PROPRIETĂȚI ALE COMPOZITELOR PE BAZĂ PE POLIPROPILENĂ RECICLATĂ ȘI DEȘEURI AGRICOLE LIGNOCELULOZICE
PROPERTIES OF COMPOSITES BASED ON RECYCLED POLYPROPYLENE AND LIGNOCELLULOSIC AGRICULTURAL WASTE

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In the present work, composites based on recycled polypropylene (PPr) and 30% treated and untreated lignocellulosic waste were prepared. The recycled polypropylene comes from recovered surgical masks used during the COVID-19 pandemic, from which the elastic band and the metal strip have been removed. The lignocellulosic waste used as a reinforcing agent consisted of the depleted substrate from the culture of Pleurotus ostreatus fungi, being a sawdust enriched in chitin pellets of corn cobs treated with borhot from the brewing of beer. The composites with 30% of treated/untreated sawdust were obtained in dynamical conditions by melt processing. The effects of sawdust, both treated and untreated, on tensile properties (strength and modulus of elasticity), dynamic-mechanical properties (storage/loss modulus and loss factor), thermal conductivity and thermal stability were studied.

The results showed improved thermal and mechanical properties of bio-composite materials based on recycled polypropylene from used face masks and sawdust, which can be used as construction materials.

În această lucrare au fost obținute compozite pe bază de polipropilenă reciclată (PPr) și 30% deșeuri lignocelulozice tratate și netratate. Polipropilena reciclată provine din măștile chirurgicale folosite în timpul pandemiei de COVID-19, la care au fost îndepărtate banda elastică și banda metalică. Deșeul lignocelulozic utilizat ca agent de ranforasare a constat în substratul epuizat de la cultura ciupercilor Pleurotus ostreatus, fiind un rumeguș îmbogățit în chitină din pelete de știuleți de porumb tratați cu borhot din fabricarea berii. Compozite cu 30% rumeguș tratat/netratat s-au obținut în condiții dinamice, prin prelucrare în topitură. S-a studiat efectul rumegușului, atât tratat, cât și netratat, asupra proprietăților la tracțiune (rezistență și modul de elasticitate), proprietăților dinamic-mecanice (modul de înmagazinare/pierdere și factor de pierdere), conductivității termice și stabilității termice.

Rezultatele obținute au arătat proprietăți termice și mecanice îmbunătățite ale materialelor biocompozite pe bază de polipropilenă reciclată din măști de față uzate și rumeguș, indicând faptul că acestea pot fi folosite ca materiale de construcție.

Keywords: *face mask, lignocellulosic agricultural waste, PP recycled, thermal insulation properties*

1.Introduction

The problems related to the amount of garbage produced in the world and the impact of waste on the environment are increasingly worrying in the last century. There is a trend to reduce the environmental impact of waste by creating smaller, lighter, more resource-efficient products, or by reusing some waste to create new products [1].

In the last 3 years, a new widely used waste that produces negative effects on the environment has appeared: the facial mask. Massively used to combat the COVID 19 pandemic, masks have become a serious source of hazardous waste. In general, masks are made of plastic material such as melt blown polypropylene nonwoven fabric, polyacrylonitrile, polycarbonate, polyurethane, polystyrene, polyester and polyethylene, all of them non-biodegradable polymers, which lead to an analysis of the impact they have on the environment and concerns about reducing it [2,3,4,5].

The most common types of masks are surgical masks and FFP2 type masks [6]. In this study, we focused on the recycling of polypropylene-based surgical masks, which are the most used in our country, both in the context of the pandemic and in the medical system.

Several studies have been carried out on the recycling of masks for potential uses in construction, in the production of pellets [7], or in food industry, for cutlery and packaging [8].

