

Strength Analysis Aspects of Psyllium/Thermoplastic Starch Films under Impact Loading Conditions

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ABSTRACT

Tensile test under quasi-static loads conditions is usually used to determine the mechanical strength of thermoplastic starch films. This kind of test does not fully illustrate the load conditions for packaging films, which, under the conditions of use, are succumb to dynamic loads. Thus, the aim of the study was to present the possibilities of using a patented soft tissues measurement testing station to analyze the mechanical strength of thermoplastic starch (TPS) films under impact loading conditions. Two groups of film specimens containing the addition of psyllium husks (TPS/PH) and psyllium flour (TPS/PF) were used for the measurements. The casting method was applied, and glycerol was used as a plasticizer. Microstructure of the specimen surface was analyzed by stereoscopic microscopy. Specimens with the addition of psyllium flour had a more uniform microstructure. The maximum breaking forces obtained during impact tests for these films were 5 times higher than specimens containing psyllium seed husk. The same behaviour was found with respect to stresses with average values of 48.6 MPa for TPS/PF and 20.2 MPa for TPS/PH. Moreover, research confirms usefulness of patented soft tissues measurement testing station to analyse the mechanical strength of thermoplastic starch films.

Keywords: impact loading, pendulum, mechanical properties, biocomposites, thermoplastic starch film, psyllium.

INTRODUCTION

Industrial development, especially in the last few decades, along with the increase in the production and consumption of goods, contributed to the increase in the supply of plastic packaging. Pollution by plastic waste poses a serious threat to the environment as a whole. It creates problems for both wildlife and mankind. Each year, from 6 to almost 15 million tons of plastic waste end up in the oceans [1, 2]. The problem of polymer plastic waste is so huge that even its selective collection does not help to reduce its quantity. At the same time, more and more new sources of plastic pollution are being discovered, posing a threat both to the environment and human health [3]. Microplastics – tiny pieces of plastic (less than

5 mm in size) accumulate in the ocean, and their small size makes it easy for marine organisms to swallow them [4]. Recent observations also indicate the presence of microplastics in the air, rainwater, drinking water, some food products, and even in human blood. It is also worth noting that plastic waste decomposes very slowly – up to several hundred years as in the case of plastic bottles or cups, which only increases the scale of the problem of environmental pollution with such waste over time. Thus, some of the plastic waste generated today will pose a serious problem for the next few generations.

The European Union requirements [5] prohibiting the marketing many kinds of plastic products, generate new challenges for scientists and producers of disposable packaging and

packaging films. These legal solutions determine the change in the perception of packaging materials and prompt scientists to develop environmentally friendly, biodegradable biopolymers from naturally abundant materials as a response to the problem of ecosystem pollution [6]. Using biodegradable films and coatings in packaging that will have similar functional properties to their plastic counterparts is a good way to reduce the amount of synthetic polymeric materials and to extend the shelf life of food products. One of the proposed solutions which is currently gaining importance is manufacturing packaging materials (including packaging films) based on thermoplastic starch (TPS). In order to increase the functional properties of TPS films, various functional additives are used. These include, for example, natural fibers, seeds, protein additives, ceramic additives, or waxes [7–10]. One of the additives that is currently gaining popularity is psyllium plantain and its various physical forms (seeds, husk, flour, or psyllium husk gum) [11, 12]. Psyllium is obtained from the seeds of the plant genus *Plantago* and *Plantago ovata*, which has more than 200 species [13]. Main advantages of psyllium seeds are: wide nutritional value, low cost, non-toxicity, biodegradability, and biocompatibility [14, 15].

Improved physical, mechanical, and barrier properties of biodegradable films after using psyllium husk have already been demonstrated. Some of studies describes films made by the extraction of psyllium hydrocolloid and manufacturing film directly from psyllium seed [11], while other research use only small amounts of psyllium husks as an additive to TPS films [16]. As reported in the literature, the use of psyllium husk as an additive to TPS films results in reduced free volume and moisture diffusion, which in turn leads to improve the water vapor barrier properties of the biodegradable films [17, 18].

