

# Wood Flour – Recycled Polyol Based Polyurethane Lightweight Composites

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**ABSTRACT:** This work is focused on the production and characterization of lightweight polyurethane (PU) composites reinforced with pine wood flour (WF), which can have applications in car interior panels, construction, and acoustic insulation. A crosslinked PU formulated from a recycled polyol was used as matrix. The mechanical performance of the reinforced composites was studied through tensile, three point bending, and dynamic mechanical tests. The strength, modulus, and storage modulus increased with filler concentration. The addition of Al(OH)<sub>3</sub> did not reduce the flammability of the composites with low WF concentrations; however, some improvements were found in the case of composites prepared with 20 wt% WF.

**KEY WORDS:** polymer–matrix composites, mechanical properties, thermomechanical response, polyurethane, wood flour.

## INTRODUCTION

THE CONSTRUCTION INDUSTRY requires large volumes of structural materials for the construction of large and small buildings, such as housing. In these projects the need for flame resistant, lightweight, and high strength materials is common. Applications of such materials range from wall panels, door skins, and door cores to roof tiles and ceiling boards. A vast variety of conventional materials exist, each with unique applications and properties. These include wood, wood composites, and various gypsum products. As it is necessary to strive towards a cleaner environment, it becomes necessary to re-use materials, as well as to convert waste materials into more useful products [1,2].

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Polyurethane foam is one of the most widely used cushion materials in automobiles, furniture, and so on. However, a considerable amount of scrapped polyurethane foam is disposed off by landfill or by incineration, because a recycle process capable of treating all scrapped polyurethane with sufficient efficiency has not been established yet. In the mechanical recycling process, polyurethane foam is smashed into a fine powder and used as recycled urethane filler [3,4]. However, the amount of polyurethane foam treated by this process has not increased significantly up to date. On the other hand, several chemical recycling techniques, such as hydrolysis [5], glycolysis [6,7], and aminolysis [8], including the decomposition process with alkanolamines have been reported. Recently [9], a flexible polyurethane foam was decomposed into a polyol mixture using diethanolamine as a decomposing agent. The resulting decomposed product was successfully tested as an alternative to virgin polyol in reclaiming polyurethane.

The properties of foams (high capacity of energy absorption, good thermal properties, and specific strength), which depend on the architecture of the cellular structure and the intrinsic properties of the polymer [10], have also been tailored by incorporating reinforcing fibers [11] or particles [12–15]. The inclusion of reinforcements into the polymeric matrix can lead to better adsorption of the heat released during the polymerization reaction, and increase of the matrix rigidity, retaining an acceptable density. The reinforced polymer architecture obviously depends on the filler dispersion, and the wall width compared to the particle size. Moreover, the efficiency of the reinforcement depends on the interaction between the filler and the polymer matrix [4,15].

This work was focused on the production and characterization of lightweight polyurethane composites reinforced with pine wood flour (WF) that can be used in car interior panels construction and acoustic insulation. A crosslinked polyurethane (PU), formulated from a recycled polyol was used as a matrix. The source of isocyanate groups utilized was a polymeric diisocyanate (MDI), for easy handling of the reactants and reinforcements.

## EXPERIMENTAL PROCEDURES

### Materials

A recycled polyol (Polyol C), provided by PEMÜ Co. (Hungary) was used for PU production. Its OH value was determined by analytical techniques [16] and corresponds to 270 mg KOH/g. The isocyanate crosslinker was a 4,4'-diphenylmethane diisocyanate (MDI) prepolymer (Luparant<sup>®</sup> M20 S from BASF Elastogran-Kemipur Co, Hungary) with NCO content = 31.2 g/100 g.

Wood flour from Pine sp., 100  $\mu\text{m}$  average size, was used as reinforcement. It was dried at 110°C overnight before use. The hydroxyl value of the wood flour was determined using a back titration technique. A measured weight of wood flour was mixed with excess of MDI. After the reaction was completed, the free NCO groups were determined according to a technique described elsewhere [17], and from this the concentration of available OH groups in WF was determined. The resulting value was 233.3 mg KOH/g.

