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Modified Wood Flour and its Effect in Polymer Matrix

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Abstract

Initial tests have demonstrated that wood flour (WF) blends exceptionally well with a wide range of commonly used rubbers, including natural rubber, styrene rubber, acrylonitrile-butadiene rubber, butadiene rubber and isoprene rubber. Effective reinforcement of rubber materials depends heavily on the fine dispersion of the filler. Combining WF with a rubber matrix is an innovative way of creating sustainable composite materials. The correct modification of the wood surface is key to success, as this allows for good dispersion, high adhesion and balanced mechanical properties. Thanks to these technologies, there are now many potential applications in the automotive industry, construction, footwear, and sealing and damping elements. In this study, WF obtained as a by-product of wood pellet manufacturing was used as the filler. Furthermore, the interaction between the filler and the rubber matrix is crucial in enhancing reinforcement, whether through chemical bonding or physical interactions. Therefore, modification of the WF is necessary. This paper deals with the incorporation of modified WF of different fractions into a polymer matrix and examines how this effects the rheological behaviour and mechanical parameters of polymer blends and vulcanizates compared to polymer blends filled with unmodified WF. The measured results suggest that selecting the correct fraction of modified WF can lead to an improvement in the resulting properties of polymer blends.

Keywords

Silane, Reinforcement, Polymer blends

Introduction

In recent decades, growing global concerns over climate change and global warming have led to increased efforts to address these critical issues [1]. In light of carbon neutrality goals and the gradual depletion of global oil resources, promoting the use of biomass materials, which are abundantly available as alternatives to petrochemical-based materials presents an effective strategy. This approach aligns with current priorities in environmental protection, energy conservation, and the development of a low-carbon economy [2, 3].

Tires represent the most significant rubber products, both in terms of their importance and production volume. The tire industry consumes the majority of natural and synthetic rubber, as well as approximately 90% of the global carbon black production [4]. In spite of the functionalities of carbon black as the reinforcing filler, it is petrochemical and nondegradablematerial, and a large amount of energy is consumed in its production [5, 6]. Moreover, the use of carbon black poses environmental hazards, beginning with its production process, which releases significant amounts of carbon dioxide into the atmosphere [6].

The wood-rubber composites offer multifunctional properties and hold strong potential for a wide range of applications [7]. WF is a significant byproduct of the wood processing industry, comprising approximately 45 – 52% of total wood waste [8]. WF is typically a residual waste and poses environmental challenges; however, its abundance and low cost make it an attractive option for use as a filler [9].

One of the primary limitations of lignocellulose-based composites is their hydrophilic nature, which results in poor compatibility with hydrophobic polymer matrices. To overcome this challenge, coupling agents are employed to block hydrophilic sites and promote stronger interactions between the filler and the matrix. Silanes, in particular, are highly effective due to their bifunctional groups, which can chemically bond with both the lignocellulose fillers and the polymer matrix. This bonding forms a crucial bridge between the two components, enhancing interfacial adhesion. Consequently, the composite structure becomes more homogeneous, leading to markedly improved mechanical properties [10].

This work is innovative in that it focuses on the use of WF as a filler in rubber matrix, which is an area that has not been sufficiently explored. The problem is the separation of WF, which can be the result of several factors. Therefore, this work is focused on the modification of WF with different fractions and incorporation into rubber matrix. The aim is improving compatibility between the WF and rubber matrix, to increase reinforcement of polymer blends, leading to improved mechanical properties. In this way, the work contributes to the development of sustainable rubber composites containing renewable raw materials.

