ersam, Shimizu, & Fatmawati, 2016; Boukandoul, Casal, Cruz, Pinho, & Zaidi, 2017; Atolani, Olorundare, Anoka, Osin, & Biliaminu, 2018; Nadeem et al., 2018; Valenga, Boschen, Rodrigues, & Maia, 2019).

The flour resulting from the extraction of oil from the moringa grains can be used either as a coagulant in water purification, or in supplementing poultry feed and producing fertilizers, as it has a high protein content, which can vary from 26.5 to 32%. This oilseed is a vegetable with multiple applications, favoring the sustainable use of the species, which can generate financial and social benefits in the places where it is grown (Garza et al., 2017; Agarwal, Dixit, & Bhatt, 2019; Leone et al., 2016).

Despite the wide employability of this crop and the growth of its cultivation, present in more than thirteen states in Brazil, there are few studies on the processing of these grains and the maintenance of their quality, which is directly related to their chemical composition. Among the stages of moringa processing, drying and oil extraction have a greater influence on the properties of the final products. Higher drying air temperatures can speed up the process of removing water from the grains and make it more economical (Dziki & Gawlik-Dziki, 2019). However, they can cause physical and chemical changes in these, and consequently the devaluation of oil and cake. Several researches have shown that temperatures, in the mass of different agricultural products, above 40 °C can cause quality losses (Alves et al., 2017; Andrade, Lemos, Dias, Rios, & Borém, 2019; Cheng et al., 2019).

In view of the above, the objective of this work was to study the chemical prospection of the oil and the bromatological quality of the moringa cake, derived from drying the grains at air temperatures of 40, 55, and 70°C, through chemical e mechanical extraction methods.

2. Materials and Methods

The scientific method used in this experiment was laboratory research using the quantitative method. According to Pereira et al. (2018) in this method, the collection of numerical data is promoted through the use of measurements of quantities that generate data sets which are analyzed by mathematical techniques such as applicable equations for process description and statistical analysis.

The experiment was carried out at the Federal University of Lavras (UFLA). Grains of *Moringa oleifera* Lam. pre-dried, from the city of Barreirinhas (MA), from the harvest of the second semester of 2018. The drying was done in the Laboratory of Processing of Agricultural Products and the extractions of oil and analyzes in the Laboratory of Oil Plants, Oils, Fat and Biodiesel, both in the Department of Agricultural Engineering of this institution.

The beans were harvested shortly after reaching the point of physiological maturity, which is identified by the dark brown color of the pods (Agustini, Wendt, Paulus, Malavasi, Gusatto, 2015), from which they were manually removed and subsequently passed through pre-cleaning.

Artificial drying was carried out to a constant grain mass, using a mechanical laboratory dryer in a fixed layer with forced convection, at an air speed of 0.33 m/s, with controlled drying air temperatures of 40, 55, and 70 °C. The water content of the samples was made before and after drying, according to the recommendations of the Rules for Seed Analysis (Brasil, 2009), where the greenhouse method at 105 ± 3 °C was used for 24 hours. The experiment consisted of triplicates, each with a mass of approximated 1.7 kg, dried at different temperatures.

Mechanical oil extraction was performed using a press type expeller, with tubular radial extraction system, model ERT 50. Samples of oil and cake were collected for each qualitative analysis. The grains were passed through the extractor for three consecutive times, with four different samples: dry grains at 40, 55, and 70 °C and the control, which did not undergo the drying process.

The oil content was made by chemical extraction by the Soxhlet method, with hexane organic solvent (AOCS, 1998). The mechanical extraction yield was calculated from the

difference in the lipid content obtained by the initial chemical extraction of the grain and residual of the cake, according to Equation 1, since the hexane solvent extraction method was determined universal for oilseeds, by have practically total efficiency (Lusas, Watkins, & Koseoglu, 1991). The oils extracted by the different methods were qualitatively evaluated for acidity, peroxide and iodine levels according to the methodology of the Instituto Adolfo Lutz (2008).