Recycled polymers show a decrease in mechanical properties, caused by thermomechanical stress during reprocessing [9], hence the need to use a reinforcing agent. In 2022, Pulikkalparambil et al. reported the preparation of

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recycled PP/sisal fibers and recycled PP/hemp fibers composites by removing the nylon cords from the PP masks and consecutively sandwiching the polypropylene parts of the facial masks with woven hemp or sisal fibers by compression molding, at 170°C. As compared to the recycled PP, the recycled PP/sisal fibers and recycled PP/hemp composites showed an increase in the tensile strength of 197% and 305%, together with an increase in the elongation at break of 574% and 161%, respectively. However, a sharp decrease in the thermal stability was recorded for the recycled PP/sisal fibers and recycled PP/ hemp fibers composites as compared to the neat, recycled PP. Due to the antimicrobial activity of lignin from both sisal and hemp fibers, the prepared composites exhibited antibacterial activity against Staphylococcus aureus, which allows their potential use in the manufacturing of packaging materials or cutlery for food industry [8]. In another work, Xiang et al. fabricated composites based on disposable mask PP and discarded loofah sponge (LS) by first removing the nose wires and ear straps from the facial masks, followed by the compression molding of the remaining PP parts at 180 °C, their shredding into smaller parts, and their melt mixing and compression molding with LS at 180 °C. At a content of 12 wt% LS in the recycled PP, a 326% improvement in the tensile strength, as well as a more than two-fold increase in the elongation at break and seven-fold increase in the notched impact strength were observed as compared to the neat PP. This indicated that LS presented both a strengthening effect and a toughness effect on the PP matrix. The TGA analysis revealed a significant decline in the thermal stability of PP following LS addition, while the DSC analysis indicated a nucleating effect of LS on the recycled PP matrix [10].

Another category of waste that has attracted interest in recent years is represented by lignocellulosic waste, both due to its origin from natural sources and abundance around the globe [11,12]. Lignocellulosic wastes have found uses in a variety of fields such as energy, biofuel production, water purification, mushrooms cultivation, soil fertilization, animal husbandry, or as reinforcing agents of polymer-based composites etc [11]. An attractive lignocellulosic waste with potential uses as reinforcing agent for polymer matrices might be the sawdust that results as a by-product from the logging and wood processing industries. Due to its low cost, low density, high specific tensile strength and stiffness, and higher flexibility as compared to the mineral fillers, sawdust may lead to the obtaining of light-weight polymer-based composites with superior mechanical properties and high filler contents [13]. A popular use of sawdust is as substrate for the growth of different edible mushrooms species [14]. Residual sawdust resulting from the mushrooms growth is often enriched in chitin, a major constituent of mushrooms' cell walls. The chitin-enriched sawdust can find further application as a reinforcing agent for different polymers, especially since chitin is a polymer with a somewhat amphiphilic character (containing both polar and non-polar moieties) [15], which is able to act as a compatibilizer between the common sawdust, which is rather hydrophilic, and the polymer matrix which is in general hydrophobic. From our research, the literature is rather poor in attempts of using sawdust as a reinforcing agent for polymers. Prabu et al. [16] prepared composites based on unsaturated orthophthalic polyester resin and epoxy resin, respectively, and sawdust. At a content of sawdust in the polyester matrix of 40%, a 14% increase in the tensile yield simultaneously with a 61% improvement in the impact strength of the polyester resin were achieved. For the epoxy-based composites, a rise in the tensile strength of 4% was observed, while the impact strength decreased by around 77% due to the poor dispersion of the sawdust in the epoxy matrix. No clear variation of the hardness with the sawdust content could be established for the polyester/sawdust and epoxy/sawdust composites, although a tendency of decreasing hardness with increasing sawdust content was noticed for the polyester/sawdust composites [16].

In this work we propose a new method for the valorization of polypropylene from facial masks waste and a sawdust from the culture of *Pleurotus ostreatus*, enriched in chitin from pellets of corn cobs treated with borhot from the brewing of beer by the preparation of polypropylene/sawdust composites. To the best of our knowledge, this is the first time when sawdust residues coming from the *Pleurotus ostreatus* growth are used as reinforcing agents for polypropylene. The effect of the sawdust on the mechanical and thermal properties of the recycled polypropylene was evaluated by tensile tests, thermogravimetic analysis (TGA), thermal conductivity and the dynamic-mechanical behavior of the composites was investigated by dynamic mechanical analysis (DMA).

