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The Impact, Thermal and Morphological Properties of Cellulosic and Cellulosic-Synthetic Reinforced Polymer Composites

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Abstract
In recent decades, there has been a growing use of filler-reinforced thermoplastics, especially those enhanced with natural fillers, within the plastic industry. These materials are preferred for their ability to improve the properties of polymers, driven by benefits such as low density, biodegradability, reduced CO_2 emissions, absence of health hazards, and cost-effectiveness. In this study, wood powder/polypropylene composites (wood/PP) and wood powder/ glass powder/ polypropylene hybrid composites with varying filler concentrations were produced using injection moulding. The objective was to investigate how filler content affects the impact mechanical characteristics of these composites. Additionally, the study compared the properties of wood/PP composites and wood powder/ glass powder/ polypropylene composites fabricated experimentally with those modelled using finite element analysis. These composites are, widely employed in automotive products. Moreover, scanning electron microscopy, Fourier transform infrared spectroscopy, thermogravimetry and differential scanning calorimetry were performed. The results indicated that the impact mechanical properties of wood/PP composites and wood powder/ glass powder/ PP composites reduced with increasing filler addition. Numerical modelling using finite element analysis results also showed that the impact mechanical properties reduced with increasing fibre additions.
Keywords: Polypropylene, Composite, Finite element analysis, Impact properties, Biodegradability, Fillers

1. Introduction

Recently, there has been increasing interest in polypropylene (PP) composites enhanced with particulate fillers. Polypropylene (PP) is valued not only for its excellent stiffness and environmental adaptability but also for its versatility in accommodating various types of fillers [1, 25]. Cellulosic fillers are commonly employed to lower production costs and enhance properties such as rigidity, strength, dimensional stability, crystallinity, thermal conductivity, and electrical conductivity. However, they can also adversely affect properties such as impact resistance and deformability [1,2,30].

Among fillers, wood powder and glass powder can be utilized to reinforce thermoplastics, especially polypropylene [1, 25, 26]. Depending on their concentration, wood powder and glass powder can improve the stiffness of materials. Wood powder-filled PP and wood powder/glass powder/ PP hybrid composites finds applications in automotive components such as heater casings, interior and exterior decorations, fan cases, bumper parts, household appliances, and engineering plastics [3, 27,30].

Cellulosic fillers like wood powder have gained popularity due to their affordability, low density, environmental benefits, reduced health risks, and improved performance in technical and standard plastics [3, 4, 28]. Wood powder, sourced from materials such as sawdust, pulp mill residue, bark, nutshell, and straw, is particularly attractive for enhancing the mechanical properties and thermal stability of thermoplastics while reducing raw material costs [1, 25, 29,30].

Impact velocity is commonly classified into low and high categories based on the mass and speed of the projectile involved. A low-velocity impact occurs when a heavy projectile, such as a dropped tool, strikes with relatively low energy. In contrast, a high-velocity impact involves a lighter projectile, like debris or small-arms fire, striking at high speed, often resulting in penetration of the target [5].

High-velocity impacts typically cause localized damage because the contact duration between the impactor and the target is short. This results in the completion of the impact event before stress waves can propagate to the edges of the structure. Conversely, low-velocity impacts involve a longer contact duration, allowing the target material to absorb more energy through elastic deformation [5].

Vieille et al. [6] discovered that carbon-fibre composites impregnated with thermoplastic resins such as PEEK and PPS showed smaller delamination areas following impacts at low velocities compared to thermosetting resins. This was due to their increased toughness. Meanwhile, Gao et al. [7] emphasized the impact of cooling rate on the behaviour of thermoplastic composites. Carbon/PEEK laminates cooled quickly (20°C/min) and exhibited higher resistance to impact loads than those cooled slowly (1°C/min).

Garcia-Gonzalez et al. [8] studied the impact performance of PEEK-based composites reinforced with short carbon fibres at low temperatures. They concluded that energy absorption in the composite decreased significantly at -50°C and -75°C due to the polymer transitioning from ductile to brittle. This change in temperature restricted the mobility of polymer chains, leading to a phase transition.