From the above considerations it is clear that psyllium seeds (in various forms) seem to be a promising raw material for producing edible films or coatings. However, so far not much research has been carried out on the properties of this type of films in the context of their mechanical strength under dynamic loads conditions. Most research papers discussing the strength of biodegradable films focus on testing the tensile strength [19–21]. This approach oversimplifies the considerations and limits them only to the analysis of the films' resistance to quasi-static loads. This approach does not allow for the analysis of the strength of

the films in the conditions of their potential use, i.e. under dynamic loads conditions. In the few manuscripts containing impact tests of biodegradable films, the authors focus only on comparing the strength of films with different compositions without making an in-depth analysis of the behaviour of films during impact [22]. Such research is carried out on commercial devices.

The aim of our study was to demonstrate the possibility of using a patented stand for impact tests of soft tissues [23], originally dedicated to strength analyses mainly of plant tissues, for dynamic strength tests of biodegradable TPS films. For this purpose, an appropriate holder for mounting films was used – a special frame, which is a registered utility model [24]. The reaction forces of the films to the action of the impactor were determined, along with the recording of their time courses and the calculation of the values of destructive stresses under impact loads. The designed impact station consisted of two parts: measuring and forcing. The rigid pendulum allowed for obtaining an appropriate speed of displacement of the central point of the tested specimens, and the measuring system made it possible to determine and record the course of the reaction force of the film depending on the displacement of the impactor.

The subject of our research was TPS films with the psyllium husk and psyllium flour addition. Similar strength analyses addressing the problem of mechanical resistance under impact loads conditions, on this type of materials, have not been conducted or described anywhere so far.

MATERIALS AND METHODS

For the preparation of the films, commercial potato starch was purchased from PPZ Trzemeszno (Trzemeszno, Poland), and the casting method was applied. Functional additives in the form of psyllium husk from Witpak (Kielce, Poland) and psyllium flour from MIPAMA E.Z. (Szafarz sp.j. Opatówek, Poland) were used. Both forms of plantain come from *plantago ovata* seeds. The solution of distilled water and psyllium husk/psyllium flour was heated to 80°C under constant stirring (300 rpm) using a Steinberg SBS-MR magnetic stirrer -1600/1T (Steinberg Systems, Zielona Góra, Poland) for 30 minutes. Then, the solution was cooled to 40°C and 1.3% (w/w) of glycerol was added in the amount given in Table 1. The complete

Table 1. The compositions and thicknesses of the films

Specification	TPS/PH	TPS/PHF
Distilled water (g)	754	
Starch (g)	34	
Glycerol (g)	10.6	
Psyllium husk/ psyllium flour (g)	2.0	2.0
Thickness (mm)	0.1 ± 0.04	0.13 ± 0.02

solution was mixed using an ultrasonic homogenizer TF-650N (Tefic Biotech CO., Beijing, China) for 50 minutes. The suspensions were poured into 200×200 mm acrylic glass molds and dried in a KBC-65 thermal research chamber (WAMED, Warszawa, Poland) at 35°C for 20 hours. After removing from the molds, all films were conditioned (for 24 hours) at the temperature of 22°C and 40% relative humidity (RH). The physical properties of films produced in the same way using plantago ovata are discussed in more detail in the article [13]. The microstructure of the specimens were observed with a Nikon SMZ18 stereoscopic microscope equipped with a DS-Fi3 digital camera.

The puncture impact test is a destructive test that allows to obtain time courses of the impact force and deformation from the beginning of the impactor’s contact with the film surface until its breakage after exceeding the maximum impact force value. Film specimens were placed between two identical frames with a centrally made hole (diameter of 120 mm) and clamped with four levers (Figure 1).

Impact tests were preceded by appropriate preparation of film specimens. For this purpose, film sheets were cut with a knife to create

specimens (140×140 mm). Thickness measurements of five locations (Figure 1b) (one in the center (point A) and four around the perimeter of each film (40 mm away from the center of the specimen) were taken with a digital micrometer (accuracy of ±1 μm). Ten repetitions of measurements were made, and the mean value was used for the calculations. The films prepared in this way were placed between the frames to create FAF.

The edge of the frame hole was rounded to limit the adverse effect of the shape of the frame on the deformed surface of the specimen (during impact). As a result, an appropriate clamp of the specimen was obtained, allowing it to be tightened during the movement of the impactor in the perpendicular direction to the film surface.