Lightweight composites containing 0, 5, 15, and 20 wt% of wood flour were prepared. One series (A) of composites was formulated with an Index (NCO equivalents/OH equivalents provided by the polyol) = 1.00 for all the filler concentrations and for the other set (B), the Index was varied linearly from 1.00 (unreinforced PU) up to 1.25

(20 wt% reinforced PU). The recycled polyol was used without dehydration (approximate moisture content: 5 wt% from thermogravimetric analysis, TGA), thus the retained humidity had also a minor contribution as foaming agent. In some samples, a commercial solvent (Pevdipox RD-12, Kemikál Co, Hungary) was used as a viscosity modifier; thus, it was purposely added to the polyurethane formulation (10 wt% with respect to the weight of the recycled polyol) to decrease the initial, unreacted system viscosity. The Fourier Transform Infrared (FTIR) spectrum of the solvent (not shown) indicates that it is a mixture of aliphatic ethers, showing a very close correlation (according to the equipment library, Genesis II FTIR spectrometer) with the di-*n*-hexyl ether. In order to improve the composite fire behavior, in some samples a flame retardant additive, Al(OH)<sub>3</sub> powder from Ajka Timföld Kft (Ajka Alumina Ltd, Hungary), was added at 25 wt% with respect to the weight of the formulation free of the fire retardant.

### Qualitative Physical Characterization of the Recycled Polyol

The polyol was studied by transmission FTIR spectroscopy, using windows of NaCl. All spectra were recorded at 2 cm<sup>-1</sup> resolution with the use of a Genesis II Fourier transform infrared spectrometer. Reported results are the average of 16 scans.

Size exclusion chromatography (SEC) was used to identify the polyol molar mass distribution. A known sample weight (~15 mg) was dissolved in 5 mL of tetrahydrofuran (THF), and 10 μL of the resulting solution was injected into the size exclusion chromatograph (SEC, Knauer K-501, RI detector Knauer K-2301, and a set of Phenomenex Phenogel 5 μ-columns: 50A, 100A, and M2). THF was used as a carrier at 1 cm<sup>3</sup>/min and the calibration was performed with polystyrene standards.

### Dynamic Mechanical Tests

A Perkin-Elmer dynamic mechanical analyzer (DMA 7) was used in these experiments to obtain the storage modulus ( $E'$ ), and loss tangent ( $\tan \delta$ ) of the samples. The tests were carried out using the temperature scan mode, the three-point bending fixture with a specimen platform of 15 mm length and dynamic and static stresses of  $5 \times 10^5$  and  $8 \times 10^5$  Pa, respectively. The frequency of the forced oscillations was fixed in 1 Hz. The specimens were cut to  $20 \times 4 \times 3$  mm<sup>3</sup>, and the linear dimensions were measured up to 0.01 mm.

### Tensile Properties

The  $150 \times 10 \times 4$  mm sized specimens were tested by ZWICK 1464 instrument at 23.2°C. The test parameters were 20 mm/min rate, and 115 mm clamping length, according to ISO 527 standard. Tensile modulus ( $E$ ), tensile strength, and stress at break ( $\sigma_{\text{break}}$ ) were calculated from the stress–strain curves. At least five specimens for each material were tested.

### Flexural Properties

The mechanical performance of the reinforced PU was studied using three point bending fixture in a Zwick Universal Tester machine. A span of 64 mm (transversal area of  $10 \times 4$  mm<sup>2</sup>) and a crosshead speed of 2 mm/min were selected, according to the EN ISO 178 standard. Flexural modulus ( $E_b$ ) and ultimate bending stress ( $\sigma_u$ ) were calculated from the stress–strain curves. At least five specimens for each material were tested.

### Impact Properties

The Izod impact strength ( $a_1$ ) was determined in a ZWICK equipment at standard laboratory temperature (25°C), using a 2.75 J hammer with 124.4° release angle (striking nose speed 3.46 m/s at the moment of impact). The size of the unnotched test specimens was  $80 \times 10 \times 4 \text{ mm}^3$  according to EN ISO 180. At least five specimens for each material were impacted.

### Scanning Electron Microscopy

The fracture surfaces generated during flexural tests were analyzed by scanning electron microscopy (SEM) using a Philips model SEM 505 microscope. The samples were sputtered with gold prior to being observed.

### Water Sorption

Samples were stored in desiccators above  $\text{P}_2\text{O}_5$  for 3 days before determining their dry (initial) weight. Then, the specimens were stored in distilled water at room temperature. At the beginning of the test, the weight of specimens was measured (the surface was wiped dry using a paper towel) every few hours and later on, every 2–3 days. Tests were conducted for 1392 hours (58 days).

### Flammability Tests

The minimum oxygen concentration to sustain burning (Limiting Oxygen Index, LOI) was measured on specimens ( $10 \times 4 \text{ mm}^2$  cross section, 70–150 mm length) using equipment developed by MÜKI (Plastic Research Institute, Budapest, Hungary), according to ISO 4589-2. The LOI index corresponds to the oxygen concentration necessary to sustain burning for at least 180 s or for reaching 50 mm length. Specimens were held vertically and heated on a gas burner (ignition flame length = 12–20 mm) placed near its bottom.