2.Experimental

2.1.Materials

For the polymer matrix, 3-layer face masks were used (100% polypropylene composition), after disinfection with alcohol, removal of elastics and metal tape and drying at 80°C for 2 hours.

The reinforcing agent consisted of lignocellulosic agricultural waste from the culture of *Pleurotus ostreatus* (S), being a sawdust enriched in chitin from pellets of corn cobs treated with borhot from the brewing of beer.

Polypropylene functionalized with maleic anhydride (PP-MA) was used as a compatibilizing agent and poly (propylene glycol adipate) (PAPG) as a sawdust surface treatment agent (ST).

2.2. Obtaining of composite materials

PP based composites with 30% sawdust were prepared using both whole masks (after removing the elastic and the metal band) and granules obtained by thermomechanical recycling of mask waste with the help of a double screw extruder, equisens, type Leistritz 30.34, at a main screw speed of 100 rpm and an extrusion head temperature of 180 ± 5 °C. The extruded filaments passed on a conveyor belt, air-cooled and then granulated in a granulator mounted in-line with the extruder. All compositions contain 5% compatibilizing agent MA-PP. The masks were disinfected and dried at 80°C for 2 hours in an oven with air circulation. The sawdust was dried under vacuum, for 2h at 120°C, ground in a knife mill and sieved (dimensions < 0.5 mm). The sawdust was treated with 6% PAPG under dynamic conditions, at 80 °C, for 60 min.

The composites with 30% untreated/treated sawdust were obtained either by mixing the recycled PP granules with the compatibilizer and the treated sawdust, in a rotary gravimetric mixer, followed by homogenization in the melt on Leistritz extruder, at a main screw speed of 80 rpm and an extrusion head temperature of 170 ± 5 °C, resulting in composite granules or by distributing the sawdust between the filter layers of the disinfected and dried mask and obtaining some rolls that were then homogenized in the melt on an AMUT single screw extruder, at 40 rpm and 180°C, resulting in composite melt.

Standard specimens were obtained by injection of composite granules, on the Injection Molding Machine ENGEL VC 60/28, at the temperature of 190 ± 5 °C and the mold temperature of 70-90 °C and plates of 150x150x4 mm were obtained by pressing the composite melt in the Collin press P 200 E, at a temperature of 190 ± 5 °C and a pressure of 160 atm. The samples were denoted with PPr, PPr-S and PPr-ST for recycled PP granules and composites with 30% untreated/treated sawdust, respectively.

2.3 Methods used

The effects of filler content on tensile properties (strength and modulus of elasticity), dynamic-mechanical properties (storage/loss modulus and loss factor), thermal conductivity and thermal stability of the composites were studied. The tensile properties of the composites were determined according to ISO 527, at 23°C and 50% relative humidity, with 50 mm/min for tensile strength and 2 mm/min for modulus of elasticity, using 5 specimens for each test, from each sample. Thermal conductivity was determined using the λ-

The thermal stability of PP recovered from masks and composites based on recycled PP was characterized by thermogravimetric analysis (TGA) using TA Q5000 equipment (TA Instruments Inc., New Castle, DE, USA). Measurements were performed from room temperature to 700ºC, with a heating rate of 10º/min, in a nitrogen atmosphere. The viscoelastic properties, such as storage modulus (E′), loss modulus (E″), loss factor (tan δ) of PPr and composites as a function of temperature were determined using a DMA Q800 equipment (TA Instruments Inc., New Castle, DE, USA), which operated with dual cantilever bending. Data processing was performed with Universal Analysis 2000 software (Version 4.5). Rectangular specimens having a width of aprox.10 mm, a thickness of 4 mm, and a length of 60 mm (were cut from injection-molded plates) were examined according to the testing program conducted, DMA-Multifrequency- Strain, at a single frequency of 1 Hz, from -40 to 150 °C, with a constant heating scan of 3 °C min^{-1}, and an oscillation amplitude of 15 μm.

