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Chemometric approach for assessing the quality of olive cake pellets

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ABSTRACT

This article investigates the influence of processing parameters (conditioning and binder content), on quality of fuel agro-pellets produced from four olive cultivars (Istarska Bjelica, Buža, Pendolino, Leccino) grown in experimental filed in Croatia (Istria). Physical and chemical properties of pellets have been determined to assess their quality. Low ash and sulphur levels were detected, with elevated nitrogen levels for all samples. Analysis of variance and post-hoc Tukey's HSD test at 95% confidence limit have been utilised to show significant differences between various samples. Low coefficients of variation have been obtained for each applied assay (0.09–2.98%), which confirmed the high accuracy of the measurements. Score analysis and principal component analysis have been used for assessing the effect of process variables and variety of cultivars on final quality of pellets. For PCA modelling, experimental data for physical and chemical properties have been used. Standard score analysis revealed that equally good physical and chemical characteristics of pellets can be obtained with conditioning at 50 °C, but also without conditioning. The use of binders didn't affect the quality of pellets as much.

1. Introduction

Olive oil production, as one of the most important branches of Croatian agriculture and other Mediterranean countries, results in vast amounts of organic residues, with fresh olive cake as the main by-product. Estimated quantity of residues generated in the production of olive oil in the EU is around 6.8 million tonnes per year [1], while Croatia produces slightly less than 40 000 tonnes of olives per year [2], out of which approximately 16 000 tonnes of fresh cake is produced. Olive cake consists of solids and a part of vegetable water, and its consistency is similar to a paste. Such material is difficult to manipulate and it dries slowly, therefore many problems occur by its disposal [3].

Due to high amount of waste that olive oil production generates, it would be useful to find an environmentally friendly way of its utilisation. One of the methods of olive cake utilisation is production of thermal energy by combustion. The main technical and technological problem of combustion of olive cake is the low energy value per unit mass. Additionally, olive cake requires large storage spaces. The simplest way to enable more efficient usage of this residue as a source of energy is to process it by pelletization process.

Various factors can affect the quality of the pelleted product, such as moisture content, particle size and shape, chemical composition of raw material, and type of processing equipment. Agricultural residues, due to their low bulk density and specific chemical composition are hard to compress, while wood residues, such as sawdust and wood chips, have a structure with good compression properties and contain natural binders, such as lignin. Thus, the addition of material containing these natural binders or addition of commercial binders can improve the quality of pellets [4].

Conditioning of the material before pelletization by addition of steam or water may also improve pellet quality as this pre-treatment affects moisture content of the material. Conditioning is usually done by adding saturated steam at a given pressure, which increases the temperature and moisture of the material. Higher moisture content of the material reduces the energy consumption during pelletization [5], as well as lowers the friction in pellet press which affects pellet quality and the pelletization process itself [6,7]. The application of steam to prepare material for pelletization is used to obtain higher quality pellets [8,9]. Addition of heat and moisture affects the components in the material, such as starch and protein, activating their binding properties [10]. However, the application of too much heat or water can reduce the production capacity and quality of pellets [7].

Agro-pellets from olive cake are a product of uniform shape and size, which is much easier to manipulate than the starting material. Namely, pelletization facilitates transport (easier material handling, reducing transport costs), storage (reducing storage space), feeding

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in to combustion furnace and burnout. Also, it is important that pelletization increases the heating value per unit mass of cake [10,11].

Olive cake was previously investigated as a potential bio-fuel, where its chemical composition and characteristics for energy utilisation have been analysed, as well as the combustion of this residue. It has been proven that the use of olive cake as fuel significantly reduces the emissions of sulphur oxide, which is very high when burning fuel oil [12] and that it is a good energy source for the production of “clean” energy, considering that all of flue gas emissions were lower than recommended by EU directives on emissions of pollutants from large burning facilities [13].

It is shown that different fractions of waste from the production of olive oil have different combustion characteristics. Olive cake together with olive kernel emerged as the best fuel, with the smallest proportion of unburnt residue and maximum combustion efficiency [14].

Olive cake can be pelletized in combination with other materials. Thermal decomposition of pellets made of olive cake in combination with wood biomass depends primarily on the chemical composition of the samples, i.e. on ratio olive cake/wood biomass. With increase of share of olive cake in pellets, emissions of nitric oxide (NO) and sulphur dioxide (SO_2) also increase, which is understandable because the cake has more sulphur and nitrogen, than wood biomass [15].

In comparison with wood biomass pellets made from olive cake have higher calorific value. Olive cake pellets have low sulphur content, but higher ash and nitrogen content. Pellets made from olive cake can be considered to have acceptable properties for thermal utilisation. However there are restrictions for content of ash and nitrogen, which can lead to problems during combustion in the furnace. Physical properties of olive cake pellets can be improved by addition of wood biomass [16].