## RESULTS AND DISCUSSION

### Polyol Characterization

Figure 1 shows the FTIR spectrum of the recycled polyol. The broad band of  $-\text{OH}$  stretching vibration at  $\sim 3400 \text{ cm}^{-1}$  is associated with free  $\text{H}_2\text{O}$ , and  $\text{OH}$  groups within the polyol polymeric structure. The peaks at 2970, 2923, and  $2866 \text{ cm}^{-1}$  corresponds to the  $\text{CH}_2$  asymmetric stretching of the polyol chains.

The band with a maximum at  $\sim 1732 \text{ cm}^{-1}$  can be either due to the stretching vibration of  $\text{C}=\text{O}$  in ester linkages and/or the vibration of the urethane carbonyl. The peaks attributed to hydrogen-bonded urea carbonyls ( $1640 \text{ cm}^{-1}$ ) and free urea carbonyl ( $1695 \text{ cm}^{-1}$ ) are barely noticeable [18]. The very strong peak at  $1100 \text{ cm}^{-1}$  belongs to the stretching of  $\text{CH}_2-\text{O}-\text{CH}_2$  linkages in aliphatic ester structures.

Figure 2 shows the SEC chromatograph of the recycled polyol. Several low molecular mass species, with elution times ranging from 18 to 28 min are identified, while the major contributing species has a higher molecular mass (elution time less than 18 min).

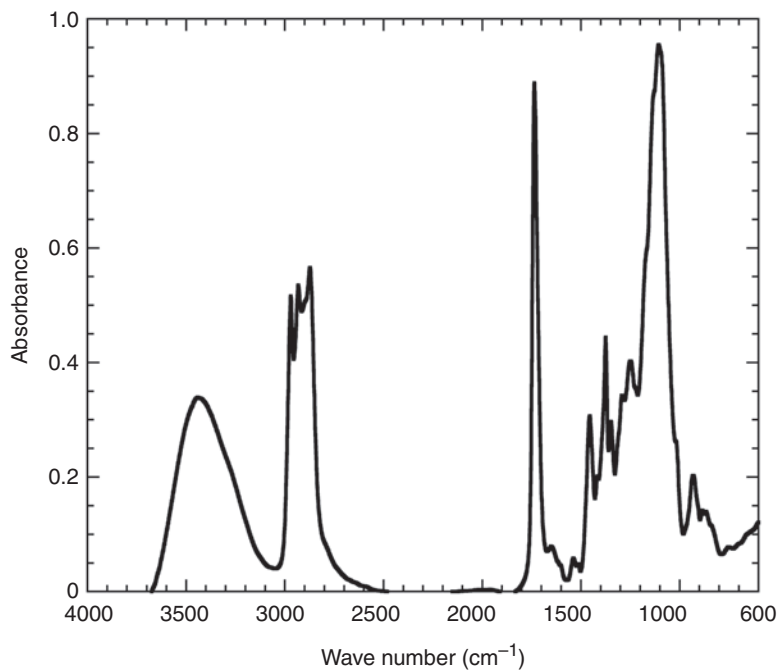


Figure 1. Recycled polyol FTIR spectrum.

