

Use of mufla oven calcination method in oil quantification of chia seeds

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Abstract: Chia (*Salvia hispanica* L.) is an annual herbaceous plant considered as natural omega-3 fatty acids source. To quantify this oil in seeds, a time-consuming method involving financial costs with chemical reagents is used. The objective of this work was verifying existence of correlation between the quantification of oil in chia seeds by means of the Soxhlet method with the ash mass of these seeds obtained by calcination in a muffle oven. For this, samples of chia seeds and the oil quantified by Soxhlet were collected. The same samples were calcined in ash. The simple correlation test was performed, obtaining linear regression. To method validation, a completely randomized design experiment was carried out. The treatments were to quantify the oil by the Soxhlet method and calcination in the muffle using the equation obtained. The objective was to verify the existence of correlation between the oil content of chia seeds, obtained by the Soxhlet method with ash content obtained after burning in the muffle and, therefore, the validation of this method of determination. There is a significant positive correlation between the oil content obtained by Soxhlet method and the ash mass of chia seed. By equation $Y = -2.5319x + 36.518$, it was noted that it is feasible to quantify the oil content.

Key words: *Salvia hispanica*; oil meaning; soxhlet.

Utilização do método de calcinação em forno mufla na quantificação de óleo de sementes de chia

Resumo: A chia (*Salvia hispanica* L.) é uma planta herbácea anual considerada como fonte natural de ácidos graxos como o ômega-3. Para quantificar o teor de óleo em sementes, é utilizado um método demorado que envolve custos financeiros com reagentes químicos. O objetivo deste trabalho foi verificar a existência de correlação entre a quantificação de óleo em sementes de chia por meio do método Soxhlet com a massa de cinza dessas sementes obtida por calcinação em forno mufla. Para isso, amostras de sementes de chia foram coletadas e o óleo quantificado por Soxhlet. As mesmas amostras foram calcinadas em cinzas. O teste de correlação simples foi realizado, obtendo-se regressão linear. Para validação do método, foi realizado um experimento de delineamento inteiramente casualizado. Os tratamentos foram quantificar o óleo pelo método Soxhlet e calcinação na

mufla usando a equação obtida. Objetivou-se verificar a existência de correlação entre o teor de óleo de sementes de chia, obtido pelo método Soxhlet, com o teor de cinzas obtido após a queima na mufla e, portanto, a validação deste método de determinação. Pode-se concluir que existe correlação positiva significativa entre o teor de óleo obtido pelo método de Soxhlet e a massa de cinza da semente de chia. Pela equação $Y = -2,5319x + 36,518$, notou-se que é possível quantificar o teor de óleo.

Palavras-chave: *Salvia hispanica*; teor de óleo; soxhlet.

Introduction

Chia is an annual herbaceous plant belonging to the family Lamiaceae, native to Mexico (Busilacchi et al., 2013). This plant species has a wide geographic distribution (Hernández-Gómez et al., 2008) being cultivated on a large scale in Paraguay (Irala, 2013) and, lately has been gaining world market (Migliavacca et al., 2014).

With oil-rich seeds, classified as simple lipids and highly energetic organic compounds (Pinheiro et al., 2005). The lipid quantification is done in most cases by extraction with chemical solvents such as hexane in a Soxhlet-type apparatus, followed by the removal by evaporation or distillation of the solvent used (IUPAC, 1998; IAL, 2008). Such a determination is time-consuming, costly and laborious, as well as generating chemical waste.

There are several laboratory methods that quantify substances, such as the determination of organic matter in the soil by the high temperature calcination method (Silva et al., 1999). Residue by incineration or ash is the name given to the residue obtained by heating a product at a temperature close to 550 °C in a muffle-type furnace. The residue represents the inorganic substances present in the sample (IAL, 2008).

Heating at high temperatures promotes the evaporation of organic substances as well as structural water (Silva et al., 1999), the oil being an organic compound that is lost by

volatilization. knowing the original content, it is possible to quantify what was lost to atmosphere.

Other methods of indirect determination are currently used, which do not involve reagent expenditures and are instantaneous. Therefore, the objective of the work was to verify the existence of a correlation between the oil content of chia seeds obtained by the Soxhlet method with the ash content obtained after burning in the muffle and, therefore, the validation of this determination method.

Material and Methods

The experiment was conducted in a laboratory of the Fazenda da Universidade Estadual de Maringá, Regional Campus of Umuarama, State of Paraná, Brazil.

Chia seeds were collected randomly in experiments from the university itself. For the laboratory determinations, the samples were submitted to drying in an air ventilation oven, at a temperature of 65 °C for 72 hours. The quantification of the oil was performed by the Soxhlet extraction method (IUPAC, 1998; IAL, 2008), using hexane as the solvent.