A set of frames and film (FAF) connected to the vertical plate was part of the test stand (Figure 2). The test stand used for film impact tests (Figure 2) consists of: two columns connected by three horizontal connectors and fixed to the wall; a steel base plate; and the main plate (the main plate can be moved vertically). The main plate is equipped with FAF lever clamps. The pendulum arm is mounted on the axis of an incremental angular displacement sensor by Heidenhain (Germany), model RON 275, with an accuracy of 0.005°. A piezoelectric force sensor by Endevco (USA), model 2311–100, with a sensitivity of 2.27 mV·N⁻¹ and a measuring range of ±220 N was attached to the sensor holder placed on the pendulum arm. A spherical impactor with a diameter of 25 mm was screwed to the sensor. Correct positioning (centering) of the impactor in relation to the FAF was carried out by screws to adjust the vertical displacement of the main plate. The

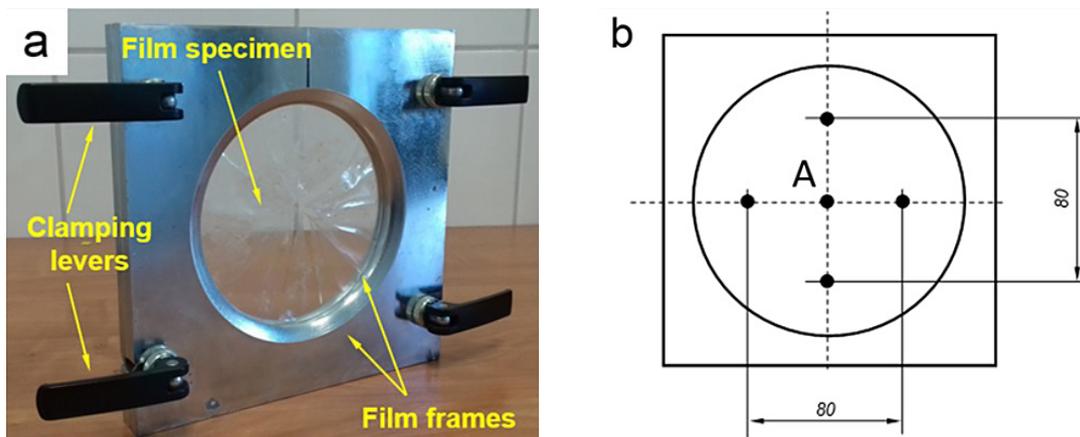


Figure 1. Film specimen mounted in frames: a – frames and films (FAF), b – diagram of the arrangement of points for measuring the thickness of the specimens with the outline of the frames for mounting – A – the center point of the specimen

sensor was connected to the SCB-68 measurement card by National Instruments (USA) which sent signals to the application created in LabView ver. 8.6.1 (National Instruments), from which the measurement of the angular displacement of the pendulum in time was directly triggered.

The dynamic puncture test was realized through hitting a spherical impactor in the middle of a film specimen placed between metal frames (Frames and Film). Appropriate immobilization of the film (pressing it with a clamping levers) allowed for permanent fixing, enabling the film to be stretched in two directions. The impact force was generated as a result of the dynamic impact of the falling arm of the physical pendulum, and the value of the impact velocity V_{imp} was $1 \text{ m}\cdot\text{s}^{-1}$. Figure 3 shows a film specimen mounted in frames (FAF). This system was, in mechanical terms, a thin-walled shell fixed around the circumference, which was dynamically affected by the impactor causing its deformation; and after exceeding the breaking stress value, the film cracks. Before starting the test, the pendulum was set vertically and the indications of the angular displacement sensor in the LabView dialog window were set to zero. Then, the recorded values of the force course and the angular displacement were started in the software dialog box and the pendulum arm was tilted by the angle allowing to obtain the

specified velocity value. The course of the reaction force of the film and the angle of rotation of the arm over time was observed on the monitor screen. Six repetitions of the impact strength test were performed for each group of specimens.