The objective of this study was to investigate the quality of pellets made from different cultivars of olive cake with and without addition of binder and with or without conditioning of the starting material. To assess the quality of pellets their physical and chemical characteristics have been determined. Experimental results have been subjected to analysis of variance (ANOVA) to show relations between applied assays (physical and chemical). In order to enable more comprehensive comparison between investigated samples, standard score (SS) has been introduced. Principal component analysis (PCA) has been applied to classify and discriminate analysed samples.

2. Materials and methods

2.1. Material

Four olive cake cultivars have been used in the research: Buža, Leccino, Pendolino and Istarska Bjelica grown in an ameliorated experimental field near Novigrad, Istria, Croatia. Olive cake was obtained after oil extraction from the fruit intended for the production of extra virgin olive oil from an olive oil mill in Istria in Novigrad, Croatia. The olive cake was a product after three way extraction method. The chemical composition of obtained cake was analysed, and the results were published in a previous investigation [17] and are presented in Table 1.

2.2. Preparation of the material

Olive cake obtained from the mill had an average moisture content of 50%, so it was dried at 40 °C in a biomass dryer until it reached 12%, which is optimal for storage and pelletization.

The cake was then milled using a laboratory hammer mill (“ABC Engineering”, Pancevo, Serbia) where a sieve with a mesh size of 4 mm was used. Hammer mill was used to increase specific surface area of the material particles thus making the pelletization easier, because larger surface area of particles per unit volume is available for absorption of water and condensed steam during the process of conditioning. Grinding also improves the quality of the pellets due better packing of particles and reduction of gaps between them, so there are fewer weak points in pellets.

Table 1
Chemical composition of olive cake.

%, dry basis	I. Bjelica	Buža	Leccino	Pendolino
pH	4.47 ± 0.02^d	7.07 ± 0.06^a	6.24 ± 0.23^b	5.13 ± 0.02^c
Protein	6.17 ± 0.10^b	4.39 ± 0.10^c	6.68 ± 0.10^a	6.61 ± 0.19^a
Raw cellulose	40.09 ± 0.08^a	40.34 ± 0.03^a	40.72 ± 0.04^a	38.91 ± 0.04^a
Oil	6.28 ± 0.02^b	9.74 ± 0.04^a	4.58 ± 0.07^c	4.50 ± 0.02^c
Ash	1.84 ± 0.01^b	2.07 ± 0.07^a	1.56 ± 0.02^c	1.60 ± 0.03^c
C	55.54 ± 0.72^a	54.21 ± 0.63^b	56.64 ± 0.56^a	55.19 ± 0.77^a
H	7.38 ± 0.34^b	7.52 ± 0.24^b	8.10 ± 0.08^a	7.79 ± 0.27^b
N	0.99 ± 0.02^b	0.70 ± 0.02^c	1.07 ± 0.02^a	1.06 ± 0.03^a
S	0.069 ± 0.01^b	0.068 ± 0.01^b	0.083 ± 0.01^a	0.084 ± 0.01^a

The results are presented as mean ± SD; different letters within the same row indicate significant differences ($p < 0.05$), according to Tukey's test.

Olive cake can be pelletized with the addition of binders (as prescribed by DIN standard 51 731 to a maximum of 2%). In order to determine the least amount of binder without negative influence on pellet quality, material was pelletized without binder and with addition of binder in an amount of 1 and 2%. Lignocelluloses material was used as a binder in order to avoid introduction of any inorganic components to biomass pellets.

Material was used as unconditioned or it was conditioned by addition of steam. Steam conditioning was carried out in two-shaft paddle conditioner/mixer (SLHSJ0.2, Muyang Group, China) where steam was added directly into the material at a pressure of 2 bars, to achieve the desired temperature of 50 and 80 °C, respectively. Material was held in the conditioner/mixer until it reached the desired temperature, and afterwards was released into the heat insulated receiving hopper below the mixer. Time necessary to achieve desired temperatures was approximately 45 and 60 s, respectively.

The combination of the parameters: conditioning and addition of binder resulted in nine combinations of materials for each olive cake cultivar, which makes a total of 36 combinations for pelletization (shown in Table 2).

2.3. Pelletization of the olive cake

Prepared olive cake was pelletized in vertical pellet press (Pellet Press 14-175; Amandus Kahl) using die with a diameter of die openings of 6 mm and a thickness of 24 mm. Temperature of the die during pelletization ranged from 70 to 75 °C, and the throughput was 20 kg/h.

The pellets were cooled in vibrating drier/cooler (model FB 500 × 200, “Amandus Kahl”, Germany), for 10 min at air temperature of 20 °C and the material flow of 18 kg/h.

2.4. Chemical analyses

Proximate analyses on the samples were done: moisture content [18], ash content [19], volatile mater [20] and fixed carbon (calculated by the difference between 100 and the sum of volatile matter, ash and moisture). Ultimate analyses done on the samples were: content of C,

Table 2
Combination of material.

	Conditioning	Binder
1	No conditioning	No binder
2		1% binder
3		2%binder
4	Conditioned at 50 °C	No binder
5		1% binder
6		2%binder
7	Conditioned at 80 °C	No binder
8		1% binder
9		2%binder