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THE MORPHOLOGY AND FUNCTIONAL PROPERTIES OF THERMOPLASTIC STARCH FILMS WITH PSYLLIUM HUSK (*PLANTAGO OVATA*) FLOUR ADDITION

ABSTRACT

This study examines the influence of psyllium husk flour (PF) on the structure and physical properties of thermoplastic starch (TPS) films. Films with 2 g and 6 g PF were produced using the casting method. Their morphology was analyzed by stereoscopic, scanning electron, and atomic force microscopy, while mechanical and wettability properties were evaluated. Increasing PF content improved impact strength but also increased brittleness. Higher PF concentration raised the water contact angle and reduced its change over time. The results indicate that psyllium husk flour effectively enhances the functional performance of TPS films, supporting their potential for sustainable packaging applications.

Keywords: *TPS/psyllium husk flour films, mechanical properties, water contact angle*

INTRODUCTION

Due to environmental pollution caused by the enormous amount of plastic waste, legal regulations have been introduced to limit the circulation of single-use packaging materials as a plastic waste [1]. The so-called Plastics Directive was implemented across the entire European Union. It was adopted in 2019 and focused on restricting or banning certain single-use plastic products, encouraging the use of sustainable alternatives, and promoting a circular economy. The Plastics Directive introduces several key measures to reduce plastic waste and promote sustainability across the EU. It includes a ban on specific plastic items such as straws, cutlery, plates, stirrers, and cotton buds. Additionally, member states are required to ~~take action~~ to reduce the consumption of plastic food containers and cups.

As a result, the development of packaging materials made entirely from natural, fully biodegradable materials has become an important research topic at many scientific institutions around the world. One of the promising alternative materials appears to be thermoplastic starch (TPS) [2]. Thermoplastic starch-based films are currently gaining much interest as an alternative to traditional polymeric materials. These materials owe their growing popularity to

their full biodegradability and wide availability [3]. The main barrier to the commercial use of thermoplastic starch (TPS) in film production for packaging applications is its low mechanical strength and high sensitivity to water [4]. These facts have led researchers to modify of TPS films using various functional additives [2]. Common additives used to improve the functional properties of thermoplastic starch films are for example, mineral additives [5, 6] as well as various types of natural extracts, fibers, and waxes [2, 4]. One of the interesting and promising functional additive, whose potential has not yet been fully tested and described in the literature, is *Plantago psyllium* (psyllium seed powder, psyllium husk) [7-9].

Psyllium seeds are commonly used in dietetics. The available information regarding their application mainly concerns:

- the positive impact on the digestive system (psyllium seeds are rich in fiber and mucilaginous substances; they support intestinal function) [10];
- lowering blood glucose and LDL cholesterol levels [11].

Psyllium husk is an inexpensive and readily available raw material. The few published studies on biodegradable films produced with this additive or directly manufactured from psyllium have thus far primarily utilized psyllium extracts, specifically psyllium mucilage/hydrocolloid [11, 12] psyllium seed gum [13]. Following by these authors, the modifications of biodegradable films using these extracts have shown promising results in terms of improving both mechanical and hydrophobic properties. However, obtaining psyllium extracts on a larger scale than laboratory scale entails significant costs, which undermines the feasibility of using them as a low-cost and widely accessible biopolymer filler. In the study by Tóth and Halász [7] authors demonstrated that psyllium seed husk and husk flour are suitable materials for preparing polysaccharide based edible films for food packaging just like psyllium seed mucilage. However, there is almost none research on psyllium husk / husk flour as a thermoplastic starch films filler.

The present study continues the authors' previous research on the direct use of psyllium husk flour (PF) to enhance starch-based films [8,9]. This form is readily available and does not require any special preparation prior to use in film casting. As such, it offers potential for the low-cost and straightforward production of packaging films.

To date, however, there is limited literature addressing the influence of PF on the properties of starch-based films, and no studies have investigated the effect of varying psyllium husk flour content on the microstructural and physical properties of such films. Therefore, the objective of this study was to examine the microstructural and physical characteristics of starch-based films containing different levels of psyllium husk flour (PF). The authors' previous work [8] investigated films based on TPS with the addition of unground psyllium husk. In that study, the films were tested under quasi-static loading conditions, whereas in the present work, the effect of psyllium husk flour on mechanical properties under dynamic loading conditions was evaluated. Assessing the behavior of the films under dynamic loading is important for the potential future use of these materials as packaging materials for example in agriculture. Our findings may serve as a valuable basis for the broader application of PF as a raw material in the large-scale production of packaging films.