The results were expressed in percentage, obtained through the mass difference. Samples of 0.2 g of the same seeds were placed in crucibles whose masses were recorded and, after being cooled, the crucibles with the ashes were weighed and left in a muffle for five hours at a temperature of 550 °C, resulting in

the ash mass, by rule of three the result was expressed as a percentage of ash. It should be emphasized that the weighings were performed on an analytical precision scale, noting all the houses after the comma. The data were analyzed by means of simple linear correlation, using 1% of significance, aiming at obtaining linear regression.

To validate the method, 50 seed samples were collected, randomly in experiments collected on the same university campus. Soxhlet oil quantification and ash quantification were performed by the muffle method. The percentage of ash in oil was converted by means of the regression

obtained. For statistical analysis, a completely randomized design was used, with two treatments (Soxhlet and mufla), with 100 replications. A 1% significance analysis was performed for comparison between the two methods.

Results and Discussion

Figure 1 shows a significant positive correlation between Soxhlet oil quantification and the amount of ash. It is also noted that the correlation coefficient is high (0.89) and significant at 1% probability, thus indicating that it is possible to use the regression obtained to convert the amount of ash to oil content.

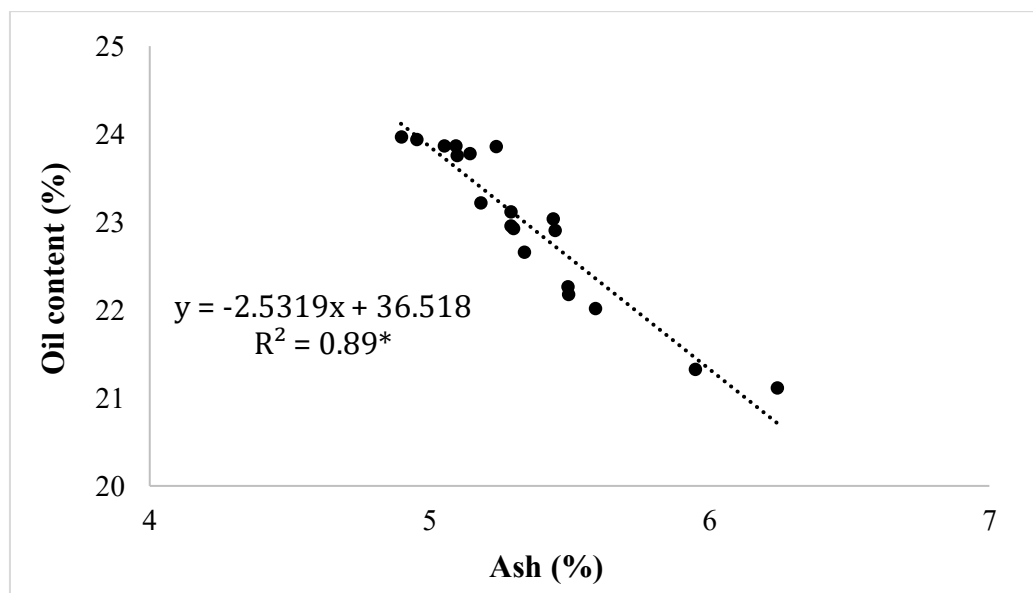


Figure 1. Correlation between oil content obtained by the Soxhlet method and percentage of ash obtained by the muffle method. ** = significant at 1% probability of error. Equation $Y = -2.5319x + 36.518$ consists of: $Y =$ oil content (%); $x =$ ash (%).

After obtaining the equation, 100 seed samples were used, being analyzed by the Soxhlet method and the regression obtained, verifying that there was no

significant difference between them (Table 1). Thus, the method is consistent with the actual oil content quantified by the Soxhlet method.

Table 1. Oil content obtained by the Soxhlet method and by calcination in a muffle oven.

Determination method	Oil content (%)
Soxhlet	22.99
Muffle	23.02
M.D.S.	0.21
V.C. %	3.45

Mean followed by the same letter, does not differ from one another by the test t, at 1% probability of error. M.D.S. = minimum difference significative.

V.C. = variation coeficient

In crambe, this methodology has already been used to quantify oil in the seeds (Silva et al., 2015), demonstrating the viability, besides being a non-polluting method, since it does not use chemical products. Other evaluations also use non-polluting methods, such as checking the chlorophyll content in plants, which is performed through indirect measurement through portable meters. Besides these there are also other reports of apparatuses, where there is no expense of reagents and destruction of the pigment. The analysis of chlorophyll using chlorophyll meter (SPAD-502), according to Silva-Lobo et al. (2012), is more accurate in determining chlorophyll content, in addition to reducing working time. In corn leaves, the chlorophyll concentration and the readings provided by the SPAD-502 were positively correlated (Argenta et al., 2001).