The oil samples obtained by chemical extraction were subjected to determination of the composition of fatty acids from the analysis of methyl esters, in order to verify the effects of drying air temperature on the quality of the extracted oil. The oil samples were esterified and, subsequently, 10 µl aliquots were removed and diluted in 1 ml of hexane and analyzed in a GC2010 SHIMADZU gas chromatograph, equipped with a flame ionization detector (FID), an automatic injector and a SP 2560 column (100 m X 0.25 mm X 0.20 µm). The initial oven temperature was 150 °C, and remained for 5 min. Then, the temperature was raised to 240 °C, with a heating rate of 15 °C/min, remaining at that temperature for another 5 minutes (IAL, 2008). FAME 37 (Fatty Acid Methyl Esters) was used as a standard. The catalytic tests were carried out at the Chemical Analysis and Prospecting Center (CAPQ) of the Department of Chemistry at the Federal University of Lavras.

The pies from the mechanical extractions of the different drying air temperature treatments were quantitatively evaluated for water content, ether extract, crude protein, ash and fibers in neutral detergent, according to Silva (1990).

The extractions and analyses were performed in three replications. The experimental design adopted was randomized blocks. The data obtained were subjected to analysis of variance and the means were compared with each other by the Tukey test, ($P < 0.05$), using the Sisvar program (Ferreira, 2014).

3. Results

Samples of moringa grains processed at different drying air temperatures and oil extraction methods. Their main indices that determine oil quality are shown in Table 1. According to low efficiency in the mechanical extraction of oil from dried grains at 70 °C and excess sludge contained therein, there was not enough quantity for qualitative analyses.

The temperature of the drying air, as well as the extraction method, significantly affected the acidity of the moringa oil. For the mechanical method, the acidity index increased, from dried grains at 40 to 55 °C, respectively, from 1.17 to 2.18mg NaOH/g. The

values in this quality parameter, were not significant between the drying treatments, in the chemical obtaining of the oil, however it was superior to that extracted using a press, dried at 40 °C. The extraction method with hexane organic solvent, has been shown to be more efficient compared to other technologies for obtaining greater yield, since the sample remains in contact with it, until the total removal of the oil.

Moringa oil had the lowest value of peroxide index, the sample of dried grains at 40 °C and extracted by the mechanical method had 1.94 meq/kg. The chemical extraction of the oil, associated with the heating and drying time of the product, had expressive values of peroxides. According to ANVISA Resolution No. 482/1999, the maximum limit for the peroxide index for edible oils is 10 meq/kg. Oxidation processes in foods are responsible for reducing the shelf life of raw materials and industrialized products, leading to significant economic loss.

The degree of unsaturation of the oil can indicate the tendency to oxidize it, so the iodine index is used to predict the presence of double bonds in a fatty acid ester. For this, there was no significant variation in the methods of oil extraction and drying treatments, with the exception of the sample dried at 70 °C. Table 2 presents the fatty acid composition of the moringa oils extracted by the chemical method, from the samples of the dried grains at different drying air temperatures.

Table 3 shows the quality analysis of the pies from the dried moringa grains at different temperatures of the drying air. The elevation of the drying air temperature of the moringa grains significantly influenced the nutritional parameters of the cake. The extraction of oil by mechanical press, resulted in high extract value ether, due to its low efficiency, for dried grains above 55 °C. Crude protein lost more than 3% in pies from high temperatures in the drying process. The husks of the moringa beans are rich in fibers, making the percentage of this in the cake to be high. There was a significant variation of NDF in the moist moringa pies to the dried grains at temperatures of 55 and 70 °C, decreasing from 37.45% to 32.24% and 27.78%, respectively.

4. Discussion

The acidity index is one of the main quality parameters for the acceptability of vegetable oils, as it determines the amount of free fatty acids present. This, if high, can indicate chemical changes, compromising the use of oil for both food and fuel purposes (Zeeshan, Vasudeya, & Sarma, 2016; Oyeyinka & Oyeyinka, 2018).