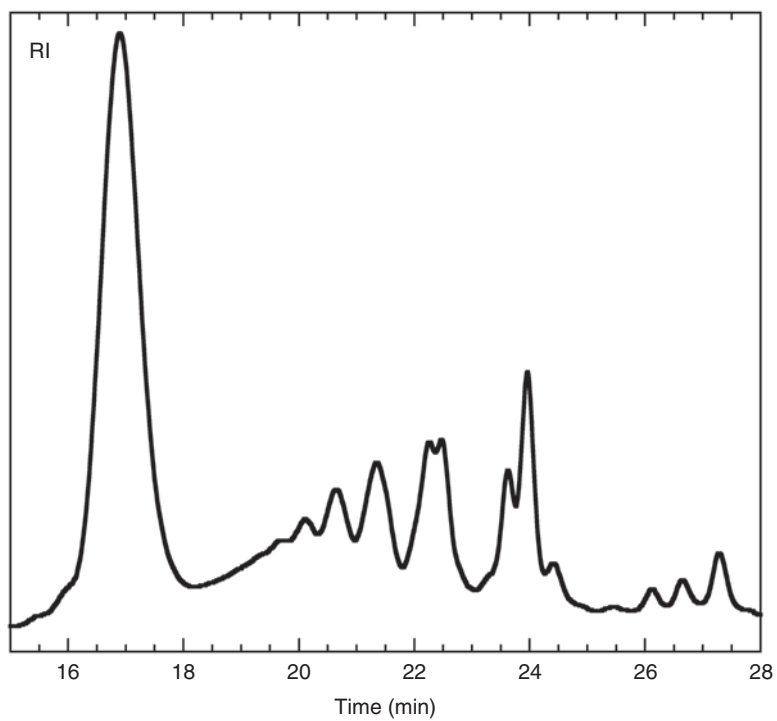


Figure 2. Recycled polyol SEC chromatograph.

In addition, the recycled polyol presents a high initial viscosity. Thus, the previous characterization indicates that the recycled polyol is a complex mixture of high molecular weight polyols, which was used “as received” in the preparation of the lightweight polyurethanes and derived WF composites.

## Composite Characterization

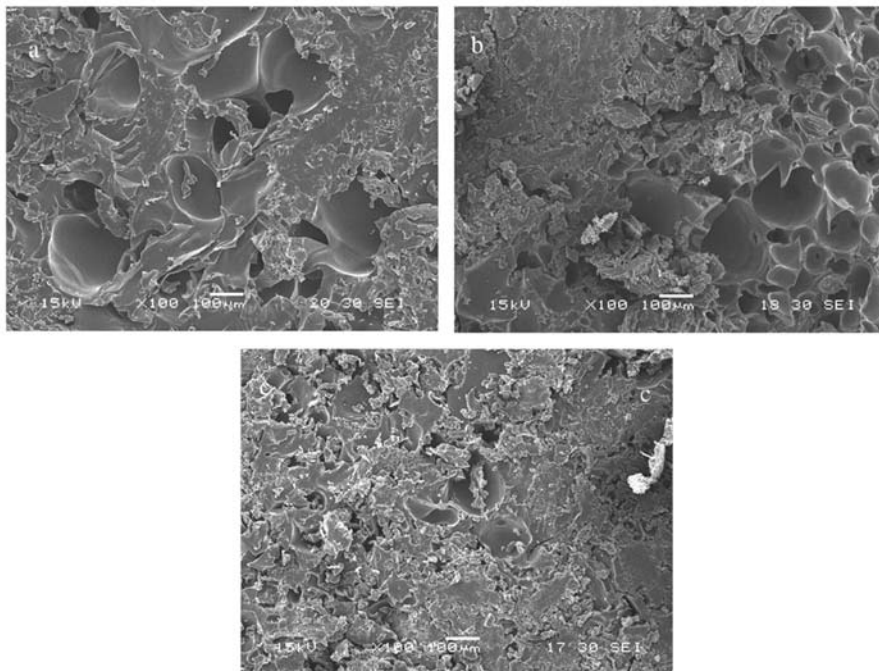
### *MORPHOLOGY OF THE COMPOSITES*

The morphology of the lightweight composites was analyzed by scanning electron microscopy of the surfaces of the samples broken in the flexural tests. The micrographs (Figure 3) show the presence of irregularly foamed regions, which are responsible for the light weight of the composites and were formed during the curing reaction by evaporation of humidity and low molecular weight components that could be present in the recycled polyol.

Moreover, the wood flour incorporation severely affects the morphology of the composite and that of the foamed regions. In this case, the addition of filler induces the increase of the proportion of open/broken cells, since the reinforcement is large enough to interact with more than one cell.

### *MECHANICAL AND FRACTURE PROPERTIES*

The effect of the filler content on the mechanical properties of the materials was investigated. Figure 4 shows the storage modulus (Figure 4a) and  $\tan \delta$  vs. temperature (Figure 4b) for the wood flour reinforced PU, B series. As expected, the storage modulus increases with



**Figure 3.** SEM micrographs of selected lightweight polyurethanes: (a) neat polyurethane, (b) 20% WF – A series, (c) 20% WF – B series.