Hazzard et al. [9] explored the influence of fibre orientation in thin UHMWPE composite laminates under 150 J impact energy using a blunt hemispherical impactor. Their study revealed that quasi-isotropic laminates exhibited an average of 43% lower maximum back-face deflection compared to laminates with 0°/90° orientation. Additionally, they observed a significant reduction in the time taken to reach maximum back-face deflection.

Karduman et al. [10] investigated the impact of stacking sequences involving jute/PP nonwoven and flax/maleated polypropylene (MAPP) woven fabrics in hybrid composites using the film stacking method. They found that the NWWN sequence demonstrated higher energy absorption during impacts. This superior performance was attributed to the energy-absorbing properties of the top-layered needle-punched nonwoven fabric and the stress concentrations at adjacent interfaces.

Jogur et al. [5] carried out an extensive review of the impact behaviour of composite materials, focusing on how different materials and testing parameters affect their properties. The article thoroughly examined the impact of integrating high-performance fibres, natural fibres, or hybrid combinations into thermoplastic (TP) composites. Additionally, it provided a concise overview of the essential properties of TP polymers. The study explored the effects of hybridizing fibres and matrices, varying environmental conditions, diverse textile preform structures, and treatments designed to modify fibre surfaces to enhance the impact characteristics of thermoplastic composites. The research underscored the potential applications of TP composites across industries such as automotive, aerospace, and medical sectors, emphasizing their versatility and promising prospects in advanced industrial applications.

Asaithambi et al. [11] developed hybrid composites consisting of banana/sisal fibres (30 wt%) reinforced with PLA (70 wt%) using an injection moulding process to investigate how surface treatments affected mechanical properties. Both types of fibres were treated with benzoyl peroxide (6 wt%) to enhance adhesion between the fibres and the PLA matrix. The study demonstrated improved wettability and enhanced interfacial adhesion, effectively restricting the movement of the PLA matrix. Surface treatment resulted in higher tensile and flexural moduli in the treated hybrid composites compared to untreated hybrids and virgin PLA.

Pérez-Fonseca et al. [12] examined the influence of coupling agents on mechanical properties of pine/agave natural-fibre reinforced HDPE hybrid composites. Their findings indicated that malleated polyethylene (MAPE) coupling agent had a significant impact, particularly with agave fibres, leading to improved tensile, flexural, and impact properties in the hybrid composite. However, the inclusion of pine fibres reduced fibre uptake. In pine/agave composites with a 20/80 ratio and 30 wt% total fibre content, the impact, flexural, and tensile strengths increased by 41%, 22%, and 13%, respectively, compared to composites containing pine fibres alone.

In this study, wood powder-reinforced polypropylene composites (wood/PP) and wood

powder-glass powder reinforced polypropylene hybrid composites with varying filler contents were manufactured using injection moulding to evaluate how filler content affects their impact mechanical properties. The study also stretched the boundaries of knowledge by using finite element analysis to model these composites, since this method has not been used before to evaluate the impact mechanical properties of the composites. The finite element results were then compared to the experimental results. The subsequent sections detail the methodology, results, discussions, and conclusions of this study.

Materials

The materials selected for this study included polypropylene (PP) pellets with a melt index of 15 g/10 min as the matrix material. PP is widely preferred in the automotive industry for its suitability in injection moulding processes. As reinforcements, glass powder and wood powder obtained from Marple trees sourced from South Africa, were utilized. The wood powder underwent pelletization using a granulation machine provided by Herald Co., Ltd., with the addition of crystalline polyalpha olefin (CPAO) as a binder during the processing of the pellets.

Injection Moulding Process

Dumbbell-shaped specimens were prepared using the TMC injection moulding machine. This included variations of polypropylene with different filler contents: 0%,10%, 20%, 30%, 40% and 50% by weight, respectively.

All dumbbell-shaped test specimens were fabricated using a 30-ton TMC PSS TT-30F6 injection moulding machine, with barrel temperatures set between 150 and 210°C. Prior to injection moulding, the fillers underwent drying in an oven at 90°C for a minimum of 16 hours to remove moisture.

Fourier Transform Infrared Spectroscopy (FTIR)

Composite samples were analysed using an FTIR instrument covering wavelengths from 300 to 3500 cm^{-1}. Spectral data were collected at a resolution of 4 cm^{-1}, averaging data from 20 scans.

