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IDENTIFICATION AND OPTIMIZATION OF FIBER EXTRACTION METHODS FROM OLEAGINOUS PLANTS

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Abstract
Globally, oleaginous plants (OP) production has recorded significant growth, due to their large applications in biofuels industry or vegetable oil production for cosmetics, pharmaceuticals, dyes or industrial applications. The oleaginous plants chosen for this study are Carthamus tinctorius L., Silybum marianum L., Linum usitatissimum L. and Helianthus annus L., which are renowned for their high oil and fiber content. In order to optimize the methods for determining the total fiber content and the global digestion process of the non-cellulosic components of these oil plants, experimental design statistical techniques have been developed using statistical models such as multiple regression with variance analysis applied using the ANOVA test. The present study aims at a comparative statistical analysis on the identification and optimization of fiber extraction methods in the four OP chosen. Moreover, the study allowed the identification of the optimal values of the experimental parameters and the 'desirability' function to be used to evaluate the final responses. In conclusion, this study helps us to found a favorable way of obtaining fibers using modern extraction methods.

Keywords: extraction methods, oleaginous plants, optimization.

1. INTRODUCTION

Oleaginous Plants (OP) are a group of plants cultivated in Romania especially for oil production and industrial or nutritional consumption. *Carthamus tinctorius* L., *Silybum marianum* L., *Linum usitatissimum* L. and *Helianthus annus* L. were chosen in this study for their high fat and protein content.

It is well known the most important benefits, in all areas, of each of these OP. *Carthamus tinctorius* L. is known for its properties as analgesic, antiaging, antihypoxia, antifatigue, anti-inflammator, purgative, antinociceptive, immunosuppressive, antitumoral, antipyretic, antidote to poisoning, anticoagulantive, vasodilative, antihypertensive, anticonvulsant, even in post-partum hemorrhage, cerebrovascular, rheumatism, cardiovascular, gynecological, whooping cough, chronic bronchitis and sciatica disease (Asgarpanah et al., 2013; Zhou et al., 2014; Wang et al., 2014). *Silybum marianum* L. extracts have application in medicine as hepatoprotective, antioxidant,

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hypoglycemic, immunomodulator, anti-inflammator, antihepatotoxic, antiproliferative, liver and plasma lipidaemic controler, anti-arthritic, anti-HCV, anti-HBV, anti-fibrotic, anti-herpes, anti-carvinogenic, anti-tumoral, neuroprotective, cardioprotective etc. (Trappoliere et al., 2009; Bhattacharya, 2011; Pendry et al., 2017) and in industry as biodiesel (Fadhil et al., 2017). *Linum usitatissimum* L. is used as biodisel (Bacenetti et al., 2017) in enviromental areas; as antifungal, antioxidant, cancer chemoprotective; in various disease treatment (e.g. cancer, cardiovascular diseases) in medicine and as nutraceutical in food industry (Ford et al., 2001; Wang et al., 2017). *Helianthus annus* L. is famous for its medicinal properties as analgesic, anti-inflamator, (Emamuzo et al., 2010) and for its contributions to the renewable oil and fuel industry (Gerçel, 2002).
The aim of this study is to develop optimization methods in order to determine the total fiber content and to optimize the global process of digestion of non-cellulosic components, using experimental statistical design techniques. The interpretation of the experimental plans aims establishing the relationship between the studied factors and the dependent responses.

2. MATERIALS AND METHODS

Principal raw materials used for this study are shreds of *Carthamus tinctorius* L., *Silybum marianum* L., *Linum usitatissimum* L. and *Helianthus annus* L.
In order to determinate and optimize the total fiber content and the global process of digestion of non-cellulosic components, for all the oleaginous plants reminded before, were used a lot of practical and theoretical methods and procedures. Therefore, in determinating and optimizing of the total fiber content step, experimental design techniques were used. Was investigated the influence of two independent and continuous variables as *KOH concentration* and the *boiling time* on the global digestion process of the non-cellulosic components in the system, selecting as the dependent variable the total fiber content left behind acid-base digestion.
Interpretation of experimental plans aims at establishing the relationship that results between studied factors and responses, and the relationship between the factors is expressed by a polynomial equation as:

$$Y = b_0 + \sum_{i=0}^{n} b_i X_i + \sum_{i<j}^{n} b_{ij} X_i X_j + \varepsilon$$

where: Y represents the measured response, b_0 represents the mean value of the Y response, b_i represents the main effects of the independent variables X_i, b_{ij} are the terms that give the measure of the interaction between the variables and ε is the random error.
The chosen method helps us to elucidate the effects of several factors and possible interactions at the same time and to assess their relative importance for the intended responses.
The validation of the model was accomplished by evaluating the multiple regression coefficient, by analysis of variance (ANOVA) and tracing the response surfaces.