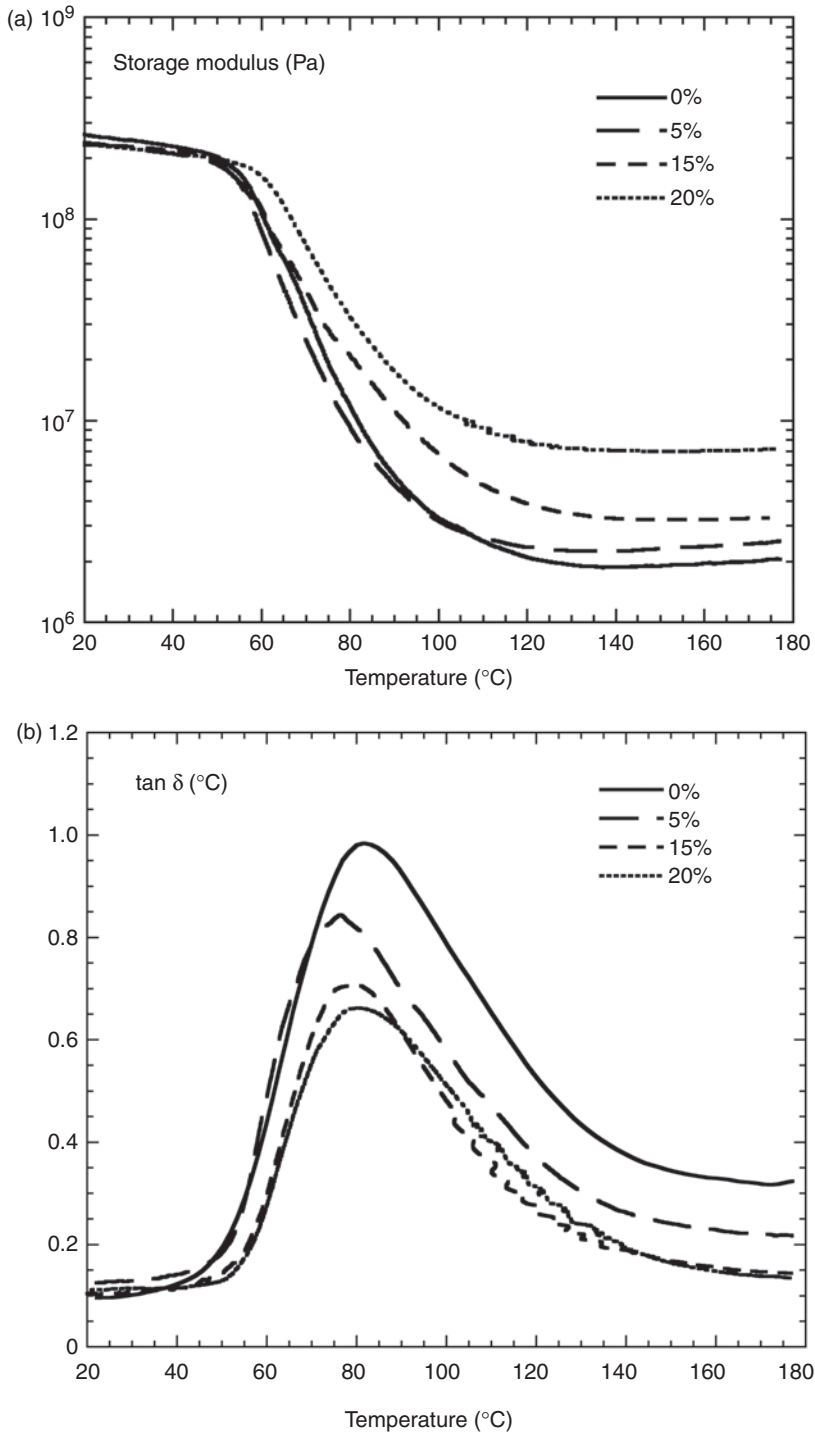


Figure 4. (a) Storage modulus and (b)  $\tan \delta$  vs. temperature for wood flour reinforced foams (B series).