There is also the nuclear magnetic resonance (NMR) method which is also a non-destructive method that can quantify the oil content in seeds. Ungaro et al. (1992) compared the quantification of oil content in sunflower seeds by NMR and the soxhlet method, concluding that there is a high correlation between the two methodologies.

Other authors, such as Amarante et al. (2010), found positive results in the use of non-degradation methods, such as a colorimeter in determining the intensity of color and amount of chlorophyll in corn leaves. By means of this similarity of reading between the

devices, indirect measurement of the chlorophyll content can be performed in both devices.

Conclusion

There is a significant positive correlation between the oil content obtained by Soxhlet method and the ash mass of chia seed.

By equation $Y = -2.5319x + 36.518$, it was noted that it is feasible to quantify the oil content.

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References

- AMARANTE, C.V.T.; STEFFENS, C.A.; SANGOI, L.; ZANARDI, O.Z.; MIQUELOTO, A.; SCHWEITZER, C. Quantification of chlorophylls in maize leaves by non-destructive optical methods. **Revista Brasileira Milho e Sorgo**, v.9, n.2, 39-50, 2010.
<http://dx.doi.org/10.18512/1980-6477/rbms.v9n01p39-50>.
- ARGENTA, G.; SILVA, P.R.F.; BARTOLINI, C.G.; FORSTHOFER, E.L.; STRIEDER, M.L., 2001. Relationship of reading of portable chlorophyll meter with contents of extractable chlorophyll and leaf nitrogen in maize. **Revista Brasileira de Fisiologia Vegetal**, v.13, n.1, 158-167, 2001.

- BUSILACCHI, H.; QUIROGA, M.; BUENO, M.; SAPIO, O.; FLORES, V.; SEVERIN, C. Evaluación de *Salvia hispanica* L. cultivada en el sur de Santa Fe (República Argentina). **Cultivos Tropicales**, v.34, n.4, p.55-59, 2013.
- COELHO, M.S.; SALAS-MELLADO, M.M. Composição química, propriedades funcionais e aplicações tecnológicas da semente de chia (*Salvia hispanica* L) em alimentos. **Brazilian Journal of Food Technology**, Campinas, v.17, n.4, p.259-268, 2014.
- HERNÁNDEZ-GÓMEZ, J.A.; MIRANDA-COLÍN, S.; PEÑA-LOMELÍ, A. Cruzamento natural de chía. (*Salvia hispanica* L.). **Revista Chapingo Serie Horticultura**, v.14, n.3, p.331-337, 2008.
- IAL. Instituto Adolfo Lutz. **Métodos físico-químicos para análise de alimentos**. São Paulo: Ministério da Saúde, 4 Ed., 1 Ed. digital, 2008. 1020p. <http://www.ial.sp.gov.br/ial/publicacoes/livros/metodos-fisico-quimicos-para-analise-de-alimentos>.
- IRALA, R.L. **Cultivo da chia (*Salvia hispanica*)**. Assuncion: Agrofield, 2013. 16p.
- IUPAC. **Standard methods for the analysis of oils, fats and derivatives: Determination of oil content (Extraction method)**, 1998.
- MIGLIAVACCA, R.A.; SILVA, T.R.B.; VASCONCESOS, A.L.S.; MOURÃO FILHO, W.; BAPTISTELLA, J.L.C. O cultivo da chia no brasil: futuro e perspectivas. **Journal of Agronomic Sciences**, v.3, n. especial, p.161-179, 2014.
- PINHEIRO, D.M.; PORTO, K.R.A.; MENEZES, M.E.S. **A química dos alimentos**. Maceio: UFAL, 2005. 52p.
- SILVA-LOBO, V.L.; FILIPPI, M.C.C.; SILVA, G.B.; VENANCIO, W.L.; PRABHU, A.S. Relationship between chlorophyll content in leaves and panicle blast in upland rice. **Tropical Plant Pathology**, v.37, n.1, 83-87, 2012.
- SILVA, A.C.; VIDIGAL-TORRADO, P.; ABREU JÚNIOR, J.S. Métodos de quantificação da matéria orgânica do solo. **Revista Universidade Alfenas**, v.5, n.1, p.21-26, 1999.
- SILVA, T.R.B.; ROGÉRIO, F.; SANTOS, J.I.; POLETINE, J.P.; GONÇALVES JÚNIOR, A.C. Oil quantification of crambe seeds calcination method in muffle furnace. **Journal of Agronomic Sciences**, v.4, n.1, 106-111, 2015.
- UNGARO, M.R.G.; TOLEDO, N.M.P.; TEIXEIRA, J.P.F.; SUASSUNA FILHO, J. Analysis of oil content in sunflower seeds with nuclear magnetic resonance and sohlet methods. **Bragantia**, v.51, n.1, p.1-5, 1992.