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Original research paper

THE INFLUENCE OF OIL CAKE GRANULATION AND ULTRASONIC PRETREATMENT ON THE PROPERTIES OF BIOPOLYMER FILMS BASED ON *CAMELINA SATIVA* OILSEED CAKE

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Abstract: This study aimed at investigating the synthesis of biopolymer films based on the *Camelina sativa* cake, an agricultural waste which remains after cold pressing of the oil from seed. During the film synthesis, three different granulations of the camelina cake were used: the whole cake, fractions with a size of 180-250 µm and fractions smaller than 180 µm. Half of the samples were pre-treated with an ultrasonic bath in order to examine the influence of the native mucilage from the cake on the properties of the obtained films. The biopolymer film samples were tested for mechanical, barrier, physicochemical and structural properties. The obtained films were dark, firm and flexible. Application of mucilage removal pre-treatment contributed to lower tensile strength and higher elongation at break. Significantly lower water vapour permeability was recorded in the samples not pre-treated regarding mucilage removal. A foil with optimal physicochemical characteristics was produced using oilcake with a particle size less than 180 µm, regardless of the pre-treatment application. There were no structural differences or differences in thermal behaviour among the tested samples. Statistical analysis (Z-Score analysis) showed the sample $CSoC_{<180,wo}$ was optimal due to good mechanical, barrier and physicochemical properties.

Key words: *agro-industrial waste, particle size, mucilage, barrier properties, mechanical properties, Z-score*

INTRODUCTION

Fossil raw materials are not an infinite resource and it is crucial to find not only new energy sources but also innovative materials alternatives to petroleum-based plastics (Letcher, 2020). In addition, the resistance and durability of traditional plastics led to huge waste disposal problems and consequent environmental pollution (Porta, 2019). The growing environmental concerns surrounding plastics have prompted research into alternative food packaging materials. Biodegradable materials, such as biopolymers, bio-

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plastics, bio-nanocomposites, and edible coatings, have been developed in an attempt to replace plastics (Perera, Jaiswal & Jaiswal, 2023; Šuput, Lazić, Popović & Hromiš, 2015). The idea of biodegradable polymers from renewable sources - biopolymers, comes from the need to close the natural cycle of matter, with the completion of one cycle marking the beginning of the next cycle. Biopolymers extracted from biomass are made from plant and animal materials, and they are, due to the large availability of raw materials for their production, the most common on the market (Popović, Lazić, Hromiš, Šuput & Hromiš, 2018). In this scenario, biopolymers from agricultural sources are an interesting option for plastics production since the agricultural industry generates a high quantity of different by-products containing biomacromolecules (such as proteins and polysaccharides), potentially able to mimic the oil-derived polymeric matrices (Mirpoor, Giosafatto & Porta, 2021). Functional package materials from underutilized crop-based renewable feedstock can replace conventional plastics (Sydor, Kurasiak-Popowska, Stuper-Szablewska & Rogozinski, 2022). Strategies for sourcing biopolymers include utilizing agricultural waste, instituting efficient culti-vation practices, and researching innovative biopolymer production technologies (Perera et al., 2023). Nevertheless, despite the huge worldwide production of agricultural biomass, only a small fraction of it is today utilized for applications different from animal feed (Mirpoor et al., 2021).

Oilseed crops have long been in the spotlight due to their multiple applications, such as producing functional vegetable oils, as well as animal feeds, pharmaceuticals, biofuels and other industrial usages, which led to an increase in oilseed crop cultivation areas. The global production of oilseeds, mainly intended for edible oil production, reached 659 million metric tons in 2023 (USDA, 2023) but the generation of by-products after the oil extraction, known as oilseed cake, accounts generally for about 50% of the original seed total weight. Therefore, among the biopolymers derived from renewable resources, SOC could be a potential raw material for bioplastic products since they are abundant, biodegradable and inexpensive. These plastic films could be successfully employed to manufacture one-time or short-term use items, mostly in the food packaging sector, replacing at least a portion of non-biodegradable materials currently used (Mirpoor et al., 2021).

Camelina sativa is an annual sustainable oilseed crop within the *Brassicaceae* family, with emerging ongoing interest. It is garnering renewed attention due to many positive attributes when compared with conventional oilseed crops since it possesses considerable agrotechnical and industrial benefits: the crop can be grown under different climatic conditions and with low input, as it is tolerant to drought, and low temperature and heat (Hunsaker, French, Clarke & El-Shikha, 2011; Matthäus & Zubr, 2000). Due to the lower demand for nutrients and water, it can be cultivated with no irrigation and a lower fertilization level, and often on marginal and saline soils (Mohammed, Chen & Afshar, 2017). Camelina is more resistant to many pests and diseases than other *Brassicaceae* family plants and, therefore, its cultivation is more environmentally friendly due to the reduced use of herbicides and pesticides (Bacenetti, Restuccia, Schillaci & Failla, 2017; Juodka et al., 2022; Arshad et al., 2022; Neupane, Lohaus, Solomon & Cushman, 2022). Among oilseeds containing interesting fatty acid profiles, camelina is identified as one of the main candidates to be used in the future bioeconomy (Lopez et al., 2023).

The two main products derived from camelina seed – oil and oilseed cake – are now the raw materials in various applications. Additionally, straw is also a valuable part of the plant. Seeds and straw are currently used to produce animal fodders, functional food, diet supplements, bio-lubricants, chemical derivatives, cosmetic formulations, biofuel (biodiesel, jet fuel), and other products such as ethanol, biogas, energy pellets, or compost to fertilize the soil.