3.Results and discussions

3.1 Influence of the filler on tensile mechanical properties

The relationship between the obtained tensile properties (tensile strength and modulus of elasticity) and the amount of filler added to the PP matrix is shown in Figure 1, both for the injection-moulded specimens and for those obtained by compression molding.

The results obtained for the injected samples show an improvement in stiffness of 50% for the untreated sawdust composite and of 80% for the treated sawdust composite, the Young Modulus increasing from 1339.93 MPa to 1999.89 MPa and 2314.9 MPa, respectively. This behavior can be explained by the existence of interactions between the components, which allow an efficient transfer of mechanical stress from the polymer matrix to the reinforcing agent. Thus, in the case of PPr-S, PP-MA acts as a bridge between the PP and the filler due to the interactions between the maleic anhydride and the -OH groups in the chitin present in the sawdust, on the one hand, and on the other hand, due to the interactions between the polypropylene end of PP-MA with the polymer matrix. Similar behaviors were found by other researchers [17,18]. In the case of PPr-ST, the adhesion at the polymer matrix-filler interface is stronger due to the additional interactions between PAPG and each of the composite components. Thus, PAPG can interact through -OH groups with chitin from sawdust, through ester groups with maleic anhydride from PP-MA and through propylene groups with the polymer matrix. These strong interactions are reflected in a uniform

Meter EP500 thermal conductivity measurement system.

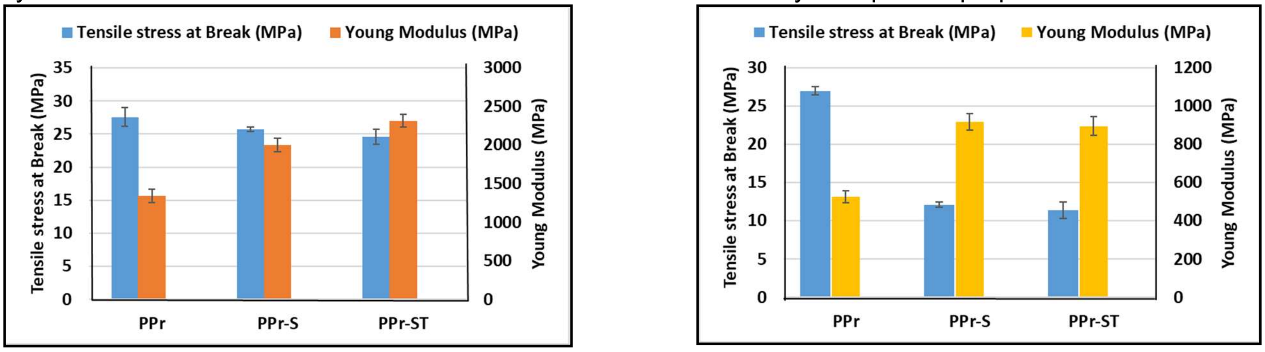


Fig. 1 - Mechanical properties of PP based composites compared with PPr (results obtained on injected samples obtained by injection moulding (left) and on samples stamped from pressed plates (right))/ Proprietățile mecanice ale compozitelor pe bază de PP comparativ cu PPr (rezultate obţinute pe epruvete injectate (stânga) şi pe epruvete stanţate din plăci presate (dreapta))

dispersion of the filler in the polymer matrix and ultimately in improved properties. The tensile strength of the composites shows insignificant decreases, of up to 11% in the case of PPr-S and up to 7% in the case of PPr-ST, which shows that PAPG acted as a compatibilizer, improving the treated sawdust composite properties. Yin et al [19] showed that a slight decrease in tensile strength is possible if the filler content exceeds the loading capacity of the polymer matrix.