Scanning Electron Microscopy (SEM)

SEM images were acquired using Zeiss SEM 5000 equipment from Hilden, Germany, operating at an accelerating voltage of 30 KV. Prior to imaging, samples were coated with gold to enhance conductivity, facilitating detailed and precise micrography of specimen surfaces.

Thermogravimetric Analysis (TGA)

Thermal stability analysis was conducted on mono and hybrid composite samples using a Perkin-Elmer STA-6000 instrument from Shelton, USA. TGA was performed under a nitrogen atmosphere with a heating rate of 10ºC/min, ranging from 20ºC to 600ºC. The nitrogen flow rate was maintained at 100 mL/min to ensure consistent testing conditions.

Differential Scanning Calorimetry (DSC)

Differential scanning calorimetry (DSC) was employed to determine the glass transition temperature (Tg) and melting temperature (Tm) of the composite samples. The analysis involved heating the samples from 20ºC to 600ºC in a nitrogen environment at a flow rate of 100 mL/min. By monitoring the heat flow during this process, the Tg and Tm values of the samples were determined.

Impact Testing - Finite Element Analysis (FEA)

A detailed finite element model (FEA) of the specimen was developed using the ABAQUS software version 2022. This process involved accurately representing the geometry, material properties, and meshing of both injection-moulded mono and hybrid composite formulations. Subsequently, material properties such as elasticity and plasticity, based on experimental data, were defined in ABAQUS to simulate the mechanical behaviour under impact loading. Boundary conditions were applied to simulate the fixture or clamping mechanism holding the specimen during the impact test. Loading conditions were set to replicate the impact event by applying an impulse to a specific region of the model representing the impactor. Solver settings within ABAQUS were adjusted for explicit dynamics analysis, which is adept at capturing high-speed transient phenomena such as impact loading.

Impact Testing - Experimental

The Charpy impact test was carried out using a 5.5 J pendulum impact tester (Toyoseiki) according to ASTM D 256-05 standards. The specimen size was 10 mm x 60 mm. Each test was performed with five specimens to ensure accuracy and reliability of the results.

Validation of Results

Experimental findings were utilized to validate the outcomes of the finite element analysis (FEA) within a tolerance range of ± 5% [13]. This standard protocol facilitated the assessment of percentage errors. Additionally, a statistical approach using Analysis of Variance (ANOVA) was employed for validation. ANOVA calculations involved determining the F-statistic and P-value. The F-statistic compares mean squares to assess significant differences between group means, while a P-value below 0.05 indicates a significant test result. Meeting both criteria confirm the significance of the test outcomes.

Results And Discussions

FTIR

Figure 1 presents the FTIR spectra of various samples: wood powder/glass powder/ PP hybrid composites, wood powder/polypropylene (WP) and neat polypropylene (PP). These spectra highlight differences in peak intensities across different wavenumbers, aiding in comparative analysis. Fourier Transform Infrared Spectroscopy (FTIR) is invaluable for characterizing material composition and structure.

In neat Polypropylene (PP), characteristic peaks associated with its molecular structure were evident. Notably, a strong peak in the range of 2960-2860 cm^{-1} corresponded to the stretching vibration of C-H bonds in methylene (-CH2-) groups. Additionally, peaks around 1450-1375 cm^{-1} were attributed to CH_2 bending vibrations, with another prominent peak around 1150-950 cm^{-1} indicating (-CH2-) bending vibrations. Furthermore, a peak at approximately 840 cm^{-1} was identified as the rocking vibration of (-CH2-) groups. The absence of peaks related to functional groups such as hydroxyl (-OH), carbonyl (C=O), or epoxy groups confirms the purity of the polypropylene.

For Wood Powder/PP Composites, the FTIR spectrum exhibited characteristic polypropylene peaks alongside additional peaks associated with wood components such as lignin, cellulose, and hemicellulose. Specific peaks included bands around 1730-1600 cm^{-1} indicating C=O stretching vibrations in hemicellulose and lignin, and peaks in the 1240-900 cm^{-1} range corresponding to C-O stretching vibrations in cellulose and hemicellulose. Additionally, a broad peak around 3400-3200 cm^{-1} indicated O-H stretching vibrations in cellulose and lignin.