Figure 3a shows the deformation of the film specimen due to the action of the spherical impactor. The displacement in the direction of impact, at the moment preceding the fracture, is marked as l_r . During impact, the concentrated impact force P from the impactor is distributed on the spherical contact surface in the form of surface pressure p (Fig. 3b). For the part cut off by the $B-B$ meridian (Fig. 3b), assuming that the surface pressure $p = \text{const.}$, the resultant impact force $-P$ (acting on the part of the shell that is in contact with the spherical impactor) – can be calculated from the formula [25]:

$$P = p\pi r^2 \quad (1)$$

where: P is the load acting on the film surface (shell) at the moment before the specimen breaks (impact force); p is the surface pressure occurring on the contact surface of the impactor and the film specimen; π is the angle between the normal plane to the coating surface passing through the center of the impactor and the direction of the impact force; r is the radius of the shell for the part cut off by the $B-B$ meridian.

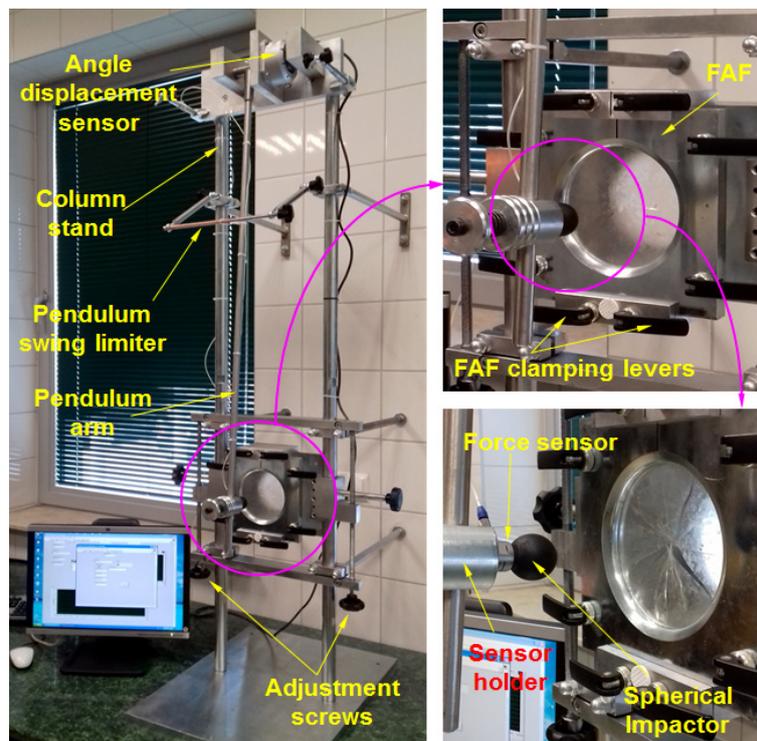


Figure 2. Measuring stand for dynamic testing of film specimens

Therefore, the surface pressure can be calculated from the formula:

$$p = \frac{P}{\pi r^2} \quad (2)$$

The general form of Laplace's equation for a spherical shell (contact of the impactor with the film specimen) [25] can be written as follows:

$$\frac{\sigma_p}{\rho_1} + \frac{\sigma_t}{\rho_2} = \frac{p}{g} \quad (3)$$

where: σ_p is the meridional stress; σ_t is the latitudinal stress; ρ_1 is the radius of curvature in the meridian plane; ρ_2 is the radius of curvature in the latitudinal plane; and g is the thickness of the film specimen.

Because for a spherical shell: $\rho_1 = \rho_2 = R$ and $\sigma_p = \sigma_t = \sigma$ (Figure 3b, c), after substitution into Eq. (3), it leads to:

$$\frac{2\sigma}{R} = \frac{p}{g} \quad (4)$$

The stress in the cross-section of the specimen is expressed as follows:

$$\sigma = \frac{p \cdot R}{2g} \quad (5)$$

Taking into account Eq. (2), the following is obtained:

$$\sigma = \frac{P \cdot R}{2\pi g r^2} \quad (6)$$

The boundaries of the contact surface of the spherical impactor with the film specimen are determined by the value of r , which can be expressed in terms of the radius of curvature of the

impactor R and the angle α . It can be determined from the formula:

$$r = \sqrt{R^2 - (R \cos \alpha)^2} \quad (7)$$

or:

$$r = \sqrt{R^2 - (R - h)^2} \quad (8)$$

where: h is the height of the shell cut off by the $B-B$ meridian, equal to the displacement of the impactor along the direction of action from the beginning of contact with the specimen surface until its fracture (see Figure 3b).