**Table 1. Dynamic mechanical properties of recycled polyol composites.**

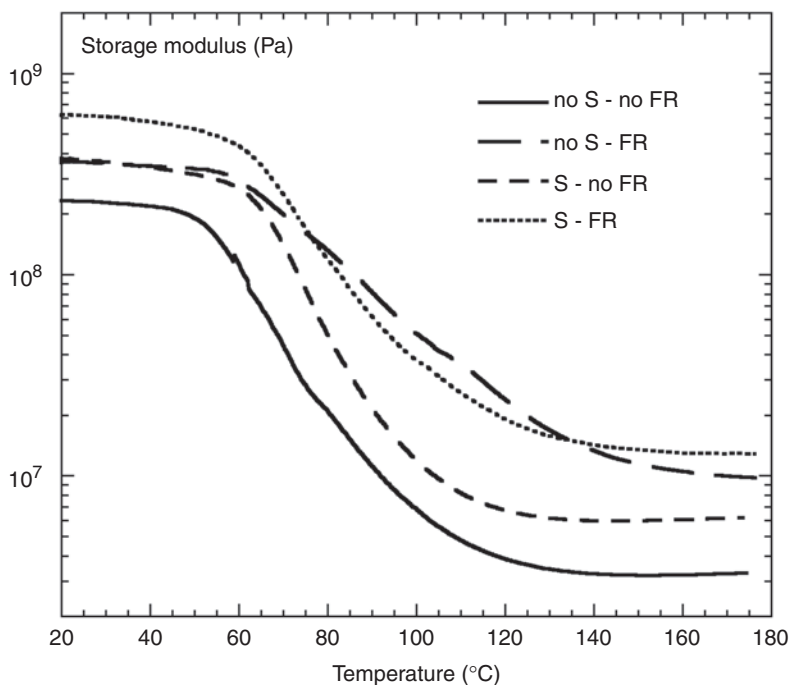
Series	% WF	Glass transition temperature (°C)	Storage modulus (MPa), 30°C	Storage modulus (MPa), 150°C
A	0	81.6	246.6	1.9
	5	70.2	134.4	1.6
	15	68.3	215.0	3.5
	20	67.2	290.6	6.0
B	5	76.0	224.5	2.3
	15	78.8	228.4	3.2
	20	79.6	223.7	7.0

filler concentration and the changes are more noticeable in the rubbery region, where the contribution of the matrix is small. The glass transition temperature (related to the position of the maximum in the  $\tan \delta$  peak) of the matrix in the composites is slightly lower than that of the unfilled polymer, possibly because the filler still contains some moisture that reacts with the isocyanate, thus reducing the formation of urethane linkages. Additionally, the corresponding storage modulus and glass transition temperature for the A series composites (Table 1) are lower than those of the B series, which suggests that incomplete crosslinking was achieved with the lower stoichiometric index. Since the index was defined as the ratio of NCO groups to OH groups provided by the polyol, the OH groups incorporated through the vegetable reinforcement were not considered. Thus, insufficient NCO groups were incorporated to these samples to complete crosslinking and this deficiency was even more aggravated by the presence of moisture.

The effect of solvent and fire retardant additions on the storage modulus of the 15 wt% WF-B series composites is shown in Figure 5. It is noticed that the storage modulus of the samples prepared with solvent and/or with fire retardant addition is higher than that of the simple formulation (polyol + MDI + wood flour). Regarding the solvent, it is added to decrease the unreacted system's viscosity, allowing a more efficient mixing. Thus, functionalities that could not react due to diffusion problems are now able to increase the crosslinking density of the sample with the consequent slight increase of the storage modulus, as seen in the rubbery region. Although indirectly, crosslinking also increased the cohesion density of a material and thus, the modulus in the glassy region is also increased. Concerning the fire retardant addition, it is incorporated to the sample as  $\text{Al}_2\text{O}_3$  (a ceramic particulate material with a very high modulus), which acts as an additional reinforcement in the system. This hypothesis is also supported by the large increase observed in the rubbery region, where the effect of a rigid filler with high modulus is more noticeable.

The tensile properties of the PU-WF composites are shown in Table 2. As expected, the general trend shows that the modulus, the maximum stress, and the strength, as well as the density of the composites increase as the wood flour content increases. However, at 5 wt% WF a slight decrease in those properties is noticed if compared to those of the neat matrix. The reason for this could be that at low filler contents, the particulate fillers added can act as defects, which promote crack propagation, hence reducing the mechanical performance of the composites [15,19,20]. Moreover, the materials corresponding to series B exhibit higher values than the composites of series A. These results are a consequence of the co-reaction between wood-flour and the PU matrix, which leads to the improved load transfer between filler and matrix, thus improving the mechanical properties of the composites.





**Figure 5.** Storage modulus vs. temperature curves for the 15 wt% wood flour reinforced foams (B series). S: solvent, FR: fire retardant.