Materials and Methods

Materials

The biowaste - WF derives from the production of wood pellets of the company ECPU, s.r.o. was used for research. The shape of the WF was characterized by SEM instrument (VEGA 3; TESCAN). Images were taken at 500 x magnification and a scale bar of 50 µm. The WF sample was dried due to the reduction or elimination of the excess moisture and the WF structure or fraction was prepared to investigate the two different particle sizes. The first particle size was between 63 - 125 µm and the second particle size was between 40 - 63 µm. The particle size distribution of both WF fractions was determined using a Laser Diffraction Particle Size Analyzer (Bettersizer 2600). Subsequently, the fractions were modified with 3-aminopropyltriethoxysilane (APTES), serving as a coupling agent to enhance the interfacial interaction between the WF filler and the rubber matrix, in which the filler was intended to be incorporated.

Preparation of modified WF

For silane modification, WF was treated at room temperature (25 °C) with an aqueous solution of APTES. The APTES was added at a concentration of 5 wt% based on the weight of the WF, into a solvent mixture of ethanol and distilled water in a 9:1 ratio. The high volatility of ethanol aided in the rapid drying of the modified WF. To facilitate the hydrolysis of the silane, the solution's pH was adjusted to 5 using acetic acid [11]. The stirring time was 10 min. The WF was added to the solution and stirred at 2000 rpm for 2h to produce the modified WF. After the treatment of the WF was an excess solution separated on centrifuge (Eppendorf 5810 centrifuge) and obtained modified WF was dried at 100 °C for 4 h. The elemental composition of prepared WF samples was analyzed using Energy-dispersive X-ray (EDX) spectroscopy to confirm modification of WF by silane.

Preparation of polymer blends

Five rubber compounds were prepared using a Brabender mixer (ASTOGRAPH EC PLUS) with a chamber volume of 80 cm^3. The mixing was carried out in a single-step process at a temperature of 80 °C and a rotor speed of 50 rotations per second. Natural rubber (SMR 20) based compounds were prepared by incorporating WF and modification WF of two different particle size ranges (63 - 125 µm, 40 - 63 µm). The sample is an unfilled sample (US), without filler. Formulations of samples are presented in table 1.

The mixing process began with a 3 min mastication of natural rubber, followed by the addition of zinc oxide and stearic acid. Subsequently, the fillers were incorporated and mixed for an additional 3 min. Finally, N-cyclohexyl-2-benzothiazole sulfenamide and sulfur were added to the blend. The total mixing time was 16 min. A detailed schematic of the rubber compound preparation process is presented in figure 1. From the figure 1, an increasing mixing temperature due to incorporating of filler. The addition of filler will affect compound properties, viscosity, and strength of the compound [12]. This confirms that viscosity or stiffness is the main factor responsible for the higher mixing temperature [13]. After Brabender mixing, the compounds were further homogenized using a two-roll mill and they allowed to rest for 24 h at room temperature. The prepared rubber compounds were subjected to compression molding at 160 °C, in accordance with their individually determined optimal cure times.

Rheometric characteristics

Rheological behaviors of WF and modification WF of two

Table 1: Formulations of samples.

Component Sample	Rubber (100 dsk)	Filler (15 dsk)			
US	SMR 20	-	-	-	-
WF63-125	SMR 20	WF63–125 µm	-	-	-
MWF63-125	SMR 20	-	Modified WF63–125 µm	-	-
WF40-63	SMR 20	-	-	WF40–63 µm	-
MWF40-63	SMR 20	-	-	-	Modified WF40–63 µm

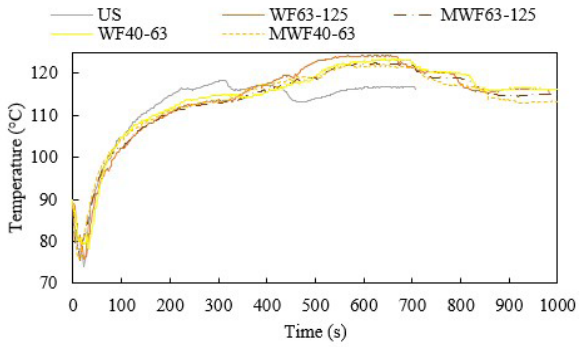


Figure 1: Mixing process of polymer blends.