Camelina seeds are rich in oil (30% to 49%), protein (24% to 31%) (Berti, Gesch, Eynck, Anderson & Cermak, 2016; Hossain, Johnson, Wang, Blackshaw & Gan, 2019), *ω*-3 acids, *ω*-6 acids, tocopherols, phytosterols, and phenolic compounds (Berti et al., 2016; Kurasiak-Popowska & Stuper-Szablewska, 2020). Camelina seeds contain carbohydrates in the form of monosaccharides, disaccharides, oligo-saccharides, polysaccharides, and fibre. Camelina oil contains from 50.8 to 66.6% polyunsaturated fatty acids with 31–40% alpha-linolenic acid, which contributes to the interest in

its human nutrition and health benefits. The consumption of camelina oil provides health benefits, such as reductions in blood serum cholesterol levels and improvements in serum lipid profiles, as recently reviewed (Neupane et al., 2022). Recently, camelina seed oil has received the Generally Recognized as Safe (GRAS) status from the United States Food and Drug Administration (Ubeyitogullari & Ciftci, 2020).

Large quantities of by-products (cake, meal) are left after oil extraction. These products have good potential to be used as a cheap alternative protein feedstuff and a valuable source in animal nutrition (Ilić et al., 2022; Rakita et al., 2023; Riaz, Ahmed, Sizmaz & Ahsan, 2022; Juodka et al., 2022). Ibrahim and El Habbasha (2015) reported that camelina meal contains ≈5.5% of sucrose, ≈1% of starch, less than 1% of pectin, 6.7% of mucilage, and 7.4% of lignin. The content of crude fibre in camelina meal is ≈15% (d.b.), with the major part being cellulose (Mondor & Hernández-Álvarez, 2022). Camelina cake consists of approximately 10% fat, 45% crude protein, 13% fibre, 5% minerals, and a small number of other substances. The addition of camelina oilseed cake, in combination with other bioplastics (e.g. polylactide, chitosan, gelatine, starch, cellulose, poly (vinyl alcohol) and polyhydroxyalkanoates and their derivatives), can be the basis for obtaining material with beneficial properties used in food packaging (Sydor et al., 2022).

Besides being a potential source of omega-3-rich oil, camelina seed has an underutilized, high content of mucilage (~10%) (Sarv, Trass & Diosady, 2017). Mucilage is a soluble fibre found in the outermost layer (mucous epidermis) of the seed. Even though the composition of the mucilage depends on the source, mucilage is a complex compound containing carbohydrates, uronic acids, proteins, and other bioactive compounds. Camelina seed mucilage can be a new source of hydrocolloid that possesses thickening, gelling, emulsifying, film-making and stabilizing properties which therefore has the potential to be used in a variety of pharmaceutical and food applications (Soukoulis, Gaiani & Hoffmann, 2018; Ubeyitogullari and Ciftci, 2020; de Oliveira Filho et al., 2021). Camelina mucilage also demonstrated film-forming properties (Qi, Li, Sun, Shi & Wang, 2016). Once hydrated, mucilage distends, breaks the mucilaginous cells and gradually expands outside. It is generally constituted of an adherent part, strongly bound to the seed and a more loosely adsorbed one. According to the extraction processes, mucilage dry matter can finally represent 7–10% of the mass of the seed and is mainly composed of polysaccharides (~80%) but also proteins (10–15%) and minerals (~6%) (Sarv et al., 2017).

Ultrasounds have been successfully employed to extract polysaccharides from a variety of plants (Silva, Sinnecker, Cavalari, Sato & Perrechil, 2022). The creation of cavitation bubbles and their subsequent collapse generate high spots with very high temperatures and pressure, able to break the bonds between the seed coat and the mucilage. Hence, the amplitude of the ultrasounds, extraction time and temperature must be correctly chosen to avoid the extraction of undesired compounds and mechanical disruption of the seeds (Fabre, Lacroux, Gravé & Mouloungui, 2020).

To increase the variety of species available in Balkan agriculture, two camelina genotypes have recently been developed in Serbia using superior breeding stock (NS Zlatka and NS Slatka). These camelina genotypes are well-suited to the Balkan region's environmental circumstances and have good production qualities (Marjanović Jeromela et al., 2021; Čanak et al., 2022). The study of Ilić et al. (2022) evaluated the nutritional value of two Serbian genotypes of camelina seed. Because they included a significant number of proteins, amino acids, and tocopherols as well as a moderate amount of minerals, the examined Serbian camelina seed genotypes displayed an outstanding nutritional profile (Ilić et al., 2022).

This work aimed to develop biopolymer composite films based on the agro-industrial by-product camelina oilseed cake and to examine the effect of the cake granulation on the properties of the obtained films.

In addition, another no less important objective was to investigate the effect of pretreatment with an ultrasonic bath to remove mucilage and to examine the effect of the presence/absence of mucilage on the characteristics of biopolymeric films based on camelina cake.

MATERIALS AND METHODS

Materials

Seeds of *Camelina sativa* (CSoC) were kindly provided by the Institute of Field and Vegetable Crops (Novi Sad, Serbia). Camelina seeds were subjected to mechanical cold-pressing to obtain oil, while the remaining cold-pressed cake was used for further analysis. The basic chemical composition of oil cake was 16.2% oil, 35.1% protein and 9.3% cellulose.

The cake was ground, sieved to different granulation degrees and stored at 4 °C before use. All other reagents used in this study were of analytical grade.

Biopolymer film preparation

Biofilms based on *Camelina sativa* oilseed cake (CSoC) were prepared using the casting method. Filmogenic suspension was prepared by dispersing CSoC (3%, w/v) in distilled water with 40% glycerol (w/w, by weight of CSoC) which was used as a plasticizer.