According to the requirements of the food and biofuels industry, for oil use, this index must be equal to or less than 1.0 mg NaOH/g, so that there is no need for treatment of the product, which usually demands time, costs and waste generation (Candeia et al., 2009; Rodrigues Filho & Souza, 2009).

The peroxide index represents another important indicator of quality, as it indicates the degree of oxidation of the oil. Changes in its sensory characteristics are generally attributed to the presence of peroxides in the grease matter (Nour & Ibrahim, 2016). Aspects such as seasonality of oilseeds or different types of processing, can change the degree of oil installation (Matt Yussof et al., 2020).

Vegetable oils contain different types of fatty acids which, depending on the length of the chain and degree of unsaturation, may represent the parameter of greatest influence on the properties of these compounds (Knothe, 2005).

Oils rich in oleic acid and saturated fatty acids have high oxidative stability, being resistant to thermal processing and long storage periods (Nguyen et al., 2011; Leone et al., 2016). Andrade, França, Ramos, and Silva (2016) proved the efficiency of biodiesel from *Moringa oleifera* Lam. As a natural antioxidant, adding 2000 ppm to soy biodiesel, increasing its oxidative stability. The moringa grains submitted to different drying air temperatures, had no significant difference in the composition of the main fatty acids that make up the oil.

Most proteins are denatured when exposed to moderate heating, around 60 to 90 °C, for an average of one hour (Araújo, Galvão, Miranda, & Araújo 2002; Carvalho *et al.*, 2009). Excessive denaturation results in its insolubilization if consumed, as it affects its functional properties and increases its viscosity.

The percentage of proteins presented in the moringa cake is of commercial importance, since its use stands out as a natural coagulant in water treatment, having efficiency similar to aluminum sulfate, being a low cost technology and favouring a more environmentally acceptable purification (Elghandour et al., 2017; El-Naggar, 2018; Mahmoud, 2019). Pitkin et al. (2019) corroborated the antimicrobial effect of the moringa coagulant protein, tested on *Escherichia coli*, *Pseudomonas aeruginosa*, and *Bacillus thuringiensis*. Nascimento, Oliveira, Lavôr, Pereira, and Amorim (2020) state that the reduction of *E. coli* in water treated with this protein, is related to the removal of turbidity promoted by coagulation.

The neutral detergent fiber corresponds to cellulose, hemicellulose and lignin, being the best indicator to know the fiber content, having great importance in animal feed, for allowing to estimate the quality of a silage (Rodríguez, González, Domínguez, & Sarduy,

2016). Grain processing can alter the chemical composition of the oil, causing the degradation of fatty acids presented. Moringa oil had as predominant fatty acid oleic acid (73.60% - 76.61%), followed by erucic acids (5.65% - 6.67%), palmitoleic (4.90 - 5.72%) and elaidic (4.29 - 5.32%), values close to those found by Melo (2014).

5. Final Considerations

The drying air temperatures of 40 °C, 55 °C, and 70 °C significantly affected the physicochemical quality of the oil and the moringa cake. The best result is samples from grains dried at 40 °C. The composition of the main fatty acids was not altered according to the statistical method applied, these being oleic fatty acid (73.60 to 77.07%), erucic (5.65 to 6.67%) and palmitoleic (4.90 to 5.72%).

Chemical oil extraction, although more efficient than mechanic, showed higher levels of acidity and peroxide. The fiber content in neutral detergent and crude protein of the pie, decreased significantly for dry grains with drying air temperature above 40 °C.

For future researches it could be explored how other products react to the variation of drying temperatures.

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Percentage of contribution of each author in the manuscript

Bárbara Lemes Outeiro Araújo – 35%

Ednilton Tavares de Andrade – 20%

Jaqueline Damiany Portela – 15%

Rafael Peron Castro – 15%

Pedro Castro Neto – 15%