The displacement l_p , i.e. the length of the arc traversed by a pendulum of length l_p , was determined by measuring the angle of rotation of the arm during the impact test and was calculated from the formula:

$$l_p = \frac{\pi \cdot \beta_p \cdot l_p}{180^\circ} \quad (9)$$

where: β_p is the angle of rotation of the pendulum arm from the beginning of the impactor's contact with the specimen surface until its fracture measured in degrees; and l_p is the length of the pendulum arm used in impact tests. For testing purposes, it was assumed $l_p = 1000$ mm (Figure 2).

The measurements show that the angle of rotation of the pendulum arm β_p is below 2° , which means that the displacement l_p can be considered a straight section, i.e. l_r . The determined h values made it possible to calculate the radius r from Eq. (8) and the stress σ from Eq. (6).

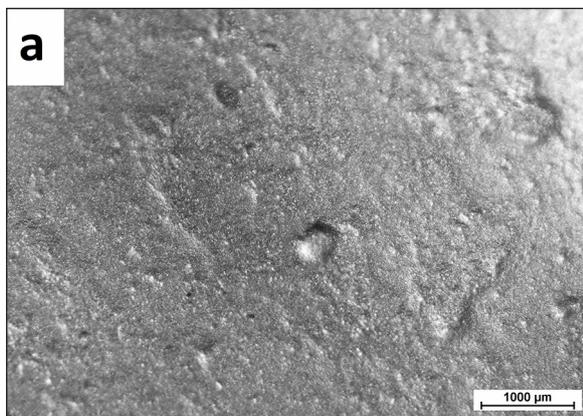


Figure 4. Surface microstructure of films with psyllium additives: a) psyllium flour – TPS/PF, and b) psyllium husk – TPS/PH

RESULTS AND DISSCUSION

Figure 4 shows the microstructures of the films with psyllium addition: flour (Figure 4a) and husk (Figure 4b). There are clear differences between the specimens. Films with flour addition have a smoother structure than specimens with the addition of husk (Figure 4b) where it is possible to see the elliptic cavity with round edges specific to *Plantago psyllium* seed [26]. No cracks and surface discontinuities were found in the tested specimens.

Food packaging requires good mechanical properties and durability to maintain structural integrity of product during shipping, handling, and storage of food [1, 27]. Therefore, testing the impact strength of this type of materials makes it possible to assess them under the conditions of their potential use. The impact tests performed on the specimens, as already mentioned, are destructive tests and enable to obtain time courses of the impact force of the impactor on the film layer attached between the frames (FAF – Figure 1). Table 2 presents a summary of the average values obtained during impact tests for both groups of specimens, i.e. TPS/PF and TPS/PH.

Figure 5 shows exemplary force-time P/t_r courses. The initially slowly progressive nature of these changes (force changes over time) can be explained by the significant flexibility of the film at the beginning of the test. However, in the final phase of the test, a faster increase of the force was observed, almost linearly, until the destructive

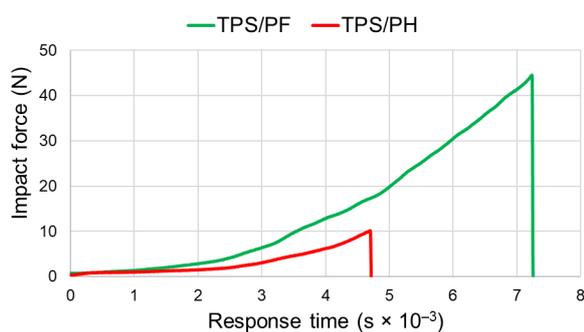


Figure 5. Representative TPS/PF and TPS/PH films responses during mechanical impact

value was reached. The average value of the maximum reaction force for the PH film was 9.55 N and the rise time was $t_r = 4.71 \text{ s} \cdot 10^{-3}$ (corresponding to the displacement of the impactor $l_i = 10.29 \text{ mm}$), (Table 2).