One half of the samples was kept in an ultrasonic bath VTUSC6 (Velleman, Belgium) for 20 min, while the other half was untreated. pH of all samples was further adjusted to 12 by the addition of NaOH solution which was determined using a pH meter (Metrohm AG, Switzerland). After adjusting the pH value, the obtained suspension was incubated in a water bath at 90°C for 20 min. After thermal treatment, the suspension was filtered through a nylon filter to remove undissolved, coarse particles from the cake, and the filtrate was used for further use.

The obtained film-forming suspension was poured onto Petri plates (50 g per plate) covered with Teflon and dried at room conditions (23±2°C, 50±5%RH) for 5 days. After drying, obtained films were removed from Petri plates and analyzed.

Ground camelina cake (CC) was separated into two fractions of different particle size by use of a universal laboratory sifter (Bühler AG, Uzwil, Switzerland) equipped with a stack of sieves (fraction between 250μm and 180μm, and fraction <180μm). Along with the two fractions, the whole ground CSoC were used to prepare 6 groups of films samples labeled as follows:

1. $CSoC_{whole,wo}$ - CSoC based biopolymer films obtained from whole cake without ultrasound pretreatment
2. $CSoC_{whole,w}$ - CSoC based biopolymer films obtained from whole cake with ultrasound pretreatment
3. $CSoC_{180\text{-}250,wo}$ - CSoC based biopolymer films obtained from cake sifted to the 180-250 μm without ultrasound pretreatment
4. $CSoC_{180\text{-}250,w}$ - CSoC based biopolymer films obtained from cake sifted to the 180-250 μm with ultrasound pretreatment
5. $CSoC_{<180,wo}$ - CSoC based biopolymer films obtained from cake sifted to the less than 180 μm without ultrasound pretreatment
6. $CSoC_{<180,w}$ - CSoC based biopolymer films obtained from cake sifted to the less than 180 μm with ultrasound pretreatment

Biopolymer film characterization

Film thickness

Film thickness was surveyed with 1 μm sensitivity micrometer. Eight replicates were carried out on each sample.

Mechanical properties

Tensile strength (TS) and elongation at break (EB) were measured on an Instron Universal Testing Instrument Model No 4301 (Instron Engineering Corp., Canton, MA, USA), according to standard method EN ISO 527-3:2018. The film samples were cut into rectangular tubes with a length of 80 mm and a width of 15 mm. The initial distance between the instrument terminals was set to 50 mm, while the speed was set to 50 mm/min. Tensile strength and elongation at break are expressed as mean values of 8 repetitions ± SD.

Barrier properties - water vapor permeability

The water vapor permeability (WVP) of the obtained biopolymer films was determined by the gravimetric method (dish method) based on the standard method (ISO 2528, 2017). Anhydrous silica gel was used as a desiccant in the test dishes. A sample of the film is placed on the container, sealed hermetically and the sample prepared in this way is left in an air conditioning chamber (Binder, KMF 240 (E6), Neckarsulm, Germany) at testing conditions t = 23°C ± 1°C and RH = 50% ± 2%. The mass of silica gel was measured after every hour, for up to 5 hours. The rate of water vapor transfer

through the sample was calculated as the ratio of the difference in the mass of silica gel before and after treatment (absorbed moisture) and the surface of the film sample and expressed in g/m^{2}h. Three tests were performed for each sample, and the result was expressed as mean value ± SD.

Physicochemical properties

Moisture content

Film samples (2x2) cm were measured at analytical balance (m_1) and dried at (105 ± 2)°C in a drying oven (Instrumentaria, Zagreb, Croatia), until constant mass, after which their mass (m_2) was measured again. The moisture content was determined as a percentage of the loss of the initial weight of the film after drying. The result was expressed as the mean value of three independent measurements ± SD, for each sample, on the weight of the wet sample, according to the equation:

$$MC\ (\%) = (m_1-m_2)/m_1 * 100$$

Total soluble matter (film solubility)

Film samples (size 2×2 cm) were first dried in an oven (Instrumentaria, Sutjeska, Croatia) at 105 ± 2 °C to a constant mass and measured on an analytical balance in order to obtain the initial dry matter of the films (m_1). After the measurement, each film was left in a container with 50 mL of distilled water for 24 hours at room conditions, with occasional shaking. After 24 hours, the water was decanted and the films were dried again in an oven (Instrumentaria, Zagreb, Croatia) at 105 ± 2 °C to a constant mass. The mass measured on the analytical balance represents the dry matter of the film after dissolution (m_2). The total solubility of the films (TSM) (%) was calculated according to equation:

$$TSM\ (\%) = (m_1-m_2)/m_1 * 100$$

where m_1 represents the dry matter of the film before dissolving in water, and m_2 the dry matter of the film after dissolution in water. The test for determining the total solubility was performed in three independent replicates, and the results are given as mean ± SD.

Swelling degree

Film samples (size 1x2 cm) were measured on an analytical balance (m_1), and then immersed in a container with 25 mL of distilled water for 2 minutes with shaking. The water was decanted and the excess water from the sample was removed with filter paper, and the new mass of the samples (m_2) was measured. The amount of absorbed water was calculated according to the equation:

$$SD\ (\%) = (m_2-m_1)/m_1 * 100$$

where m_2 and m_1 represent the masses of the wet and dry samples, respectively. For each sample test was repeated three times, and the swelling degree was expressed as the mean value ± SD.

Structural properties

Structural properties were determined by using Fourier transform infrared spectroscopy. FTIR spectra of biopolymer films were recorded at room temperature on a Nicolet IS10 FT-IR spectrophotometer (Thermo Fisher Scientific, MA, USA) according to method ASTM D5576:00 (2013). All spectra were recorded in the spectral range 4000-400 cm^{-1}, at a resolution of 4 cm^{-1}. Each sample was scanned 16 times while a blind sample (background) was recorded before analyzing each sample. Software Omnic 8.1 (Thermo Fisher Scientific, MA, USA) was used to collect, manage and process the FTIR spectra.