In the Wood Powder/Glass Powder/PP Composites, FTIR analysis revealed a mixture of peaks originating from wood and glass components, alongside those from the polypropylene matrix. Specifically, the hybrid composite exhibited a distinct O-H stretching bond functional group ranging approximately from 2950-2917.28 cm^{-1}. Additionally, a peak at 2155.7 cm^{-1} indicated the presence of hemicellulose. Moreover, the spectrum showed peaks spanning from 1458-975.42 cm^{-1}, suggesting the presence of lignin. The FTIR findings are consistent with previous studies conducted by Cazon et al. [14], Fu et al. [15], Arguello et al. [16] and Ferreira et al. [17].

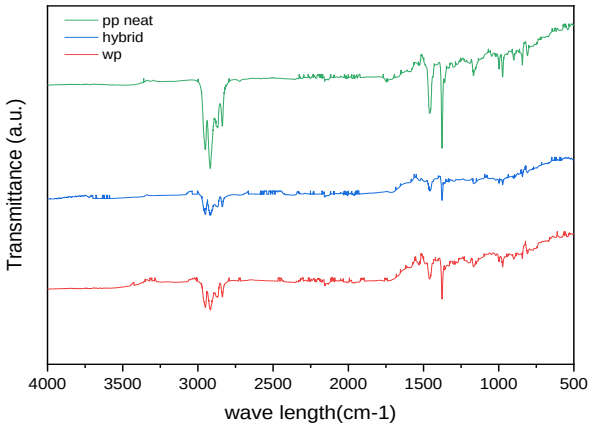

Figure 1: FTIR peaks of hybrid composites, wood powder/ PP composites (WP), and Neat polypropylene (PP)

TGA

The thermal decomposition behaviour of neat PP, wood powder/PP composite (WP), and hybrid composites were investigated using TGA, as depicted in Figure 2a, revealing a multi-stage degradation process. Initially, weight loss was observed at 300°C for both wood powder/PP composites and hybrid composites, while neat polypropylene exhibited this at 350°C. This initial weight loss in all samples is attributed to moisture evaporation. The second stage of weight loss occurred between 490°C and 510°C for all composites, with wood powder/PP composites losing 85% and hybrid composites 60% of their weight. Neat polypropylene experienced complete degradation with a 100% weight loss during this stage. The third stage showed a plateau in all

samples, indicating complete decomposition, leaving behind ash or char residue.

The weight loss up to 180°C in both wood powder/PP composites and hybrid composites is primarily due to moisture evaporation from the fillers. Subsequent stages involved the degradation of cellulosic components between 200°C and 550°C, with hemicellulose decomposing around 400°C and lignin degradation spanning a wide temperature range starting below 200°C. Cellulose degradation concluded around 500°C, followed by the final phase of lignin degradation. These observations are consistent with previous studies by Li et al. [18], Jamahat et al. [19], and Zorah et al. [20].

Figure 2(b) presents the Derivative TGA (dTGA) Curves for neat polypropylene (PP), wood powder/PP composites (WP), and hybrid composites. The dTGA curve for neat PP illustrates the rate of weight loss over temperature, with peaks indicating maximum degradation rates. The peak at 450°C corresponds to a dTG weight loss of 20% for neat PP. For wood powder/PP composites, the dTGA curves highlight differential degradation rates between the polypropylene matrix and wood powder filler, with a peak degradation rate also at 450°C, corresponding to a dTG weight loss of 15%. Similarly, for wood powder/glass powder/PP hybrid composites, the dTGA curves show differential degradation rates among polypropylene, wood powder, and glass powder constituents, with a peak degradation rate at 450°C and a dTG weight loss of 12.5%. These findings are consistent with those of Li et al. [18], Jamahat et al. [19] and Zorah et al. [20].

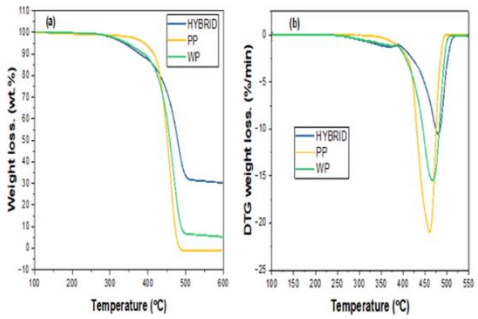

Figure 2: (a) TGA traces of the percentage weight loss as a function of temperature (b) derivative TGA (dTGA) curves.