MATERIALS AND METHODS

For the experiment, commercial potato starch from PPZ Trzemeszno (Trzemeszno, Poland) and psyllium husk flour from MIPAMA E.Z. Szafarz sp.j. Opatówek, Poland were ordered. The practice of casting thermoplastic starch films (Figure 1) with 2g (TPS/PF_2G) and 6g (TPS/PF_6G) of psyllium husk flour addition was as follows. Distilled water was used

to prepare film-forming solutions. Potato starch and psyllium husk flour were added to distilled water in the amount given in Table 1. The film casting process consisted of the following stages: 1. Heating and mixing; 2. Cooling and plasticizer addition; 3. Homogenization; 4. Casting into molds; 5. Drying; 6. Removal from molds and conditioning. Below is a detailed description of the process of producing starch-based films. The solution of distilled water and potato starch was heated to 80°C under constant stirring (300 rpm) using a magnetic stirrer - Steinberg SBS-MR-1600/1T (Steinberg Systems, Germany) for 30 minutes. Subsequently, the solution was cooled to 40°C and glycerol (Glycerol 99.5%; Stanlab, Poland; 1.3% w/w) was added as a plasticizer. The entire solution was mixed using an ultrasonic homogenizer TF-650N (Tefic Biotech CO., China) for 50 minutes. The mixtures were poured into round acrylic glass molds with an internal diameter of 90 mm and dried in a KBC-65 thermal research chamber (Amed, Poland) at 35°C for 20 hours. The drying process was carried out at 35 °C for 20 h in a laboratory oven under ambient pressure. These conditions were selected as a compromise between sufficient moisture removal and the limitation of starch chain reorganization associated with retrogradation. According to literature data, moderate drying temperatures in the range of 30–40 °C allow for effective dehydration of thermoplastic starch while preventing extensive recrystallization of amylose and amylopectin chains [14-16]. In contrast, prolonged storage at low temperatures or exposure to cyclic humidity changes has been shown to accelerate retrogradation due to enhanced molecular mobility and hydrogen-bond rearrangement. Although a certain degree of structural ordering may still occur under the applied conditions, it is expected to be limited and not to alter the general comparative trends between samples. Further studies will include X-ray diffraction (XRD) and differential scanning calorimetry (DSC) analyses to quantitatively assess the extent of retrogradation resulting from the drying process. After removal from the molds, the films were conditioned at the temperature of 22°C and 40% relative humidity (RH) for 24 hours.

The thickness of each film was measured with a digital micrometer with an accuracy of $\pm 1 \mu\text{m}$ just after it was peeled from the acrylic glass mold. Thickness measurements of five locations were taken for each film. For calculations, the mean value was used (Table 2).

Table 1. The compositions of the TPS/PF films

Ingredient	TPS/PF_2G (g)	TPS/PF_6G (g)
distilled water	754	754
potato starch	34	34
Glycerol	10.6	10.6
psyllium husk flour	2	6

Low-velocity impact tests were conducted using an Instron - Ceast 9340 drop weight testing machine. A 10 mm impactor diameter with a mass of 3.4 kg was employed, following the ASTM D 7136 standard [17]. Circular samples with a 40 mm internal diameter and 70 mm external diameter were tested, and the impactor stroke was centered on the samples by supporting the 100×100 mm specimens. Impact energy of 1 J was used, which correspond to impact velocity of 0.767 ms^{-1} . The test was conducted in 55 % relative humidity (RH) and an ambient temperature of 22°C. Four repetitions were performed for each type of material.

The microstructure of the surface of the films was observed with a NIKON SMZ18 (Tokyo, Japan) stereoscopic microscope equipped with a DS-Fi3 digital camera and NIS-Elements BR image analysis software as well as Phenom ProX electron microscope (Thermo Fisher Scientific, Waltham, MA, USA). Before SEM imaging, they were mounted without pre-treatment onto aluminum specimen stubs, using high-purity conductive double-sided adhesive carbon tabs. SEM images were collected in SE mode with accelerating voltages of 10 kV.

The fractures of the samples after impact tests were observed with Leica DM4000 M LED (Leica Microsystems, Wetzlar, Deutschland) versatile upright microscope for materials analysis with LED illumination.