different particle size ranges (63 - 125 µm, 40 - 63 µm) in the polymer matrix on the specification of the vulcanization process was measured using curing characteristics parameters which were determined via production rubber process analyzer oscillation rheometer (PRPA 2000) at the constant temperature of 160 °C, with oscillating arch of 0.5, frequency of 1.67 Hz. The parameters studied include scorch time (t_{s2}), curing time (t_{c90}), minimum torque (M_L), maximum torque (M_H) and delta torque (MH-ML). The cure rate index (CRI) was calculated, using:

$$CRI = \frac{100}{t_{c90} - t_{s2}} \quad (1)$$

Variations in rheometric torque with filler loading can be used to evaluate the filler–matrix interaction or reinforcement effect, commonly referred to as the reinforcement factor (αf), which can be calculated as follows [14]:

$$\frac{D_{max} - D_{min}}{D^0_{max} - D^0_{min}} - 1 = \alpha_f \frac{m_f}{m_p} \quad (2)$$

Where, $D_{max} - D_{min}$ represents the torque difference during vulcanization for the filled rubber, while $D^0_{max} - D^0_{min}$ corresponds to the torque change for the unfilled rubber, m_p denotes the mass of rubber in the blends, and m_f is the mass of filler in the blends [14].

Mechanical properties

Double-sided test specimens were mechanically cut from the cured rubber slabs. The mechanical properties were then evaluated using a SHIMADZU Autograph AG-X Plus universal testing machine, operated at a head speed of 50 mm. min^{-1}. Tensile strength and elongation at break were determined from the average of five tested specimens and assessed based on the corresponding stress–strain curves for each sample. The hardness was measured by a Shore A hardness tester.

Results and Discussions

Characterization of filler – WF and modified WF

The WF used in this study was derived from spruce sawdust, which is classified as softwood. Softwood is generally more flexible, while hardwood tends to be stiffer. Typically, softwood flour contains long, thin fibers due to the high presence of longitudinal tracheid. In contrast, hardwood flour consists of shorter, thicker fibers, which can be attributed to the greater abundance of radial and axial cellular components [15]. Particles of WF appear as long and thin with layered wood-like structures rather bundles than individual fibers (Figure 2). Yin described WF particles as empty cell or tube structures [16]. The particle geometry and average aspect ratio varied significantly, with particle sizes ranging widely from several micrometers to millimeters [17].

Two different fractions of WF particles were then prepared, with sizes of 63 - 125 µm and 40 - 63 µm. Figure 3 and figure 4 show the particle size distribution of the WF samples (WF63-125 and WF40-63). Figure 3 shows the WF63-125 sample and reveals two peaks. The first, wider peak represents the average particle size of the sample, which is 117.5 µm. The second, higher but narrower peak indicates the presence of larger WF particles. Based on scanning electron microscopy (SEM) analysis, we know that WF has long and thin layered structures fibers. Despite extended sieving, it was challenging to obtain particles with diameters precisely matching the mesh size [18]. This results in inconsistent particle diameters. Figure 4 focuses on particle sizes of 40 – 63 µm. The highest intensity (peak) was observed at a size of 63.42 µm, i.e., the average size of the WF40–63 particles is 63.42 µm.