Films with the addition of flour (TPS/PF) reacted differently. These specimens were characterised by much greater resistance to impact, and the average value of the maximum reaction force was 48.54 N. The addition of flour also resulted in greater flexibility of the film, the average displacement of the impactor (compared to the TPS/PH film) increased to $l_i = 16.02 \text{ mm}$, and at the same time the t_r value was extended to $7.25 \text{ s} \cdot 10^{-3}$, (Table 2).

In the study of Sukhija [28], researchers emphasise that psyllium can be a beneficial addition to biodegradable films in the context of improving mechanical properties (in this case, tensile strength). Scientists explain this by fact that the addition of hydrophilic psyllium husk might have contributed to increased tensile strength by inducing new links between starch-protein matrixes, due to its hydrocolloid nature.

Also better mechanical properties (for quasi-static loads) in the case of films with the TPS/PF addition were also obtained by the authors in the paper [1]. They found that the reason for the increased tensile strength of the film containing the addition of psyllium flour was the smaller size of additives, therefore the high surface area of the particles and the strong interactions between the filler and the matrix highly improved their strength. In our case, the form of psyllium flour was characterised by greater impact strenght. Studies using Fourier infrared spectroscopy and in-depth qualitative and quantitative analysis of the microstructure of films in order to explain the origin of these changes.

The representative punctures of the specimens occurring during the impact tests are shown in Figure 6. The puncture form in each impact tests was repeatable within each of the tested groups of specimens. Considering the test method, i.e. stretching with a spherical impactor (equality of meridional and latitudinal stresses), a relationship between the form of puncture and the form of the

Table 2. Average values of quantities obtained from the impact tests of film specimens

Additive	Impact force, P [N]	Response time, t_r [$\text{s} \cdot 10^{-3}$]	Impactor displacement, l_i [mm]	Stress, σ [MPa]
TPS/PF	48.54 ± 9.6	7.25 ± 1.14	16.02 ± 2.19	48.46 ± 9.64
TPS/PH	9.55 ± 0.92	4.71 ± 0.07	10.29 ± 0.10	20.20 ± 2.33

additive used was observed. Star-shaped cracks, see Figure 6a, observed for the TPS/PH films, indicate that the values of longitudinal (latitudinal) stresses have been exceeded. However, the damage in Figure 6b for the films with the addition of psyllium flour exhibits visible crescent moon shape, resulting from increased circumferential (meridian) stresses. This type of damage suggests the presence of unequal stress values on the material surface which may be the result of a local change in the thickness of the specimen, or possible local heterogeneity of the surface. Clarification of this issue requires further microstructure analyses using scanning electron microscopy.

Similar images of specimens cracks under the influence of impact loads (in the form of multiple radial cracks: “star-shaped”) were also obtained by the authors of [29] for TPS films with the keratin addition. The authors compare the obtained nature of the cracking of the films to the cracking of glass and indicate the brittleness of the material. In the above-mentioned studies, the analysed specimens were produced using the extrusion method, which ensured their uniformity over the entire surface.

The maximum failure stress, σ , determined from Eq. (6) for TPS/PH film specimens reached an average value of 20.20 MPa with a film thickness of $g = 0.1 \pm 0.04$ mm and was almost 2.5 times smaller than the stress for specimens with the addition of flour which was 48.5 MPa with a film thickness of 0.13 ± 0.02 mm. This allows us to determine the existence of a relationship between the influence of the type of additives (husk, flour) on the impact resistance properties of the tested

specimens. Furthermore, in conditions of relative air humidity of 50%, specimens with the addition of flour showed the much highest values of impact force, failure stress, and impactor displacement.

Previous research on mechanical tests of films often refer to tensile strength tests and the loading conditions are limited to quasi-static loads [28, 30]. In the few data published the literature describing impact tests of TPS films [22, 31] performed on commercial devices, the analysis focuses on comparing the strength of different types of films without assessing their behaviour during impact. The authors also do not attempt to assess the damage occurring during impact. The use of a patented device for soft tissues in our research also made it possible to compare the mechanical impact resistance of biodegradable TPS films. In our manuscript, we also analysed the process of deformation and tearing of the film during impact, which has not been done before for this type of materials.