The surface topography of the films was analyzed using atomic force microscopy (AFM) using an AFM NTEGRA Prima (NT-MDT BV, Apeldoorn, The Netherlands) in semi-contact mode. A silicon cantilever NSG30 (NT-MDT BV, Apeldoorn, The Netherlands) with an average resonant frequency of 300 kHz was applied. For each sample, five distinct areas measuring $10 \times 10 \mu\text{m}^2$ were analyzed at a scanning frequency of 1 Hz and a resolution of 512×512 pixels per image. The measurements were performed at room temperature, under ambient conditions, and at a relative humidity of 30%. AFM images capturing height and deflection data were obtained. These images were initially processed using plain transformations, followed by surface roughness parameter calculations using the NT-MDT software (NT-MDT BV, Apeldoorn, The Netherlands). The roughness parameters evaluated included the arithmetical mean deviation (R_a), the root-mean-square deviation (R_q), the maximum peak height (R_p), and the maximum valley depth (R_v) of the roughness profile. Analyzed parameters were calculated for 10 scan lines selected from a scanned area at equal intervals of 50 lines.

The wettability, measured through the water contact angle (WCA), was assessed using a goniometer (Drop Shape Analysis System DSA100, Krüss, Germany), which was equipped with a camera for recording and image acquisition. The contact angle was determined by observing the shape of a drop deposited on the smooth surface of the sample. The drop shape changes were recorded at 25 frames per second. Distilled water, with a volume of $2 \mu\text{l}$, was chosen as the standard liquid. Because of the initial oscillations of the deposited drops, stable contact angle readings were only possible once the kinetic energy had dissipated. The water drops stabilized after 1–2 milliseconds. Measurements continued until a maximum of 2 seconds had passed. Each measurement was repeated three times under consistent temperature (20°C) and relative humidity ($\text{RH} = 45\%$) conditions.

RESULTS AND DISCUSSION

Figure 1 (a–d) shows representative microstructure images of the surfaces of the tested films (TPS/PF_2G; TPS/PF_6G) observed using a stereoscopic microscope (Figure 1a–b) and scanning electron microscope – SEM (Figure 1c–d). Both TPS/PF_2G and TPS/PF_6G films are characterized by a quiet homogeneous structure (Fig. 1a–d). However, the specimens with a higher psyllium husk flour content display a slightly rougher and less compact surface morphology (Figure 1d) with larger amounts of ground husk residue visible (Figure 1b). For both tested cases, no discontinuities in the surface microstructure were observed.