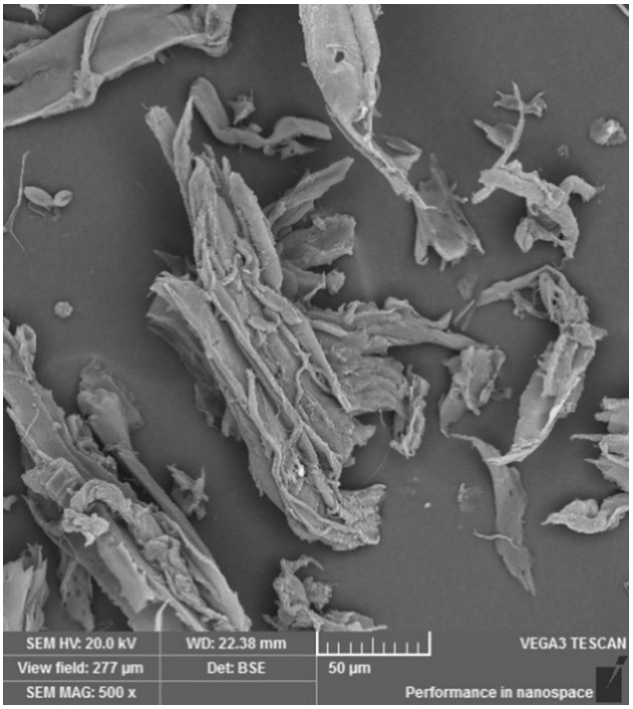


Figure 2: SEM micrographs of WF from industry, at 500 x magnification.

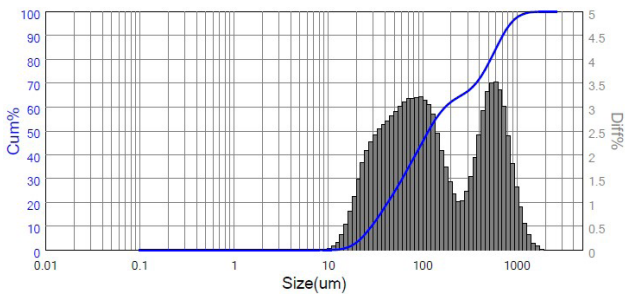


Figure 3: Particle size distribution of WF63-125 sample.

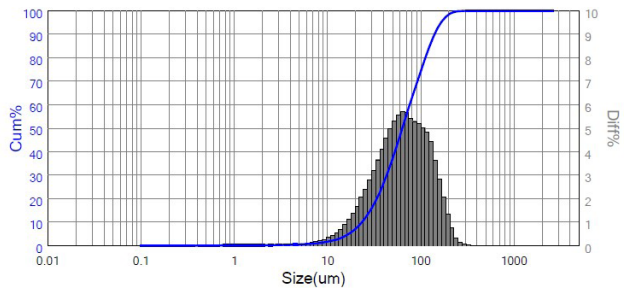


Figure 4: Particle size distribution of WF40-63 sample.

The elemental composition of the prepared WF samples was analyzed using EDX and the results are presented in table 2. Changes in the elemental composition of the WF after modification were monitored. The most represented element in the samples is carbon, which is attributed to the wood material [19]. Surface modification of cellulosic materials using alkoxysilanes under hydrolytic conditions involves three key steps: (1) hydrolysis of the alkoxy groups from silane in the presence of water, resulting in the formation of silanes (SiH_3OH); (2) adsorption of these silanol groups onto the cellulose surface via hydrogen bonding between silanol and hydroxyl groups of cellulose; and (3) chemical condensation, leading to the formation of siloxane bridges (Si–O–Si) and covalent grafting onto the cellulose surface through Si–O–C bonds [20]. Modification of WF by APTES caused a decrease the carbon and an increase in silicon (Si) for both fractions of particle sizes of WF. Autor confirmed the attachment of γ-Aminopropyltriethoxysilane to WF by the presence of a peak corresponding to Si and calculated percentage of Si in WF which was modified with this silane [21].