**Table 2. Tensile properties and density of recycled polyol composites.**

Series	% WF	Solvent	E (MPa)	Strength (MPa)	$\sigma_{\text{break}}$ (MPa)	Density ( $\text{kg} \cdot \text{m}^{-3}$ )
A	0	No	$246 \pm 11$	$5.2 \pm 0.2$	$5.0 \pm 0.2$	0.531
	5		$238 \pm 8$	$4.9 \pm 0.1$	$4.8 \pm 0.1$	0.393
	15		$342 \pm 11$	$5.8 \pm 0.5$	$5.7 \pm 0.5$	0.443
	20		$566 \pm 10$	$8.5 \pm 0.2$	$8.4 \pm 0.1$	0.615
	0	Yes	$234 \pm 28$	$4.8 \pm 0.3$	$4.6 \pm 0.2$	0.569
	5		$278 \pm 15$	$5.8 \pm 0.2$	$5.5 \pm 0.3$	0.460
	15		$451 \pm 18$	$7.5 \pm 0.3$	$7.4 \pm 0.3$	0.609
B	20	No	$484 \pm 9$	$7.4 \pm 0.1$	$7.3 \pm 0.1$	0.836
	5		$219 \pm 3$	$4.5 \pm 0.1$	$4.4 \pm 0.1$	0.446
	15		$427 \pm 48$	$6.4 \pm 0.6$	$6.3 \pm 0.6$	0.479
	20	Yes	$634 \pm 18$	$9.5 \pm 0.3$	$9.4 \pm 0.3$	0.670
	5		$268 \pm 4$	$4.9 \pm 0.1$	$4.8 \pm 0.6$	0.547
	15		$454 \pm 7$	$7.6 \pm 0.1$	$7.5 \pm 0.2$	0.765
	20		$604 \pm 21$	$9.2 \pm 0.6$	$9.1 \pm 0.6$	0.726

This chemical bonding has already been analyzed and discussed for the reaction between the OH groups of cellulose/wood flour and MDI-prepolymer in previous publications [21–23]. Additional crosslinking due to the excess isocyanate might also contribute to improve the modulus, although no indication of this was observed from the dynamic mechanical results. Reactions leading to higher crosslinking density in the PU matrix

would have resulted in a higher transition temperature for the maximum in  $\tan \delta$  of the composites, and this was not observed.

The flexural and impact response of the same composites is summarized in Table 3. When filled with small content of wood flour (5 wt%), both the flexural performance and impact strength of the composites decrease, while the reduction of the latter is more important. As the content of filler further increases, the flexural properties also increase markedly with increasing filler loadings (as it was observed in the tensile behavior, discussed above). However, the impact strength continues to decrease. The reduction of the impact resistance with filler content was also observed by other authors [24–29], who indicated that the incorporation of fibers into the matrix increased the stiffness of the composites, but decreased the absorbed energy under impact conditions. They explained this behavior as a change from ductile to brittle fracture due to the incorporation of rigid elements in the polymer. Besides, they suggest that the addition of short-fiber reinforcements, probably favored the fracture initiation and propagation in the composite. On the other hand, the reduced flexibility of the polymer matrix and the alteration of the local stress distribution as filler concentration increases are the reasons stated by Doh et al [30] and by Hristov et al. [31], respectively, to explain the reduction of impact strength in composites.

#### WATER SORPTION

Figure 6 presents the water sorption characteristics of the composites prepared with different amounts of WF and fire retardants (B series, no solvent). As was shown from SEM pictures, the medium where the water sorption takes place is highly heterogeneous and contains particles with lower sorption capacity, enclosed in a high-water-permeable matrix of lightweight polyurethane. Thus, the measurements show that composites absorb less water than the neat matrix, although a clear trend was not observed with WF concentration. No explanation for this behavior was found. Probably, the stronger interfacial adhesion in the tougher composites resulted from the interactions between WF and PU molecules (chemical bonding), prevented water molecules from diffusing through the composite interface. The bonding of the PU to the WF rigid particles reduced matrix swelling during water uptake, while consumption of WF hydroxyl groups during

**Table 3. Flexural and impact performance of recycled polyol composites.**

Series	% WF	Solvent	$E_b$ (MPa)	$\sigma_u$ (MPa)	$a_I$ (kJ · m <sup>-2</sup> )
A	0	No	193.5 ± 23.2	6.6 ± 1.0	5.78 ± 0.50
	5		177.4 ± 11.2	5.9 ± 0.4	4.48 ± 0.79
	15		215.4 ± 16.7	6.6 ± 0.3	4.42 ± 0.49
	20		354.3 ± 10.9	9.7 ± 0.2	3.99 ± 0.77
	0	Yes	191.5 ± 19.5	6.7 ± 0.6	8.60 ± 1.14
	5		164.1 ± 19.7	5.8 ± 0.4	5.27 ± 0.33
	15		288.0 ± 17.7	8.7 ± 0.4	5.91 ± 0.41
	20		325.2 ± 15.6	9.4 ± 0.3	5.60 ± 0.75
B	5	No	171.0 ± 3.7	5.6 ± 0.1	5.86 ± 0.69
	15		297.9 ± 13.8	7.8 ± 0.2	3.57 ± 0.34
	20		606.3 ± 31.0	15.3 ± 0.2	3.57 ± 0.18
	5	Yes	193.3 ± 5.3	6.3 ± 0.1	6.81 ± 0.76
	15		344.3 ± 14.8	9.9 ± 0.5	5.21 ± 0.70
	20		495.9 ± 25	13.5 ± 0.3	4.70 ± 1.01

the reaction filler-matrix, reduced the WF hygroscopicity. Moreover, the path for moisture diffusion became more tortuous due to WF addition, thus, retarding the water transport.