3. RESULTS AND DISCUSSIONS

In the all four oleaginous plants situations, 10 methods of digestion of non-cellulosic components in the system were tested, using various levels of the independent variables as: KOH concentration from 0.5% to 3.12% and boiling time from 10 minutes until 30 minutes.
In the *Carthamus tinctorius*, the adjusted coefficient of determination value (Adjusted R-squared) is presented in Table 1. R-squared (R^2) represents the common variance of all system variables, and the adjustment of its values according to the degree of freedom of the experimental plane is noted with Adjusted R-squared. Applying the ANOVA test, in this model, the significance threshold value is p=0.0001 and this value indicates us that the statistical model chosen is statistically

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signifiant and both investigated factors have a significant influence on the system response, regarding *Carthamus tinctorius* L. oleaginous plant.

In *Figure 1* is representated the combined effects and interactions between the selected independent variables (KOH concentration and boiling time) on the digestion process of the non-cellulosic components of *Carthamus tinctorius*.

Tabel 1. Adjusted coefficient of determination (Adjusted R-squared)

Nr. Crt.	Oleaginous plant	Adjusted R-squared values
1.	*Carthamus tinctorius* L.	0.9594
2.	*Silybum marianum* L.	0.9203
3.	*Linum usitatissimum* L.	0.9540
4.	*Helianthus anuus* L.	0.9874

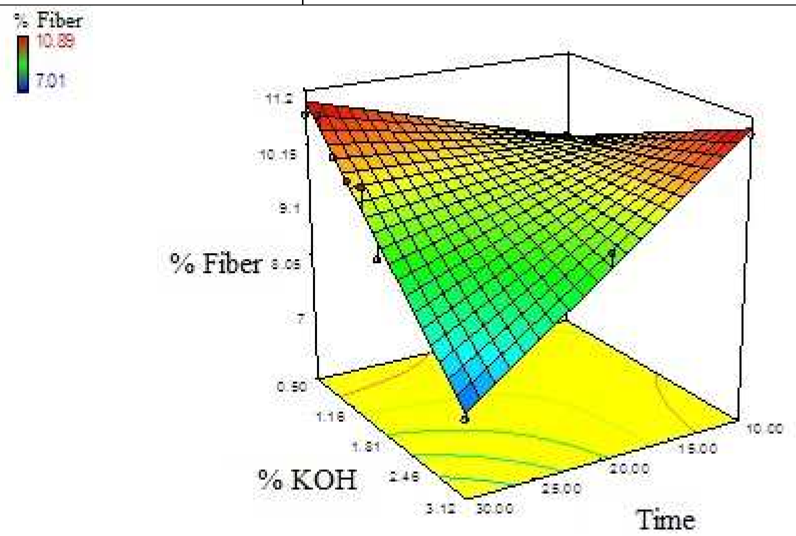

Figure 1. Response surfaces for fiber content of Carthamus tinctorius according to the digestion process variables in 3D representation

More, to optimize the global digestion process of *Carthamus Tinctorius* non-cellulosic components and to optimize the total fiber content determination method, the condition of maximizing the response on fitted model, respectively of the fiber content (%) was imposed. It has been found that there are several values of the experimental parameters that ensure integrity of fiber in the digestion process. Thus, similar results were obtained both by maintaining a low KOH concentration in the digestion system, about 0.51% for a longer time 29.15 minutes, but also using a more concentrated KOH solution about 3.07% for a shorter time 10.09 minutes. In the first case, the total fiber content percentage was 10.957% and 10.9228% for the second one.

In the *Silybum Marianum* plant model, the significance threshold is p=0.0011 from ANOVA test, and signifies that there is only a probability of 0.11% that the system variance is due to random variations of the independent variables, therefore the model is statisticaly significant. The adjusted coefficient of determination value corresponding to this model is presented in Table 1.

The representation of the combined effects and interactions between the selected independent variables (KOH concentration and boiling time) on the digestion process of the non-cellulosic components of *Silybum Marianum* plant is showed in Figure 2.

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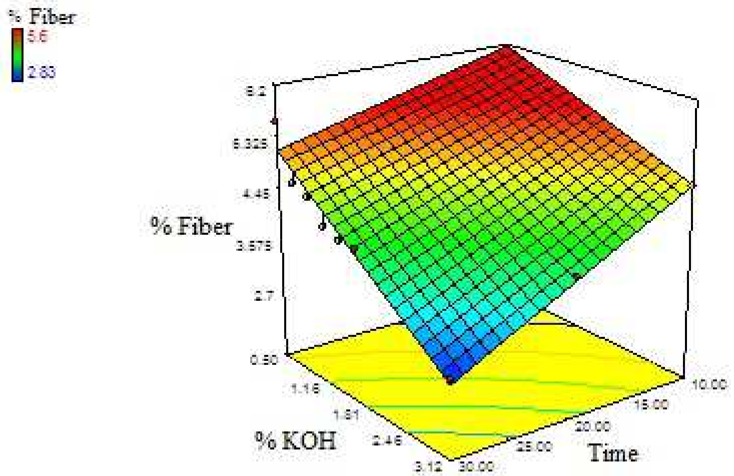

Figure 2. Response surfaces for fiber content of Silybum Marianum according to the digestion process variables in 3D representation

Regarding the purpose of this work, the study of a single response by the polynomial model analysis indicates response areas where the process is likely to yield optimal results. Therefore, the condition of maximizing the response, fiber content percentage, was imposed, taking into account that the digestion process in too soft conditions (KOH = 0.5%, Time= 15 minutes) resulted in incomplete digestion. And, the optimal experimental parameters identified in *Silybum marianum* sample are 0.62% - KOH concentration and for time variable - 10.71 minutes, for a 6.06% total fiber content in this optimised conditions.