Rheometric characteristics of polymer blends

The rheometric characteristic known as M_L, which corresponds to the torque at the initial stage of vulcanization, is indicative of the viscosity of the blends (Table 3). Introducing any type of WF resulted in some decrease in the M_L compared with the unfilled blend. The use of WF or lignin in rubber blends as a substitute for additive is driven by both economic and environmental considerations [6, 22]. This may be the cause of softening of the mixtures and decrease M_L in polymer blends with WF. The crosslinking density of vulcanizates was determined by M_H - M_L, and the greater the difference, the higher the crosslinking density. This effect can be attributed to the incorporation of the silane coupling agent, which enhances interfacial bonding between the filler and the rubber matrix. This enhanced interaction optimizes the vulcanization process, enabling the system to achieve an ideal curing state, as evidenced by increased vulcanization efficiency and higher crosslinking density. Incorporating modified WF slightly increases the crosslink density within the polymer blend, indicating improved network formation during vulcanization. The parameter t_{c90}, representing the optimum cure time for vulcanization, is critical for the vulcanization process. Accurate evaluation of this cure time is essential for the proper molding of elastomeric compounds [14]. The lowest value of t_{c90} is recorded for SW40-63 sample. It is clear that the modified WF in polymer blend caused to increase of t_{c90} value. CRI is a direct measure of the fast-curing nature of polymer blends [14]. The WF modification caused a decrease in the CRI. It is evident that αf values increase with the use of modified fillers, and the trend can be arranged as follows: MWF63-125 > WF63-125 > MWF40-63 > WF40-63. The highest value of αf indicates the strongest filler–matrix interaction and, consequently, the most pronounced reinforcing effect of the modified WF. Conversely, the lowest αf value was observed for samples filled with WF in the 40 – 63 μm fraction, suggesting a relatively modest reinforcing effect [23].

Table 2: The elemental composition of WF before and after modification.

Samples	Element (%)				
	C	Si	Ca	S	K
WF63-125	99.57	0.15	0.10	0.07	0.07
MWF63-125	99.36	0.41	0.07	0.07	0.03
WF40-63	99.38	0.20	0.15	0.08	0.08
MWF40-63	99.28	0.39	0.10	0.08	0.04

Mechanical properties of polymer samples

The mechanical properties of WF-filled rubber composites depend on a number of factors, including filler loading, the degree of filler dispersion, and the quality of filler–rubber adhesion [24]. Smaller WF particles generally result in stronger interfacial adhesion and improved mechanical properties, thanks to their increased surface area and better dispersion within the polymer matrix. Conversely, larger particles can result in weaker interfacial interactions, which can lead to debonding, void formation and reduced composite strength [24]. Table 4 presents the results of selected mechanical properties performed on polymer vulcanizates, including US, unmodified samples WF63-125, WF40-63) and modified

Table 3: The rheometric characteristics of polymer samples.

Samples	M_L (dN.m)	M_H (dN.m)	$\Delta M = (M_H - M_L)$ (dN.m)	T_{s2} (min.)	T_{c90} (min.)	CRI (min.$^{-1}$)	αf
US	1.10	17.74	16.64	2.99	4.87	53.19	-
WF63-125	1.04	24.84	23.80	3.22	5.09	53.48	2.87
MWF63-125	0.85	24.78	23.93	3.62	5.74	47.17	2.92
WF40-63	0.98	24.49	23.51	3.06	4.78	58.14	2.75
MWF40-63	0.70	24.38	23.68	3.63	5.83	45.45	2.82

samples (MWF63-125, MWF40-63). The stress-strain properties of the cured blends containing different amounts of WF were measured. The sample with modified WF containing the smallest particle sizes exhibited the highest stress–strain curve (Figure 5). The relatively low tensile strength was attributed to the geometric characteristics of the WF particles. Unlike fibres, which have a uniform circular cross-section and certain aspect ratios, these irregularly shaped fillers were unable to support the stress transferred from the rubber matrix. In other words, incorporating WF with a particle size of 63 – 125 μm was insufficient to ensure uniform stress transfer within the rubber matrix. This resulted in localized stress concentrations at the filler–rubber interface, leading to low reinforcement. However, adding modified WF to the polymer blend improved tensile strength, primarily due to the enhanced dispersion of the filler within the matrix [24].

The US sample represents the unfilled mixture. Incorporating WF into the polymer matrix increases hardness, primarily due to the filler's increased rigidity and altered surface texture. The samples were filled with a small amount of filler. The different WF fractions and their modifications only moderately influenced hardness (Figure 6). The hardness of WF - based composites is largely influenced by filler content and particle size. Optimal hardness is generally achieved at a filler loading of approximately 35 wt%, but exceeding this concentration can lead to reduced hardness due to inadequate dispersion of the filler within the polymer matrix [25].