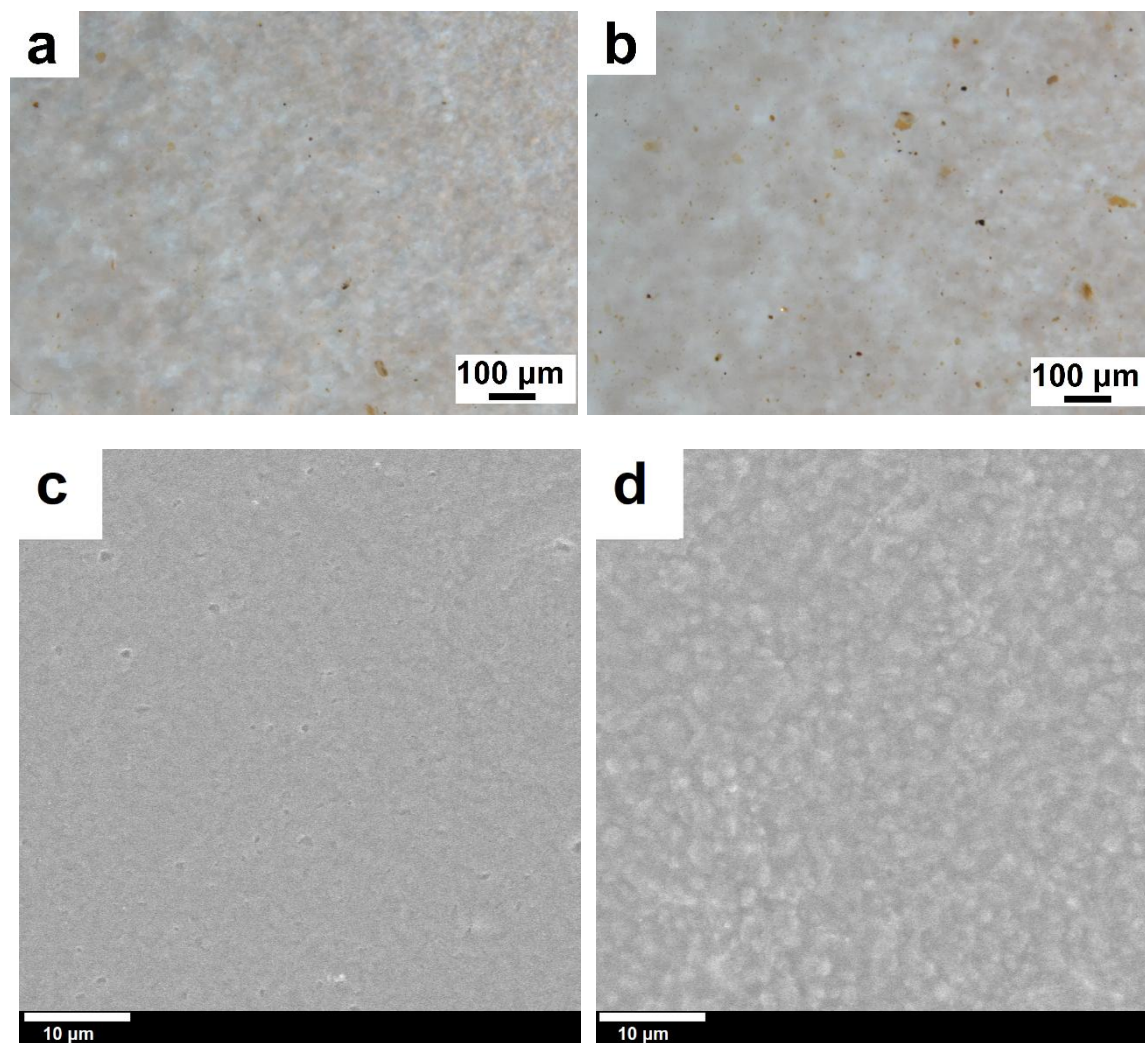


Fig 1. Surface microstructure of TPS films with PF addition: a, b – stereoscopic microscope; c, d – SEM; a, c – TPS/PF_2G; b, d – TPS/PF_6G

In the previous studies conducted by the authors on pure starch films (without psyllium), the presence of small round structures was observed, which were suspected to be remnants of incompletely gelatinized starch [8]. However, with the addition of psyllium husk, the round structures were no longer observed. Similar results were also noted for pure starch films produced in the extrusion process by Cheng et al. [18] and Huntrakul et al. and Wang et al. [19, 20]. Comparable observations were made for films produced by blown extrusion with pea protein [20] and for polyvinyl alcohol/clay nanocomposite films [21]. The absence of “round granules” in microstructure suggests that the addition of psyllium promoted starch gelatinization. An analogous effect was reached in the study by Cheng et al. [21] for TPS films with ϵ -PL addition. The authors point out that it is affected by hydrogen bonds’ interaction with starch granules under high shear and temperature conditions, which leads to a reduction in the intermolecular forces along starch chains and the disruption of starch granules. The formation of additional intermolecular hydrogen bonds in the thermoplastic starch films with psyllium husk addition was confirmed by FTIR analysis, as described in our previous study [8]. The authors assume an analogous phenomenon for the addition of psyllium flour in this study. However, hypothesis requires further confirmation through infrared spectroscopy analysis.

The surface topography characteristics of the samples are shown in Figure 2. The TPS/PF_6G surface was more diversified, with more peaks and valleys compared to the TPS/PF_2G sample. The mean maximum valley depth (R_v) and the maximum peak height (R_p) were also the greatest for TPS/PF_6G samples (Table 2). Mean values of these parameters were 226.57 ± 59.80 nm and 251.25 ± 68.74 nm respectively. In the case of TPS/PF_2G samples, these values were much lower and amounted to 118.64 ± 30.13 nm and 157.31 ± 38.26 nm respectively. The arithmetical mean deviation (R_a) and the root-mean-square deviation (R_q) were also greater for samples with the addition of 6g of psyllium husk flour. Quite large standard deviations of all measured parameters indicate large irregularities in the surface of the obtained films (Table 2). However, the analysis of variance (one-way ANOVA) performed showed significant differences in the analyzed parameters between the studied groups at a significance level of 0.05.