The third chosen shred of plant, *Linum usitatissimum* L., was investigated and was found in this model p=0.0088 value by ANOVA, that ensure us about the statistically signifiacant of it. The value for the adjusted coefficient of determination (Adjusted R-squared) is in Table 1. In Figure 3, are presented the response surfaces for total fiber content, with emphasis of interactions between KOH concentration and boiling time in the digestion process.

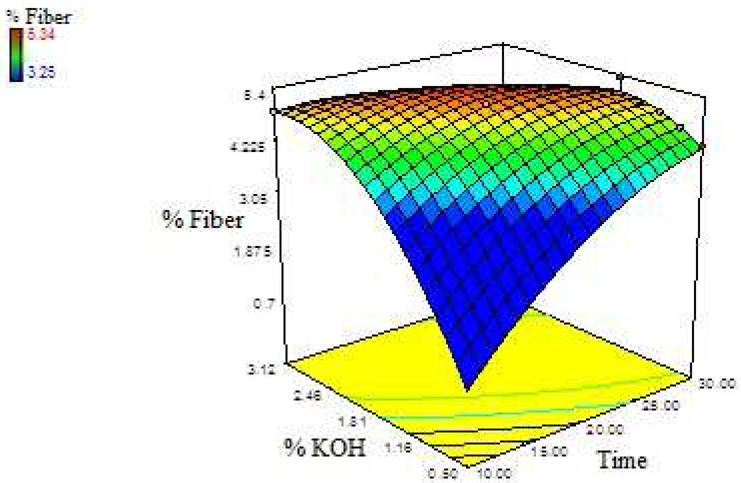

Figure 3. Response surfaces for fiber content of Linum usitatissimum according to the digestion process variables in 3D representation

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For the maximisation of the total fiber contents, the optimal experimental parameters have been set: KOH concentration is 2.14% and boiling time is 21.24 minutes, with 5.24568% total fiber content obtained.

The fourth chosen oleaginous plant, *Helianthus anuus* L., was found as beeing with higher total fiber content after digestion than the others plants, therefore, the selected model highlights the statistical significance demonstrated by analysis of variance where $p<0.0001$ was obtained, indicating that there is only a probability of less than 0.01% that the variance of the system is due to the random variations of the independent variables. In the Table 1 is suggested the adjusted coefficient of determination value for this plant also. The combined effects and interactions between KOH concentration and boiling time, on the digestion process of the non-cellulosic components of *Helianthus anuus* shred is evidentiated in Figure 4.

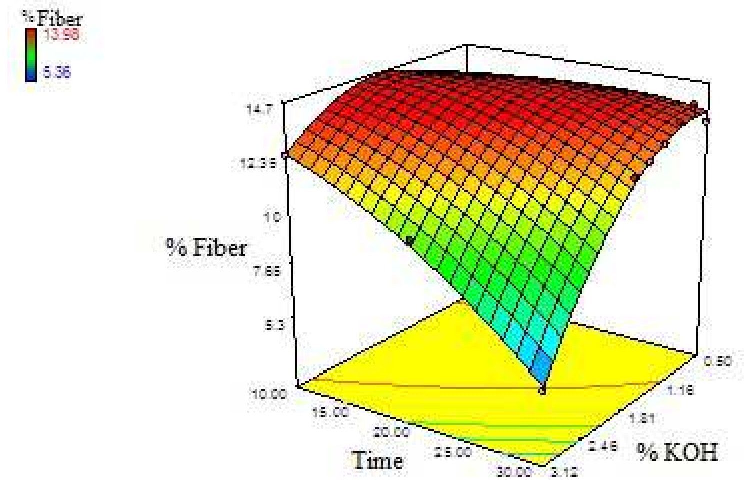

Figure 4. Response surfaces for fiber content of Helianthus anuus according to the digestion process variables in 3D representation

Following identification of the optimal experimental conditions for determining the fiber content of *Helianthus Anuus* was obtained the optimal conditions for this model as: KOH concentration equal to 1.73% and boiling time equal to 12.06 minutes, for an optimal 14.64% total fiber content.

The response, estimated in each situation, regarding the total fiber content obtained by identifying and optimizing the extraction methods, proved to be favorable, indicating that each chosen model was suitable for the type of plant studied.

The results outlined above were obtained using the FIWE Extraction unit for determining raw fiber content Velp Scientifica.

4. CONCLUSIONS

The ANOVA test indicated us that the variation of the results is due to the change of the independent variables and the selected models were considered significants.

The total fiber content of *Helianthus anuus* was highest relative to the other three plants studied. Both investigated factors (KOH concentration and boiling time) have a significant influence on the system response, and optimization methods indicates a good correlation between hypothesis and results.

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