Further research should focus more deeply on changes in the microstructure of the films (due to the use of different forms of psyllium), which determine their mechanical properties. Therefore, it seems necessary to use scanning image analysis on the micro and nano scale (SEM, AFM) and the use of Fourier infrared spectroscopy (FTIR) to examine differences in the structure of individual molecules and the composition of molecular mixtures. In order to provide a more complete description of the phenomena occurring during the mechanical impact of TPS films with the psyllium addition, further strength tests with recording the course of deformation using a high-speed

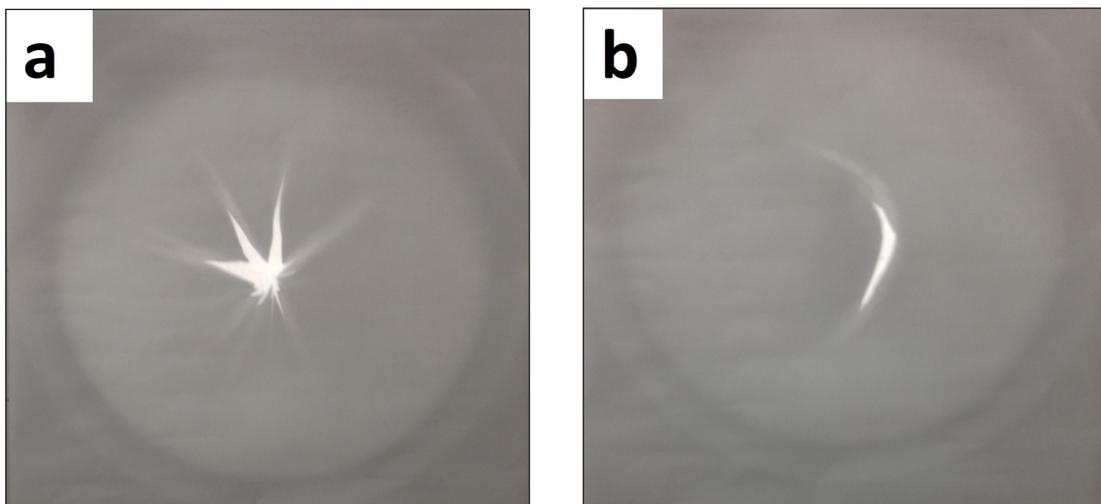


Figure 6. Characteristic cracks of the specimens obtained during the puncture impact test with the velocity $V_{imp} = 1 \text{ m} \cdot \text{s}^{-1}$, a) for TPS/PH films, b) for TPS/PF films

camera are planned. This type of test will enable the determination of the reaction force-deformation relationship, as well as the impact velocity and reaction velocities of the film and, as a result, the impact energy, absorbed energy and the coefficient of restitution. After strength tests, a thorough analysis of fractures using a scanning electron microscope will be necessary.

CONCLUSIONS

This paper studied the impact response of thermoplastic starch (TPS) films containing the addition of psyllium husks (TPS/PH) and psyllium flour (TPS/PF) using a patented soft tissues measurement testing station. The following conclusions can be drawn:

1. TPS/PF film specimens have a more uniform surface than TPS/PH specimens.
2. The obtained impactor displacement values (from the beginning of the contact with the specimen to its broke) for TPS/PF specimens were larger than for the TPS/PH film, which proves the greater flexibility of the film with the addition of flour.
3. The form of the psyllium additive used (flour or husk) influenced the values of the maximum force and failure stress obtained during the impact test.
4. Films with the addition of flour TPS/PF showed greater resistance to damage caused by dynamic loads than TPS/PH. Maximum breaking force was five times higher in the former film than in the latter.
5. Impact damage to TPS/PH films has the character of a brittle fracture, while latitudinal tearing of TPS/PF films has characteristics of destruction similar to defects observed during static tearing.
6. Star-shaped cracks in the TPS/PH films indicate that the value of latitudinal stresses was exceeded while crescent moon shaped damages found in the TPS/PF films are associated with increased meridional stresses.
7. The use of a patented device for soft tissues made it possible to compare the mechanical impact resistance of biodegradable TPS films.

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