Thermogravimetric analysis (TGA)

Thermogravimetric analysis (TGA) of biopolymer films was performed on a LECO 701 Thermogravimetric Analyzer (LECO Europe B.V., The Netherlands) from 25 to 600 °C under the flow of air at a heating rate of 10 °C/min.

Statistical analysis

Correlation analysis

The color-coded plot diagram was created using R software version 4.0.3 (64-bit version) to visualize the mean values of all observed responses. The "corrplot" function was used with the "circle" method and the upper type enabled to illustrate the correlations between the various responses observed in the samples.

Principle component analysis

Principal component analysis (PCA) was employed as a pattern recognition technique to characterize and distinguish between different analysed samples and their corresponding responses using assay descriptors.

Analysis of variance

Statistical differences were assessed using an analysis of variance (ANOVA), and mean

separations were conducted using the Tukey's Honestly Significant Difference (HSD) test. These statistical analyses, as well as PCA, were carried out using STATISTICA 12.0 software, specifically the 2013 version provided by StatSoft Europe (Hamburg, Germany).

Z-score analysis

Z-Score analysis involves applying min-max normalization to the observed responses. This normalization process transforms the original response values into a new dimensionless unit system, making these different responses comparable and suitable for further mathematical calculations. The maximum value of the normalized total Z-score represents the optimum value of all combined segment Z-scores, which include all the analyzed responses. This maximum total Z-score indicates the optimal overall quality of the observed samples. The calculation of individual segment Z-scores was performed as described by Filipović et al. 2022.

RESULTS AND DISCUSSION

Biopolymer film preparation and visual appearance

The resulting films had a lot of common visual traits: they were opaque, dark, yellow-brown in color, strong, and flexible (Fig. 1).

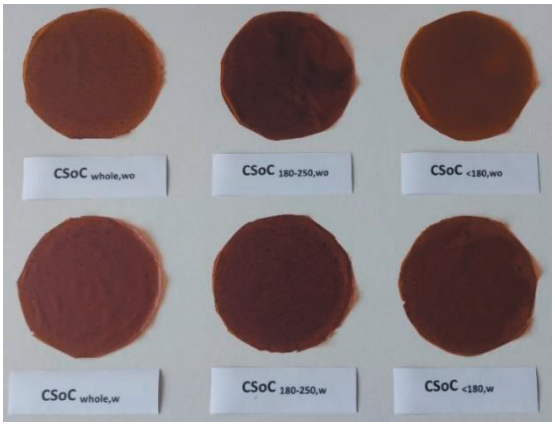


Figure 1. Visual appearance of the obtained CSoC based films

There was a distinctive odour originating from the raw material in each group of samples.

Table 1.
Film-forming suspension yield (%)

Suspension yield (%)					
Without pretreatment			With pretreatment		
$CSoC_{whole,wo}$	$CSoC_{180-250,wo}$	$CSoC_{<180,wo}$	$CSoC_{whole,w}$	$CSoC_{180-250,w}$	$CSoC_{<180,w}$
51.50	59.50	76.50	52.00	70.00	84.75

Table 2.
Characterization of the CSoC based films

Sample	Thickness (µm)	WVP (g/m^2h)	MC (%)	TSM (%)	SD (%)
CSoC $_{whole,wo}$	197.23 ± 5.92^{b}	10.07 ± 0.31^{d}	28.20 ± 0.60^{c}	50.92 ± 1.91^{b}	207.74 ± 13.42^{e}
CSoC $_{whole,w}$	177.42 ± 7.44^{a}	12.93 ± 0.45^{e}	30.61 ± 2.15^{d}	68.20 ± 2.39^{d}	166.54 ± 6.05^{b}
CSoC $_{180-250,wo}$	193.06 ± 7.71^{b}	7.90 ± 0.24^{a}	28.69 ± 0.82^{c}	50.82 ± 0.45^{b}	216.76 ± 29.77^{f}
CSoC $_{180-250,w}$	204.83 ± 9.71^{c}	13.23 ± 0.47^{f}	26.17 ± 3.56^{b}	65.07 ± 4.03^{c}	184.26 ± 5.11^{d}
CSoC $_{<180,wo}$	202.11 ± 3.72^{c}	8.64 ± 0.62^{b}	24.22 ± 0.93^{a}	45.90 ± 0.68^{a}	170.31 ± 11.42^{c}
CSoC $_{<180,w}$	210.83 ± 8.11^{d}	9.51 ± 0.08^{c}	26.01 ± 1.30^{b}	71.94 ± 2.01^{e}	126.72 ± 11.30^{a}

$^{a-f}$ Means in the same column with different superscript are statistically different ($p \leq 0.05$)
WVP (g/m^2h) - water vapor permeability, MC (%) – moisture content, TSM (%) – total soluble matter, SD (%) – swelling degree

Table 3.
Z-Score Analysis of properties of the obtained CSoC based films

	S1	S2	S3	S4	S5	S6	S7	Total Z Score
CSoC $_{whole,wo}$	0.407	0.613	0.409	0.593	0.378	0.807	0.100	**0.472**
CSoC $_{whole,w}$	1	0.183	0.741	0.056	0	0.144	0.558	**0.383**
CSoC $_{180-250,wo}$	0.532	1	0	1	0.301	0.811	0	**0.520**
CSoC $_{180-250,w}$	0.179	0.549	1	0	0.695	0.264	0.361	**0.435**
CSoC $_{<180,wo}$	0.261	0.903	0.136	0.861	1	1	0.516	**0.668**
CSoC $_{<180,w}$	0	0	0.602	0.698	0.719	0	1	**0.431**

During the synthesis procedure, a different yield of the film-forming suspension was noted about the initial amount of added components, cake granulation and ultrasonic pre-treatment applied. Table 1 shows the calculated yields (%).