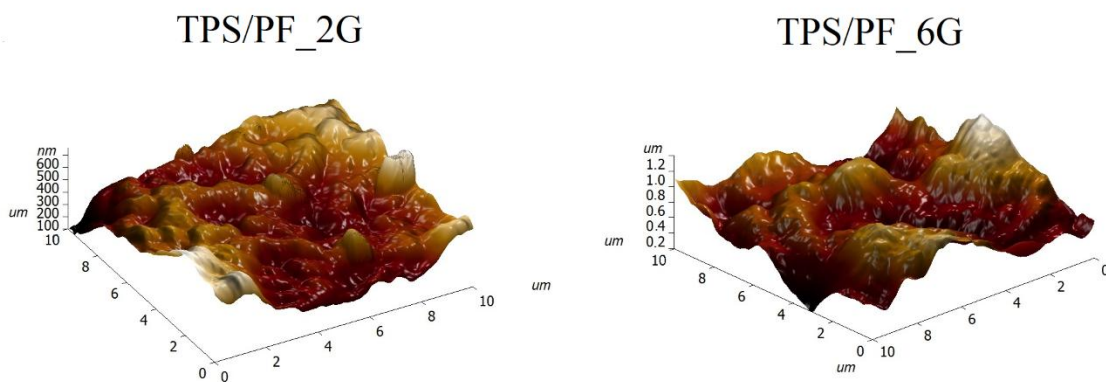


Fig 2. Atomic force micrographs of surface topography of TPS/PF films

Table 2. The thickness and surface roughness parameters of the films

Sample	Thickness (mm)	R_a (nm)	R_q (nm)	R_p (nm)	R_v (nm)
TPS/PF_2G	0.12 $\pm 0.02^a$	51.28 $\pm 17.02^a$	73.06 $\pm 16.65^a$	157.31 $\pm 38.26^a$	118.64 $\pm 30.13^a$
TPS/PF_6G	0.13 $\pm 0.03^a$	112.66 $\pm 32.43^b$	136.19 $\pm 38.38^b$	251.25 $\pm 68.74^b$	226.57 $\pm 59.80^b$

Data are shown as mean $\pm$ standard deviation. Means in a column with the same superscripts are not significantly different ($p < 0.05$).

Food packaging materials must exhibit strong mechanical performance and durability to preserve their structural integrity during handling, transportation, and storage [7]. Evaluating the impact resistance of such materials is essential to assess their behavior under real-world usage conditions. Low-velocity impacts can occur during service or maintenance activities, causing various types of damage. This damage is particularly dangerous because it is not easily detectable visually, yet it can significantly affect the residual properties [23]. In this context, destructive impact tests were carried out on samples incorporating psyllium husk flour, allowing for the analysis of time-dependent energy absorption by the films. Furthermore, the testing device's software calculates both the peak load applied and the maximum displacement of the impactor. From the curves showing the evolution of energy over time (Figure 3), it was not possible to identify the onset of the curve plateau — a characteristic feature of materials subjected to impact [24] — because complete perforation

occurred, defined as the moment when the impactor passes entirely through the sample.

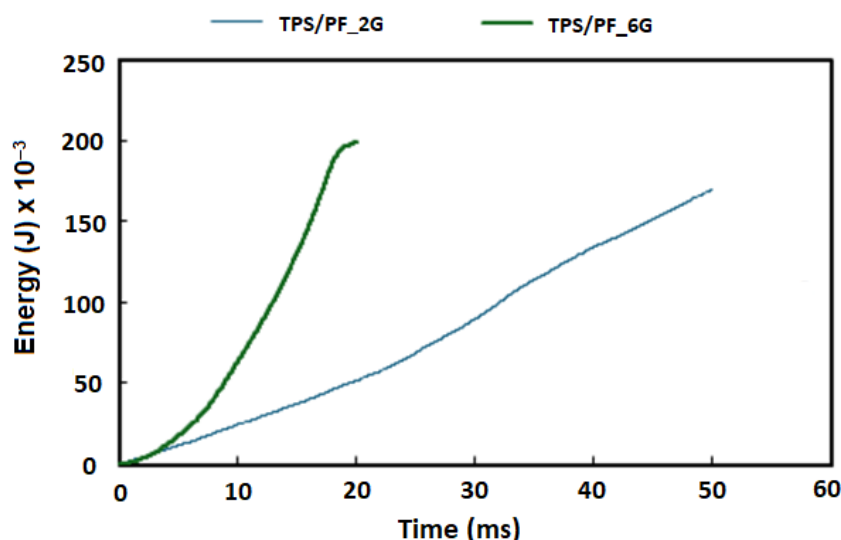


Fig. 3. Representative curves Energy-Time of the TPS/PF films registered during the impact tests