#### EFFECT OF FIRE RETARDANT ADDITION

Table 4 shows the density and flexural modulus of series B composites prepared with fire retardant. The density of the reinforced PU increases with fire retardant addition, although the increase is more noticeable for the composites than for the unfilled PU. The flexural modulus also increases with the  $\text{Al}(\text{OH})_3$  addition, although the trend is not as clear when the viscosity modifier is added to the system.

For all the samples prepared with the fire retardant, the low limiting oxygen index (LOI) to sustain burning was found to be between 24 and 28%, that is, it was found to be

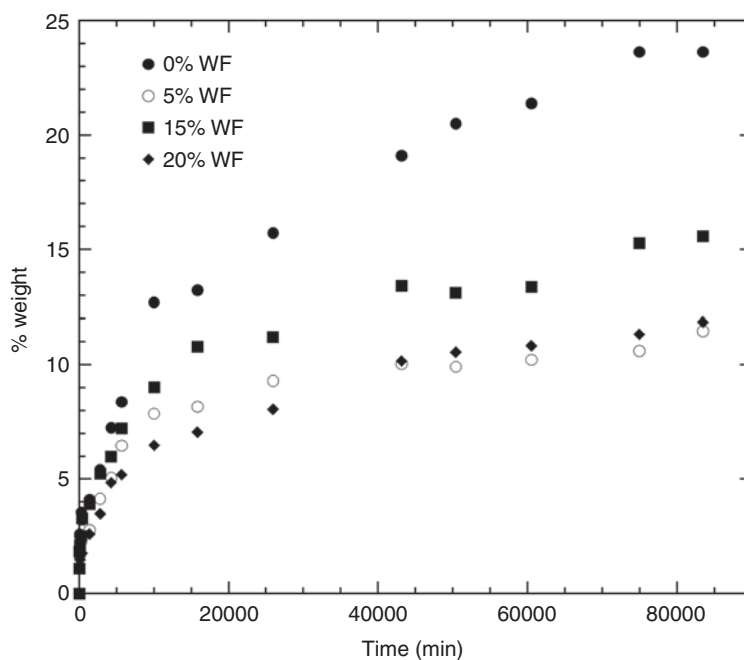


Figure 6. Water sorption of lightweight composites prepared with fire retardant (no solvent, B series).

Table 4. Density and flexural modulus of series B composites prepared with fire retardant.

% WF	Solvent	$E_b$ (MPa)	Density ( $\text{kg m}^{-3}$ )
0	No	$476 \pm 69$	0.503
5	No	$663 \pm 257$	0.567
15	No	$986 \pm 332$	0.681
20	No	$1027 \pm 250$	0.701
0	Yes	$511 \pm 106$	0.584
5	Yes	$512 \pm 257$	0.644
15	Yes	$1149 \pm 199$	0.734
20	Yes	$1037 \pm 206$	0.742

independent of solvent addition and wood flour content. Moreover, all the samples prepared with fire retardant burnt completely before the end of the test, except those prepared with the highest amount of wood flour (20 wt%), which were the only ones that completed the 180 s period and resulted in a burnt length below 50 mm.

## CONCLUSIONS

Lightweight reinforced PUs were successfully prepared using a recycled polyol and a natural particulate filler (WF). The incorporation of additives (filler, viscosity modifier, fire retardant) affected both the physical and mechanical properties and the morphology of the resulting composites. The general trend is that the mechanical (tensile and flexural) and the thermo-mechanical (DMA results) performance as well as the density of the composites increase, although the impact strength decreases, as the wood flour content increases. The density and flexural modulus of the reinforced PU also increases with fire retardant addition. The WF composites showed a clear reduction in the water uptake capacity with respect to the neat PU. The incorporation of a 25% of flame retardant did not generate the desired effect, since improvements in the fire behavior were found only in case of the 20 wt% wood flour composites. Other flame-retardants should be tested in the future.

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