Based on the obtained results, the influence of the application of pre-treatment can be highlighted because higher yields were obtained for all groups of samples according to the different degrees of granulation when the mucilage was removed.

Also, the cake with finer granulation (<180μm) had a higher yield in terms of the amount of film forming suspension. The most noticeable difference was with the $CSoC_{whole,w}$ (52%) and $CSoC_{<180,w}$ (84.75%) samples.

Biopolymer film characterization

Film thickness

Thickness of a biopolymer film is an important characteristic that directly affects the other properties, its potential application and product sustainability (Popović, 2013). The measured thicknesses of the obtained CSoC based films are presented in Table 2. Thickness values were in the range 177.42 μm to 210.83 μm.

Mechanical properties

The results of tensile strength and elongation at break determination are shown in Fig. 2. The maximum value of stress (tension) that the material can withstand is the tensile strength of the material. From Fig. 2a, it can be concluded that the application of the ultrasonic bath and mucilage removal, contributed to the reduced values of the tensile strength (TS), which in a practical sense means that the obtained films are less firm. The obtained TS values ranged from 1.02 MPa for sample $CSoC_{<180,w}$ to 1.95±0.43 for sample $CSoC_{180-250,wo}$. The obtained TS values are common for biopolymer films based on oilseed cakes. Literature data showed that the TS of biopolymer films based on pumpkin oil cake was 1.37 MPa (Bulut, 2021), the TS of biopolymer films based on sunflower oil cake was 4.37 MPa (Šuput et al., 2018a), and 1.4-3.65 MPa for films based on rape seed oil cake (Jang, Lim, & Song, 2011), etc.

Elongation at break (EB) represents the maximum change in sample length (elongation) before tearing. The obtained results indicate a positive impact of mucilage removal with an ultrasonic bath because within each group of samples, higher values of EB were observed in the samples where ultrasonic bath pretreatment was applied. The highest value (22.30±2.26%) of EB was achieved for sample $CSoC_{180-250,w}$. Films with higher values of EB are more elastic. The obtained values range (14.20 % for sample $CSoC_{180-250,wo}$ to 22.30±2.26% for sample $CSoC_{180-250,w}$) is in agreement with literature data: EB values for films based on pumpkin cake at pH 12 are about 5% (Popović, 2013), for films based on sunflower cake 22.03% (Šuput et al., 2018a) and films based on rapeseed oil cake 11.62-20.62% (Jang, Lim, & Song, 2011).

Barrier properties - water vapour permeability

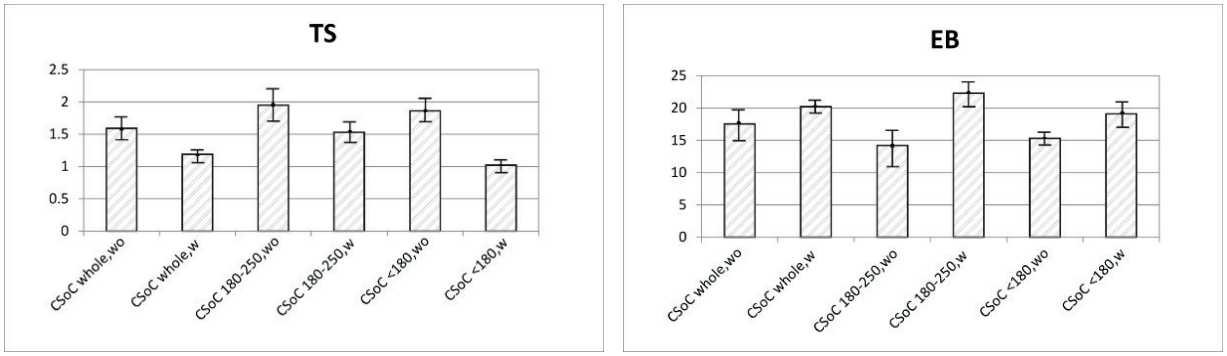
In case of packaging materials for food products sensitive to moisture content, high water vapour permeability (WVP) or water vapour transmission rate (WVTR) might be a significant disadvantage. Unlike synthetic materials, biopolymer packaging materials often have substantially greater WVP and WVTR due to polar groups in their molecular structures (Abdelhedi et al., 2018). Table 2 displays the WVP values for the obtained CSoC-based films. The obtained values were in the range of 7.90 g/m^2h – 13.23 g/m^2h and correspond to literature values of water vapour permeability (Vartiainen, Vähä-Nissi & Harlin, 2014).

Lower WVP values were obtained in all sample groups that were not pre-treated for mucilage removal, which was best observed with $CSoC_{180-250,wo}$ sample. The presence of mucilage contributed to lower values of water vapour permeability. Mucilage is commonly used as a thickener, stabilizer and emulsifier. Proteins and polysaccharides have a better emulsifying efficiency when combined than when used separately. Mucilage has the potential for emulsion stabilization due to the simultaneous presence of polysaccharides and proteins in its structure. Recent research has been done on application of mucilage to stabilize emulsions (Yang et al., 2023). This property combined with composite CSoC positively affected the water vapour permeability of the obtained films.

Physicochemical properties

The produced CSoC-based films' physicochemical characteristics in terms of moisture

content (MC), total soluble matter (TSM), and swelling degree (SD) are displayed in Table 2. In application of biopolymer-based films, one of the most important issues is the film's water and moisture sensitivity, which is related to qualities and WVP of these materials.