Conclusion

WF, a by-product of wood processing, is widely used as a natural filler in the fabrication of filler – polymer composites. Its abundance, low cost and easy availability make it highly attractive material for various industrial applications. In this study, WF with particle size of 63 - 125 μm and 40 - 63 μm was used. Both fractions of WF were then modified using APTES as a coupling agent to improve the interaction between the filler and the rubber matrix. This was done consideration of the role of WF as a filler. WF particles appear as long and thin with layered wood-like structures rather bundles than individual fibers. The modification of the WF was confirmed by an increase in silicon in the elemental analysis. After incorporating WF and modified WF into the polymer matrix, minimal differences in rheological properties were detected. WF modification was found to increase the crosslinking density and a decrease in the CRI. It is evident that αf values increase with the use of modified fillers, and the trend can be arranged as follows; MWF63-125 > WF63-125 > MWF40-63 > WF40-63. The different fractions of WF and its modification had only moderate influence on hardness. The modification resulted in an increase of 9.97% in the tensile strength of sample MWF63-125. The greatest improvement was observed in sample MWF40-63, with an increase in tensile strength of 12.20%. Based on the results obtained, several recommendations can be made for future research. Firstly, it would be advisable to focus on achieving the smallest possible particle size of WF to enhance filler dispersion and performance. Additionally, exploring various modification techniques and thoroughly investigating their effectiveness is recommended. In particular, further analysis should focus on confirming filler modification, with an emphasis on SEM imaging of both the filler and the resulting composites to better understand morphological changes and interfacial interactions.

Table 4: Mechanical properties of polymer samples.

Samples	Tensile strength (MPa)	Elongation at break (%)	Shore A hardness
US	10.97 ± 0.47	503.22 ± 15.27	41.80 ± 0.51
SW63-125	14.55 ± 1.00	554.19 ± 25.05	56.18 ± 0.93
SMW63-125	16.00 ± 1.08	586.43 ± 7.40	57.02 ± 1.04
SW40-63	16.56 ± 0.67	536.69 ± 19.88	56.02 ± 1.04
SMW40-63	18.58 ± 1.48	611.77 ± 5.28	56.98 ± 0.42

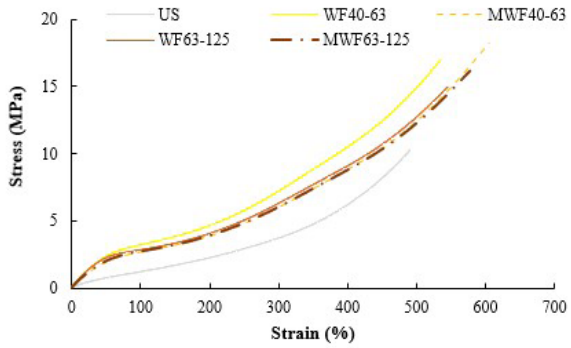


Figure 5: Stress-strain curves of polymer samples.

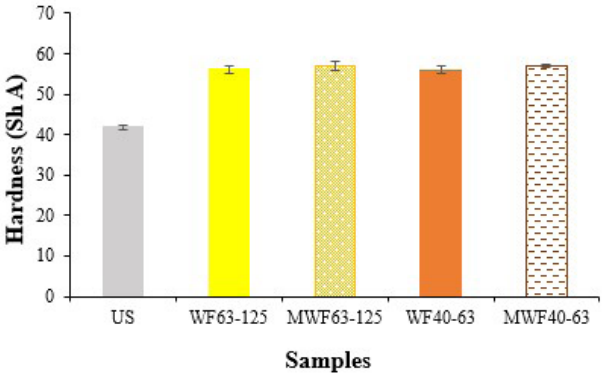


Figure 6: Hardness of polymer samples.

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Conflict of Interest

None.

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