The same behavior is observed in the case of pressed plates. The Young's modulus increases in the case of composites from 526.6 MPa (PPr) to 918.63 MPa (PPr-S) and 894.4 MPa (PPr-ST), which shows an increase in the stiffness of the composites by 70% compared to PPr. However, the tensile strength decreases by 57%.

Considering the mechanical properties obtained for the injected samples and taking into account others that highlight the acoustic performances of the recycled masks, these composites could be used both in construction and in the automotive or aerospace industry [20].

3.2 *Influence of the filler on thermal conductivity*

It is observed that the addition of sawdust leads to the improvement of the thermal insulating properties by 40% compared to polypropylene obtained from face masks (Fig. 2). This decrease in thermal conductivity is justified by the use of sawdust, a material with recognized insulating properties and its uniform dispersion in the polymer matrix. Similar results were obtained by other researchers in the case of construction materials based on sawdust [21,22].

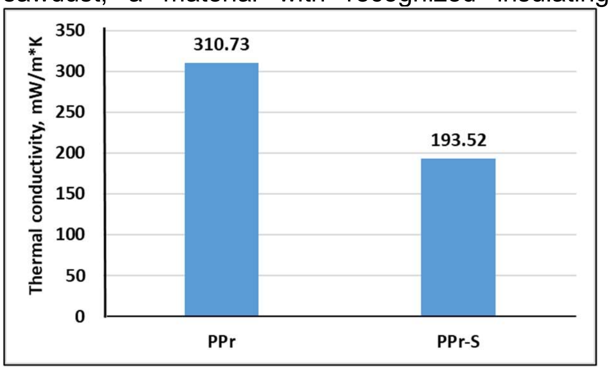
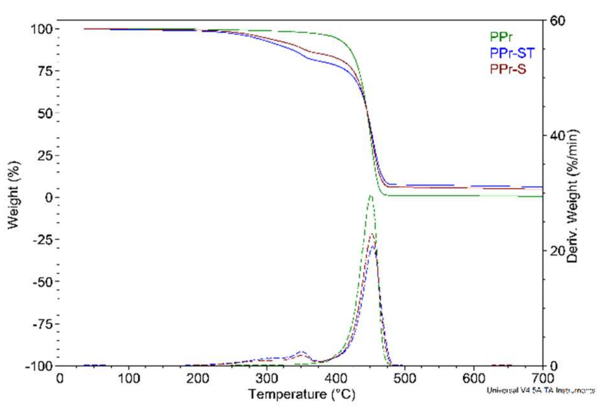


Fig. 2 - Thermal conductivity variation of PPr and PP based composite (obtained on pressed plates)/ *Variația*

3.3 *Influence of the filler on* thermal stability (TGA)

Thermal degradation studies using thermogravimetric analysis (TGA) are important to understand the thermal stability of composites used in construction. It is important to study the influence of fillers on the thermal degradation of the polymer matrix. The thermal behavior of the composites is shown in Figure 3 by the TGA curves.

It is observed that for recycled polypropylene the degradation occurs in a single stage, with the maximum speed of decomposition at a temperature of 451.2 ºC. For the composites with 30% sawdust, treated and untreated, the decomposition occurs in 2 main stages, with the maximum rate at 351.7 ºC and 351.1 ºC, respectively, on the first stage and 452.7 ºC and 453.9 ºC, respectively, on the second stage of decomposition.



conductivității termice a PP şi a compozitului pe bază de PP (obţinută pe plăci presate)

Fig. 3 - Thermal decomposition curves of PPr and composites based on PPr / *Curbele de decompunere termică ale PPr şi ale compozitelor pe bază de PPr*.