(a) (b)

Figure 2. Tensile strength (TS) (MPa) (a) and elongation at break (EB) (%) (b) of the obtained CSoC-based films

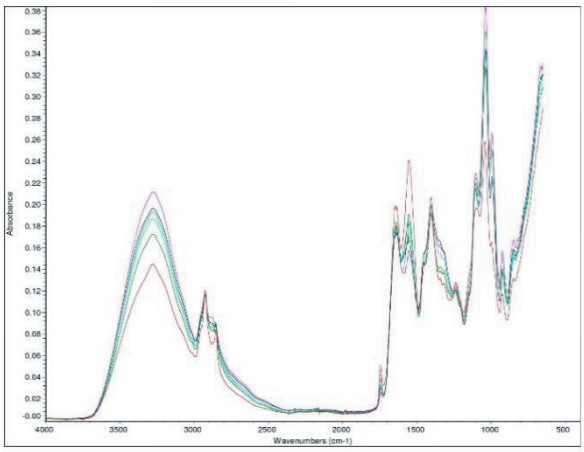


Figure 3. FTIR spectra of the obtained CSoC-based films

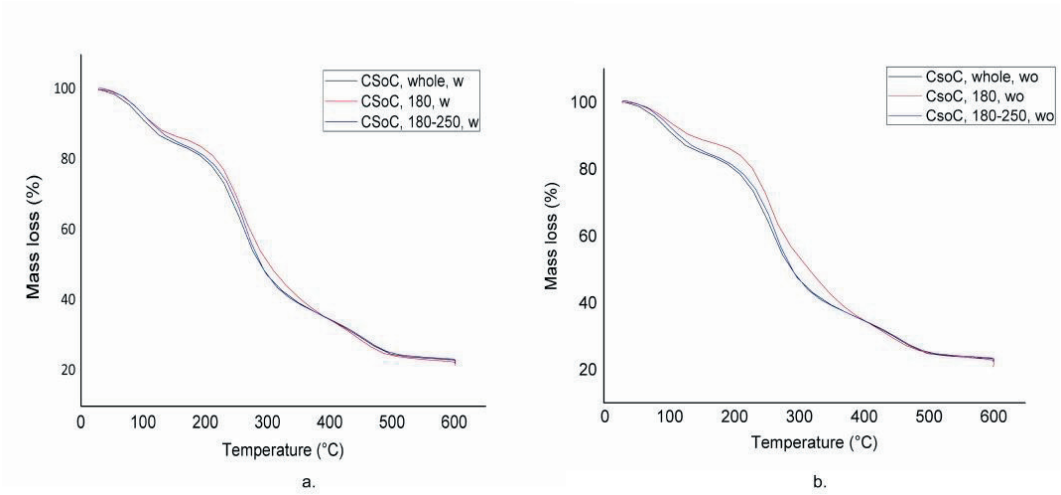


Figure 4. TGA curves of the obtained CSoC-based films

Regarding moisture content, it was uniform, in the range from 24.22% to 30.61%, but the samples prepared from whole cake retained a higher amount of moisture than those prepared with camelina cake of lower granulations. The lowest moisture content was in the $CSoC_{<180,wo}$ film.

Food protection, especially for items with high water activity, considerably depends on the TSM of packaging materials, which is related to water resistance (Hosseini, Rezaei, Zandi, M., & Farahmandghavi, 2015). TSM for the studied films was in the range of 50.82% – 71.94%. According to the available literature, CSoC-based films exhibited lower water solubility compared with gelatin films (98%) (Ahmad, Benjakul, Prodpran & Agustini, 2012), casein films (86%) (Aliheidari, Fazaeli, Ahmadi, Ghasemlou & Emam-Djomeh, 2013), sunflower proteins (93.2%) (Salgado, López-Caballero, Gómez-Guillén, Mauri & Montero, 2013), higher values compared with pumpkin oil cake based films (36%) (Bulut et al., 2020), while similar to those with hake proteins (66%) (Pires et al., 2013).

The studied CSoC-based biopolymer films showed lower TSM values in the non-pre-treated samples, indicating that mucilage significantly contributed to TSM reduction. The lowest TSM was achieved in the $CSoC_{<180,wo}$ film.

On the other hand, it seems that an application of the pre-treatment contributed to the lower SD values. In interaction with water, polar water molecules are drawn to the hydrophilic surface of the film and subsequently penetrate the matrix, adhering to its polar groups. This is the reason why swelling is an unfavourable material property, causing the polymer to swell and the hydroxyl group to dissolve locally, releasing additional groups that can act as binding sites. Sample $CSoC_{<180,w}$ had the lowest SD. SD ranged from 126.72% to 216.76%, which is in agreement with literature data for biopolymer films obtained from other types of cakes, such as sunflower (128.91%-198.73%) (Šuput et al., 2018b) and pumpkin oil cakes (148%) (Bulut et al., 2020). Based on the examination of all physicochemical parameters, the film with a granulation of less than 180 μm showed optimal performance, implying the significant influence of the cake granulation on the tested film properties.

Structural properties

To explore the possible granulation and mucilage effects on the biopolymer film structure, FTIR spectral analysis was performed. FTIR analysis found that the degree of granulation and the pre-treatment, had no effect on the structural properties of the studied films (Fig. 3). Peaks at 3280 cm^{-1}, 1640 cm^{-1}, 1403 cm^{-1}, 1038 cm^{-1} and 994 cm^{-1} could be found in all tested samples.