Table 3. Average values of parameters obtained from the impact tests

Sample	Maximum Force (N)	Max. Displ. (mm)	Absorbed Energy (J)
	Average	Average	Average
TPS/PF_2G	8.7 ± 1.8	48.5 ± 0.08	0.21 ± 0.034
TPS/PF_6G	23.7 ± 3.8	15.7 ± 2.8	0.19 ± 0.003

As can be seen in Table 3, the highest absorbed energy is obtained for the TPS/PF_2G samples, which corresponds to the greatest damage, lowest impact force and highest displacement. Compared to previous studies conducted using a patented device [25, 26], the authors obtained significantly higher maximum load values and lower impactor displacement values for samples with 2g of psyllium husk flour addition [9]. However, those earlier tests were carried out at a lower relative humidity of 40% RH.

Improved mechanical properties (under quasistatic loading conditions) were also reported by the Tóth and Halasz [1] for films prepared of psyllium husk flour compared to films manufactured of psyllium seed or psyllium seed husk. The authors attributed the enhanced tensile strength of the films to the smaller particle size of the psyllium husk flour, which resulted in a higher surface area and stronger interfacial interactions between the filler and the polymer matrix. In our study, the addition of 6g of psyllium husk flour used contributed to increased impact strength of the TPS film. It can be suspected that the mechanical strength of the films with the greater addition of psyllium husk flour may be related to the presence of more hydrogen bonds, as confirmed by FTIR spectroscopy results for samples with psyllium husk addition in previous research [8], but this is also partly consistent with other studies [27, 28]. The difference, however, is that in our case there is no protein matrix (from whey [27] or soy [28]) for the formation of hydrogen bonds, and they can only be formed between polysaccharides in starch and psyllium husk.

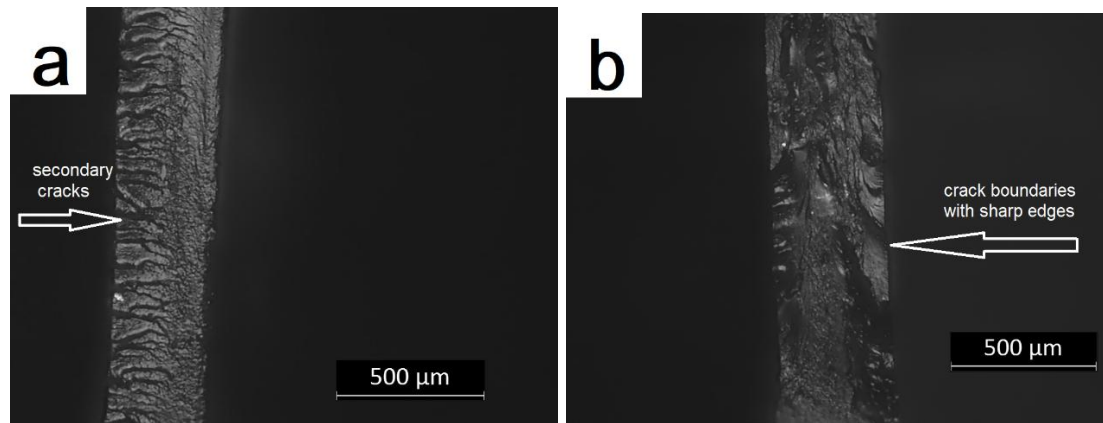


Fig. 4. Representative microstructure observations of the fractures: a – TPS/PF_2G; b – TPS/PF_6G

From the images of the fracture surfaces resulting from the impact tests of the samples TPS/PF_2G and TPS/PF_6G, it can be noted that these surfaces have significantly different morphologies. While the fracture surface of the TPS/PF_2G sample is more uniform, the morphology of the fracture surface of the TPS/PF_6G sample is more irregular, indicating that the material with the greater addition of psyllium husk flour has a more brittle fracture behaviour. This is also evidenced by the sharp edges of the fractured samples marked in Figure 4b. In contrast, for the TPS/PF_2G films, secondary cracks were observed, indicating a change in fracture behaviour towards a more ductile character – Figure 4a. By relating these observations to the values obtained in the impact tests, it can be concluded that the increase of psyllium husk flour addition improves the impact strength of the films, although it favours an increase in brittleness, which is confirmed by the reduced displacement in the impact tests and the change in the morphology of the fracture surface of the TPS/PF_6G samples.