Table 1

Decomposition temperatures and weight loss of composites compared with PPr/ Temperaturile de descompunere şi pierderea de masă ale compozitelor comparativ cu PPr

Sample	RT-230°C	Decomposition Stage 1 230-380 °C			Decomposition Stage 2 380-500 °C			Residue
	Wt. loss	Onset	T_{dmax1}	Wt.loss	Onset	T_{dmax2}	Wt.loss	700°C
	%	°C	°C	%	°C	°C	%	%
PPr	0.53	-	-	-	330.4	451.2	97.26	0.66
PPr-S	1.31	326.7	351.7	6.08	381.1	452.7	79.31	4.77
PPr-ST	1.97	323.9	351.1	8.63	379.6	453.9	73.2	6.18

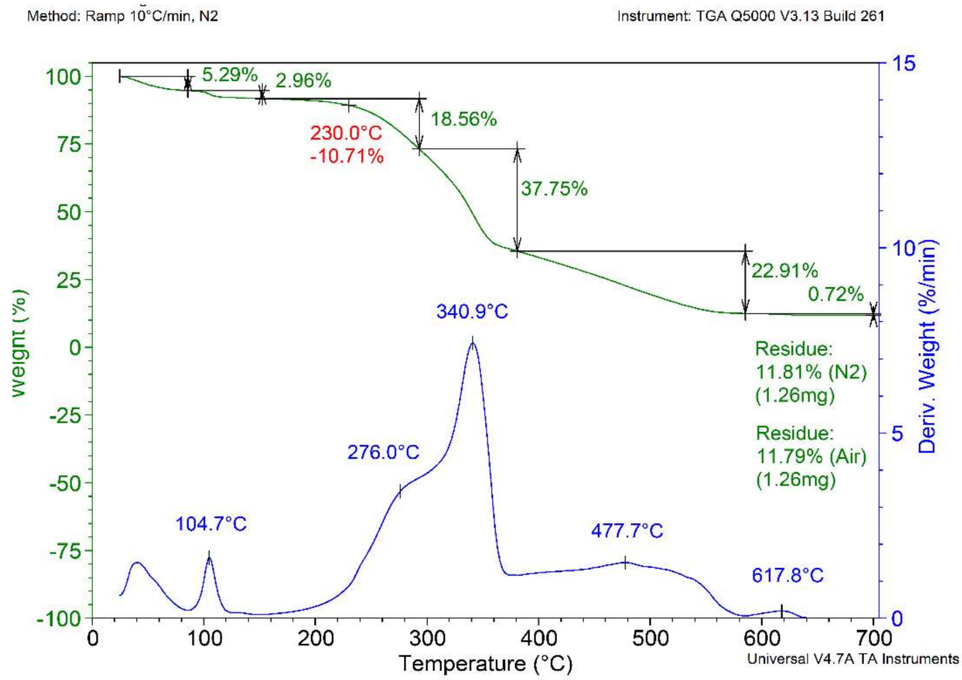


Fig. 4 - Thermal stability of sawdust/ *Stabilitatea termica a rumegușului*

The thermal behavior of composites are due to the way the sawdust degrades, as shown in Figure 4. It degrades at lower temperatures than the polymer, having two degradation maxima at 276 ºC and 340.9 ºC in the temperature range of 150-380 °C. The first peak, with the maximum rate of decomposition of about 105 °C, is due to the gradual evaporation of moisture, and the following peaks (in the temperature range of 230-630 °C) correspond to the release of volatiles from the main components (cellulose, hemicellulose and lignin) [23]. However, the thermal stability of sawdust is improved in the composite.

Table 1 summarizes the degradation temperatures and weight loss for all composites.

leads to the formation of considerable amounts of char residue during its thermal degradation [23]. There was no significant loss of mass at the processing temperatures of the composites, although a greater weight loss was observed for the composites with untreated sawdust. According to T_{onset} values, sawdust improves the thermal stability of recycled polypropylene (on the second stage of decomposition T_{onset} increases by approx. 50 °C).

It is also observed that in the case of the composite with treated sawdust, the stability is better, which shows that PAPG contributed to the increase of the interaction at the interface between polypropylene and sawdust.