The broad peak at 3200–3500 cm^{-1} (centred at 3280 cm^{-1}) represents the O–H stretching vibration caused by intermolecular hydrogen bonding and correlates to the general structure of carbohydrates (Jiang and Zheng, 2023; Ağçeli, 2022). Like in the paper of Semwal, Ambatipudi and Navani (2022) free and bound O-H from mucilage overlaps with N-H groups from the protein. Mucilage contains hemi-cellulose, protein macromolecules, and sugars providing -NH_2, -COOH, and -OH functional groups. The 1640 cm^{-1} and 1400 cm^{-1} bands are typical for Amide I and Amide II. Amide I has a contribution from the stretching vibration of the C-N bond together with the C=O stretching vibration. For the FTIR study of secondary protein structures, the Amide I band is the most relevant peak. N-H bending vibration and C-N stretching vibration produce Amide II (Pradini, Juwono, Madurani & Kurniawan, 2018). The peak at 1042 cm^{-1} comes from the C-O stretching of primary alcohols, while bands at 994 cm^{-1} are the fingerprints of the main chemical bonds of the polysaccharides (Ağçeli, 2022).

Two samples stand out from the others: in the $CSoC_{180\text{-}250,wo}$ sample, mono-substituted acetylenes were identified by the strong -C:::C-H (carbon hydrogen) stretching absorption around 3300 cm^{-1} while in the $CSoC_{<180,w}$ sample, furan compounds were detected. Furan compounds show bands due to the =C-H stretching vibration just above 3000 cm^{-1}.

Thermogravimetric analysis (TGA)

Fig. 4 shows the TGA curves of the prepared films. The thermal decomposition of films involved three steps. The films prepared with different cake granulations demonstrated a similar degradation pattern, expected considering their similar compositions. In each series of samples made from cakes of different

granulation, those prepared with the whole cake ($CSoC_{whole,wo}$ and $CSoC_{whole,w}$ sample) possessed the lowest T5% values, and the maximum degradation rate temperature in the first stage (TdmaxI), probably due to the non-uniform distribution of whole cake in the matrix and lower populations of hydrogen/secondary bonds that easily disrupt. The first step, up to 170-178 °C, with a weight loss of up to 13-17%, corresponds to water evaporation and the decomposition of carotenoids present in the films.

The second and principle degradation step, up to 401-409 °C, with a weight loss of up to 66-68%, corresponds to the flavonoid degradation, cleavage of peptide bonds in proteins, further degradation of carotenoids, and in the series of films prepared without pre-treatment with ultrasound. The third stage, up to 600 °C, can be attributed to the complete degradation and carbonation until 0.02% of residual mass. When comparing the thermograms of films prepared in two ways (with and without pre-treatment with ultrasound), no significant difference in their thermal behaviour at elevated temperatures can be observed.

Relationship between the observed parameters

A colour correlation diagram visualizes the significance of the correlation coefficients between different responses (Fig. 5). Positive correlations were depicted in blue, while negative correlations in red. The size of the circles in the diagram indicated the strength of these correlations (Ćurčić et al., 2022). As presented in Fig. 5, several noteworthy correlations existed. There was a notably high positive correlation between EB and WVP (r^2=0.922), between EB and TSM (r^2=0.789) and between TS and SD (r^2=0.773). All observed correlations were statistically significant at a significance level of $p \leq 0.05$. Negative correlations also existed. Specifically, there was a negative correlation between TSM and TS (r^2=-0.906) and between thickness and DM (r^2=-0.847).

These findings provide valuable insights into the interrelationships among the observed responses, shedding light on the factors that influence the quality characteristics of the samples. The relationships between different samples were explored using PCA, and the results are illustrated in Fig. 6. In the PCA graphic, the proximity of spots indicates the similarity in patterns among the samples. The direction of the vectors in the factor space reveals the trends of the observed variables, while the length of the vectors indicates the strength of the correlation between the variable and the fitting value. By examining Fig. 6, one can efficiently discern the correlation between the content of various compounds and the obtained compound content. The angles between corresponding variables reflect the degree of correlation, with smaller angles indicating stronger correlations. The first two principal components (PCs) collectively accounted for 82.97% of the total variance in the dataset. Specifically, the first PC explained 52.13% of the variance, and the second PC explained 30.84% of the variance

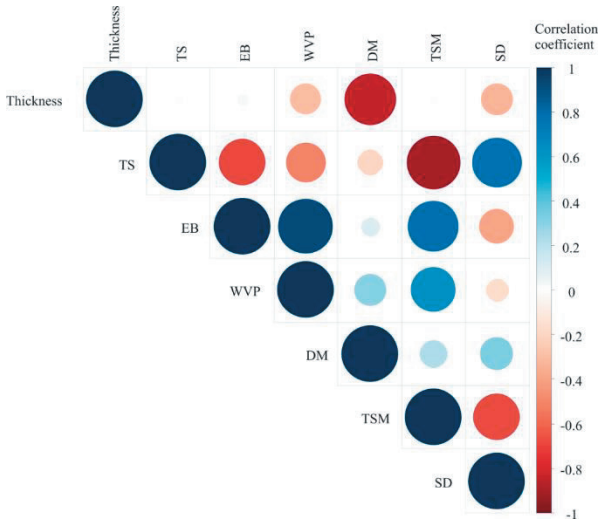


Figure 5. Colour correlation diagram between the observed responses

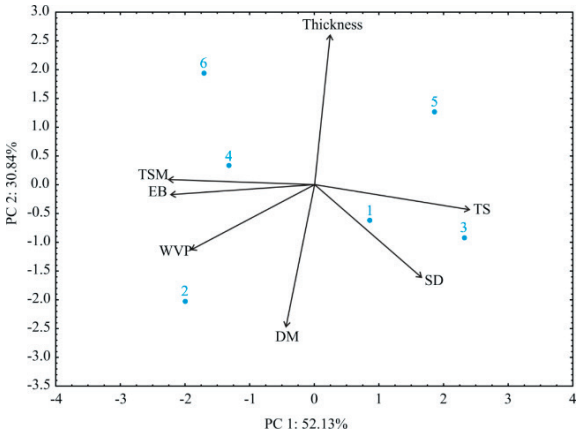


Figure 6. The PCA biplot diagram, depicting the relationships among thickness, TSM, EB, WVP, DM, SD and TS for samples $CSoC_{whole,wo}$ (1), $CSoC_{whole,w}$ (2), $CSoC_{180\text{-}250,wo}$ (3), $CSoC_{180\text{-}250,w}$ (4), $CSoC_{<180,wo}$ (5) and $CSoC_{<180,w}$ (6)