The behavior and shape of water drops on the TPS/PF films as a function of time are illustrated in Figure 6a and b. The initial value of water contact angles (WCA) measured upon water drop contact with the surface of starch films was approximately 103.5° for TPS/PF_3G and 111.05° for TPS/PF_6G. Simultaneously, a small decrease in WCA values was observed over time (Figure 5). Higher change dynamics were observed within the first second, in which the WCA value was reduced to 98.45° for TPS/PF_3G and 110.80° for TPS/PF_6G samples. This corresponded to 4.9% and 0.2% of the initial WCA value. In the time interval from 1 to 2s, WCA dynamics decreased and contact angle changes did not exceed 1.2% for TPS/PF_3G and 0.1% for TPS/PF_6G films.

Compared to Beer-Lech et al. [8] studies demonstrating the wettability of films with psyllium husk (1% w/w), films with psyllium flour addition are characterized by a significantly higher initial WCA. For films manufactured with psyllium husk, an initial WCA of 96° was noted [8]. The hydrophilic character of biodegradable films modified with psyllium husk was reported by Ahmadi et al. [11]. Their investigations of psyllium-based films without thermoplastic starch, and incorporating varying concentrations of glycerol, revealed substantially lower WCA values.

It is widely recognized that as a surface becomes more hydrophilic, its water contact angle (WCA) tends to decrease, making WCA a key parameter for assessing the hydrophobicity of polymeric materials [29]. The WCA of biopolymers can range from 0°, indicating complete wetting, to 180°, which reflects a theoretical state of total non-wettability [30]. A material is generally considered hydrophilic if its contact angle is less than 90°, and hydrophobic if the angle exceeds 90° [29, 31].

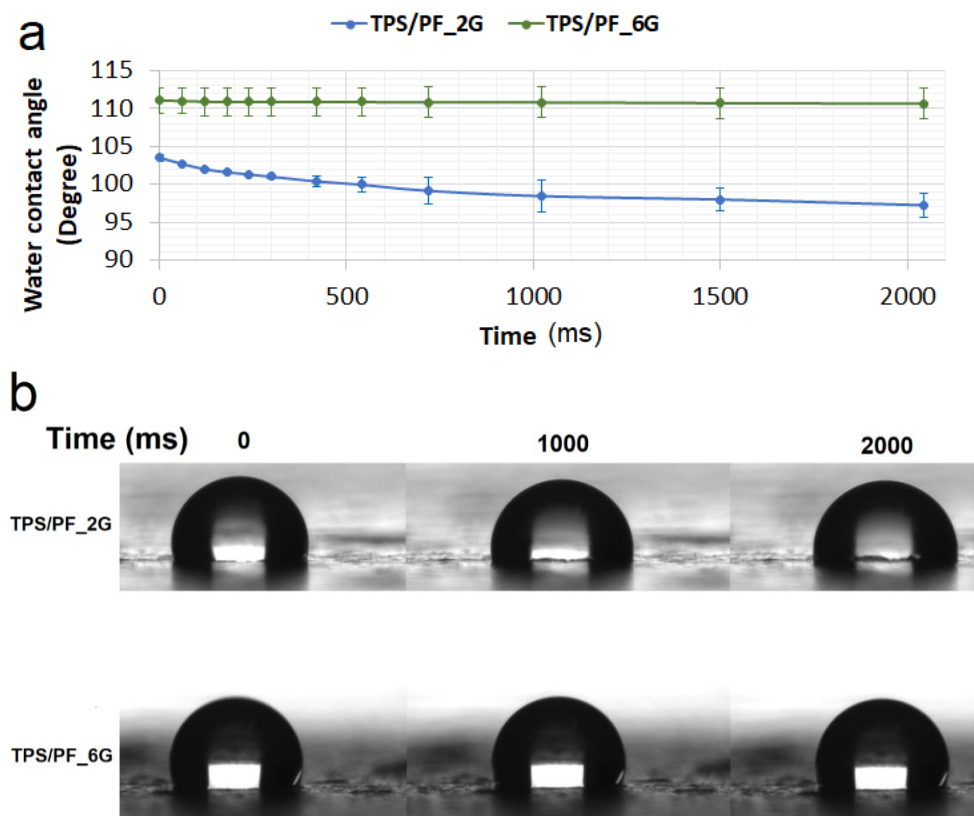


Fig. 5. Behavior (a) and shape (b) of water drops on the tested films as a function of time