The higher amount of residue obtained for the PPr-S and PPr-ST composites is due to the presence of sawdust that contain in its composition lignin, a polymer with a highly crosslinked nature and numerous aromatic rings its structure which

3.4 Influence of the filler on dynamic mechanical (DMA) properties

Through the DMA analysis, the viscoelastic behavior of the composites compared to PPr was highlighted. The storage modulus, loss modulus

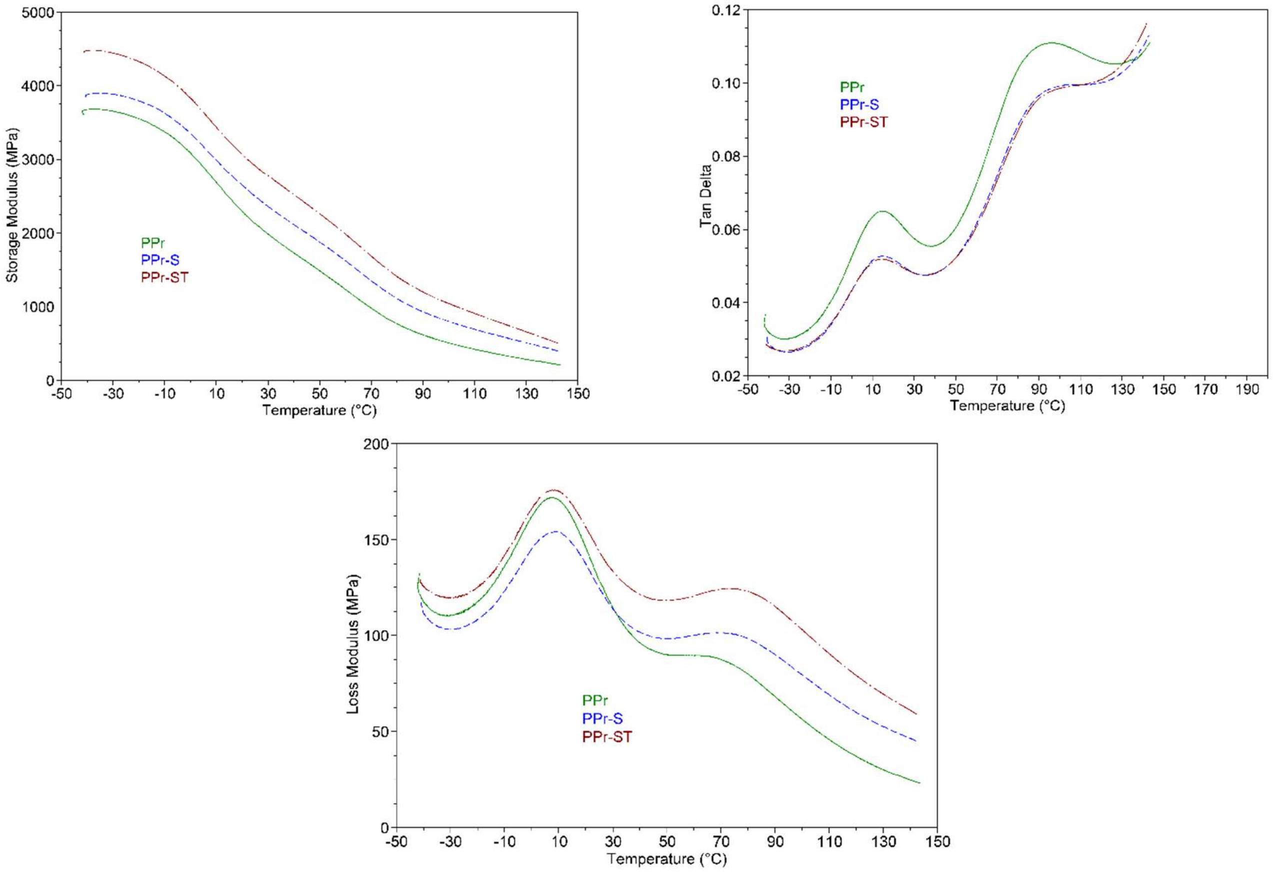


Fig. 5 - DMA results for PPr and composites based on PPr and 30% sawdust, treated and untreated / *Rezultate DMA pentru PPr și compozitele pe bază de PPr și 30% rumeguș, tratat și netratat*

and the tan δ variations with temperature are shown in Figure 5 while the main characteristics are shown in the Table 2.