among the experimental data. The percentages (52.13% for the first principal component and 30.84% for the second principal component) suggest the proportion of total variance in the data explained by each principal component. This signifies that PC1 captures 52.13% of the total variance in the original data. The higher the percentage, the more information is retained in this single component. PC1 is the direction in the data along which the data varies the most. Similarly, PC2 captures 30.84% of the total variance. It represents the second most important direction in the data, orthogonal (perpendicular) to PC1. A subset of the principal components that capture most of the variance is often chosen for further modelling, allowing for a more compact representation of the data while minimizing information loss (Jackson, 2005). In the PCA figure, a distinct separation within the samples can be observed. Samples $CSoC_{whole,w}$, $CSoC_{180\text{-}250,w}$ and $CSoC_{<180,w}$ were grouped on the left side, while samples $CSoC_{whole,wo}$, $CSoC_{180\text{-}250,wo}$ and $CSoC_{<180,wo}$ were grouped on the right side of the graphic. This grouping reflects the differentiation among the samples based on their characteristics.

The projection of the variables in the factor plane indicated that TS, EB, WVP and TSM contributed most significantly to the first principal component (PC1) at 23.03%, 22.17%, 16.84% and 24.77%, respectively. In contrast, thickness, DM and SD had a greater impact on the second principal component (PC2) at 39.55%, 38.46% and 21.61%, respectively to its variance.

Results of the Z-Score Analysis

The Z-score analysis was employed to determine the optimal preparation of film samples based on all investigated quality responses. In Table 3, the Z-score analysis results for samples, both with and without ultrasonic bath pretreatment and for all tested oil cake granulations, are presented. These results are segmented into S1–S7, corresponding to Z-score outcomes for observed responses, respectively. The presented results indicate that the $CSoC_{<180,wo}$ led to an increase in Z-score values for observed aspects of the quality characteristics. The maximal values of S6 (swelling degree) and S7 (solubility degree) and nearly maximal values of S2 (tensile strength) and S4 (water vapour permeability) were recorded for sample $CSoC_{<180,wo}$. Total Z-score values combine all segment Z-scores mathematically and indicate the optimal combination of all tested responses of the samples.

The $CSoC_{<180,wo}$ result in an optimal combination of the tested quality characteristics and achieved the most favourable combination of observed quality characteristics among the tested formulations.

CONCLUSIONS

This study dealt with synthesizing biopolymer films based on the *Camelina sativa* oilseed cake and investigated the impact of cake granulation and naturally present mucilage on properties of the obtained biopolymer films. The general conclusion is that a lower degree

of cake granulation and mucilage presence contributed to the optimal properties of the biopolymer films. The $CSoC_{<180,wo}$ film was selected as optimal because it exerted high values for mechanical properties and low values regarding barrier and physicochemical properties. This work represents a contribution to the valorisation of agro-industrial mass towards obtaining new materials with competitive properties compared to commercially available plastic materials that harmfully impact the environment. Further research will focus on optimizing the properties of the obtained films by applying different synthesis parameters (pH, temperature, concentration) to develop a biopolymer film with optimized properties for the purpose of food packaging.

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UTICAJ GRANULACIJE ULJANE POGAČE I ULTRAZVUČNOG PREDTRETMANA NA SVOJSTVA BIOPOLIMERNIH FILMOVA NA BAZI POGAČE *CAMELINA SATIVA*

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Sažetak: Cilj ovog rada je bila sinteza biopolimernih filmova na bazi pogače kameline (*Camelina sativa)*, koja zaostaje nakon hladnog ceđenja ulja iz semena. Prilikom sinteze filmova korišćene su tri različite granulacije pogače: cela pogača, frakcije veličine 180-250 µm i frakcija manja od 180 µm. Polovina uzoraka je podvrgnuta predtretmanu ultrazvučnim kupatilom kako bi se ispitao uticaj nativno prisutne sluzi iz pogače na osobine dobijenih filmova. U dobijenim uzorcima filmova ispitane su mehaničke, barijerne, fizičko-hemijske i strukturne osobine. Dobijeni su tamni, čvrsti i savitljivi filmovi. Primena predtremana uklanjanja sluzi doprinela je manjim vrednostima zatezne jačine i višim vrednostima izduženja pri kidanju. Značajno niže vrednosti propustljivosti vodene pare zabeležene su kod uzoraka koji nisu prošli predtretman uklanjanja sluzi. U pogledu fizičko-hemijskih parametera, optimalne rezultate su postigli uzorci granulacije manje od 180 µm bez obzira na primenu predtretmana. Strukturnih razlika među ispitanim uzorcima kao ni razlika u njihovom termičkom ponašanju nije bilo. Statističkom obradom dobijenih rezultata (Z-Score analiza) uzorak $CSoC_{<180,wo}$ je odabran kao optimalan, jer je imao visoke vrednosti parametara mehaničkih svojstava i niske vrednosti u pogledu barijernih i fizičko-hemijskih svojstava.

Ključne reči: *poljoprivredni otpad, veličina čestica, sluz, barijerna svojstva, mehanička svojstva, Z-Score analiza*

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