The initial water contact angle (WCA) values are predominantly governed by the surface chemical composition. Elevated initial WCA readings typically reflect a moderately hydrophobic surface character. As reported by Białopiotrowicz [32], during the film formation process, the hydrophobic and highly branched regions of the amylopectin macromolecule preferentially orient toward the air interface, forming a gel-like outer layer that critically influences the initial wettability profile of the material. This mechanism may account for the high initial WCA values observed in both TPS/PF_2g and TPS/PF_6G films. Nonetheless, steric effects may induce the formation of polar microdomains created by interactions between functional groups along the carbon chains [32].

In thermoplastic starch films with psyllium husk addition (in the form of flour or whole husks), the dominant polar moieties originate not only from glucose units (from starch), but also from additional hydroxyl-rich constituents—mainly mono- and disaccharides—further contribute to surface polarity.

The increased initial WCA and lower dynamics of its changes for films with higher psyllium flour content (TPS/PF_6G) compared to TPS/PF_3G films may be justified by the much higher surface roughness observed during analyses performed using an atomic force microscope (Figure 2; Table 2) and also visible in SEM images (Figure 1). Wenzel's theory describes the wetting of rough surfaces [33]. It assumes that the liquid's penetration into irregularities depends on the chemical nature of the surface and the effective contact area between the liquid and the solid. As a result, surface roughness may enhance both hydrophilicity (the ability of water to wet) and hydrophobicity (the ability to repel water), depending on whether the surface is originally hydrophilic or hydrophobic.

Conversely, Zhang et al. [13] demonstrated that incorporation of psyllium husk gum into whey protein composite matrices resulted in significantly elevated WCA values, ranging

from approximately 98° to 128°, indicating increased hydrophobicity relative to starch-based systems. The authors noted, following Xia et al. [34], that the hydroxyl groups from psyllium seed gum may form hydrogen bonds between protein molecules at amine, amide, carboxyl, and hydroxyl sites, which enhanced the interactions between protein chains and reduced the accessibility of hydroxyl and hydrogen bonds between the polymer and water molecules.

The presence of proteins may improve the hydrophobicity of psyllium-filled or psyllium-based films. Since films discussed in our investigations are protein-free, further studies are planned to investigate starch films incorporating whole psyllium seed flour.

CONCLUSIONS

The above results continue the authors' previous research on the effect of psyllium (*Plantago ovata*) seed materials on the properties of TPS-based films. This work focuses on the use of psyllium husk flour as an ingredient for improving the physical properties of films based on thermoplastic potato starch. Analysis of the mechanical properties of the films under dynamic loads, which is the subject of the present study, has not previously been conducted for these materials.

The casting method was used to prepare film samples containing various amounts of psyllium husk flour. To evaluate the mechanical and chemical behavior of the starch films, impact tests and contact angle measurements were performed. Microstructural analysis was conducted using optical and electron microscopy. The results lead to the following main conclusions:

- The increased psyllium husk flour content improves the impact strength of TPS films and also promotes an increase in brittleness under conditions of 55% RH. For the TPS/PF_6G films, an approximately 2.7-fold higher maximum impact force (23.7 N) and more than three-fold lower impactor displacement (15.7 mm) were observed compared to the TPS/PF_2G samples (8.7 N; 15.7 mm).
- The addition of psyllium husk flour increases the initial WCA (from 103.5° for TPS/PF_2G to 111.05° for TPS/PF_6G) and reduces the rate of WCA decrease in films based on thermoplastic potato starch.
- The addition of psyllium husk flour shows strong potential for improving the physical properties of TPS-based films.

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Conflicts of Interest

The authors declare that they have no competing financial interests or personal relationships that could have seem to influence the study reported in this paper.

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