Regarding the storage modulus (E'), a similar behavior can be observed for all samples, namely a decrease in its values with increasing temperature. This may be assigned to the gradual softening and segmental relaxation phenomena occurring in the PPr matrix with the rise in temperature. As the temperature increases, the segmental mobility of the polymer chains is enhanced and thermal expansion events occur in the PPr matrix leading to a decrease in the intensity of the interactions at interface between PPr and the sawdust filler, and hence the decrease in E' [24]. Nevertheless, an increase in the storage modulus can be observed for the composites with sawdust as compared to the neat PPr, which suggests an improvement in the stiffness of the composites. The storage modulus increased by 26% for the PP-S compared to PPr and by 52% for the PP-ST compared to PPr. This may indicate the existence of favorable interactions at the interface between the polymer matrix and the reinforcing agent interface that allows for an efficient stress transfer from the PPr matrix to the sawdust reinforcing agent and so, to an increased rigidity for the composites as compared to the neat PPr [24]. The maximum storage modulus and therefore stiffness was recorded for the PPr-ST sample, which shows that PAPG acted as a compatibilizing agent at the PPr-sawdust interface, which becomes more rigid. The Tan δ and loss modulus curves show 2 well-defined peaks, at approximately 15ºC and 96-100ºC,

Table 2

DMA results/ *Rezultate DMA*

Sample	Storage modulus/Temp, E' (MPa)	Loss modulus, E"	Loss factor

	-40 ºC	0 ºC	50 ºC	E'' peak 1, MPa	Temp, ºC	E'' peak 2, MPa	Temp, ºC	Tan δ (peak max 1)	Temp, ºC	Tan δ (peak max 2)	Temp, ºC
PPr	3672	3088	1490	171.9	7.8	89.51	57.91	0.065	14.84	0.1109	95.86
PPr-S	3866	3351	1881	153.9	7.8	101.3	68.86	0.053	15.09	0.09851	96.11
PPr-ST	4466	3834	2264	175.7	8	124.4	72.75	0.052	14.6	0.09858	100.24

respectively 8ºC and 58-73ºC. The first peak corresponds to the glass transition temperature of PP, and the second can be associated with the α-transitions of polypropylene. The glass transition temperature is not changed by the addition of sawdust, instead the intensity of the transition is lower as a result of the decrease in the mobility of the polymer chains.

A decrease in the height of the tan δ was observed for the PPr-S and PPr-ST composites as compared to the neat PPr. This indicates a decline in the damping capacity of the composites as compared to the PPr sample which can be attributed to the addition of the sawdust reinforcing agent which limits to a certain the segmental movement of the PPr chains due to the strong interactions established at the interface between the sawdust and PPr matrix [25].

4.Conclusions

This work succeeds in exploiting two waste materials with an impact on the environment, offering solutions for the valorization of surgical masks and lignocellulosic waste at the end of their life cycle.

The obtained composites have a stiffness improved by 50-80% compared to PPr, thermal insulation properties improved by 40% and a thermal stability similar to recycled polypropylene, thus being able to be used in constructions as thermal insulation materials. Better results were obtained on the injected samples, which is why these samples will find applications in several fields.

The use of a dispersing agent is useful, the PAPG bringing improvements to the composite with treated sawdust.

The obtained materials, with improved properties, easy to process into finished products by injection or pressing, recyclable and easily biointegratable at the end of the life cycle, represent a solution for valorisation of used facial masks and lignocellulosic wastes, with applications in construction.

Acknowledgements
This work was funded by project POC-A1-A1.2.3-G-2015-P_40_352SECVENT, Sequential processes to close bioeconom y side stream and innovative bioproducts resulted from these, c ontract 81/2016, SMIS 105684, funded by cohesion funds of the European Union, subsidiary projects 1501/2020

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