DSC

Figure 3(a) depicts the melting temperature thermograms for the composite samples under study. Clear peaks indicate both endothermic and exothermic reactions, corresponding to phase transitions and reactions occurring within the samples. Pure polypropylene (PP) shows a distinct melting peak around 150ºC, indicating its transition from solid to liquid phase. Additionally, the glass transition temperature for pure PP is approximately -20ºC. In wood powder/PP composites (WP), thermograms exhibit melting temperature peaks at around 150ºC, reflecting both the PP matrix and any wood powder filler present. For wood powder/glass powder/PP hybrid composites, the melting temperature peaks represent the melting behaviour of all constituents namely PP, wood powder, and glass powder, typically around 155ºC. The glass transition temperatures for these composites range from 10ºC to 60ºC. The observed behaviour in Figure 3(a) is attributed to the disruption of hydrogen bonds, leading to a decrease in cohesive energy. These findings align with studies by Panaitescu et al. [21] and are consistent with those reported by Bay et al. [22].

Figure 3(b) displays the cooling (crystallization) temperature thermograms of the composite samples. For all composite samples, the initial peak around 120°C indicates the relaxation of crystalline regions. In hybrid samples, a second peak is observed around 180°C, indicating the melting of these crystalline regions, while a third peak appears at 200°C, suggesting decomposition. These findings are consistent with those reported by Zorah et al. [20], Panaitescu et al. [21] and Bay et al. [22].

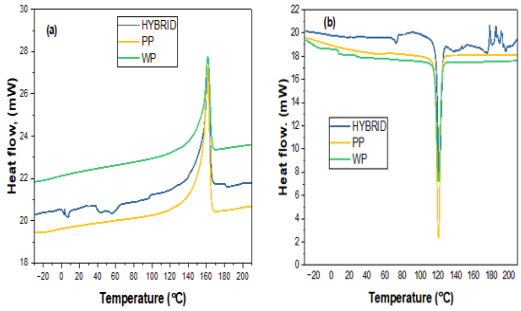

Figure 3: (a) Melting temperature thermograms and (b) Cooling (crystallization) temperature thermograms.

Impact Test Results

Impact testing is pivotal in assessing a material's ability to endure sudden loads or shocks, offering valuable insights into its toughness, resilience, and energy absorption capabilities during impact events. These tests are essential for predicting how

materials will perform in practical scenarios such as automotive crashes, structural collisions, and other impact-intensive applications. The results of impact tests provide critical data on how materials respond to sudden loading, including key parameters like impact strength. Detailed impact strength results for wood powder/PP composites are presented in Table 1 and plotted in Figure 4.

Table 1: Wood powder/PP Composite Impact Strength Results

Fibre Volume Fraction (υf %)	FEA-Results (MPa)	Experimental-Results (MPa)	Error %
0.000	105.36	107.40±15.52	1.90
10.000	96.23	94.69±2.53	1.63
20.000	87.51	86.81±1.61	0.81
30.000	81.52	78.83±0.55	3.41
40.000	75.33	74.18±2.34	1.55
50.000	61.12	60.22±0.40	1.50

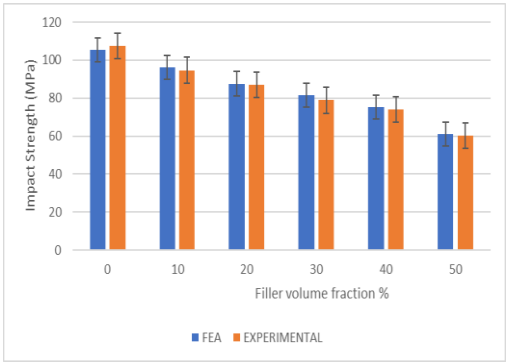

Figure 4: Impact strength Plots for wood powder/ PP composites

The impact strength results show a clear trend: as the filler volume fraction (%) increases from 0% to 50%, both the Finite Element Analysis (FEA) and experimental outcomes demonstrate a gradual decrease in impact resistance. This decline suggests that higher concentrations of wood powder filler lead to reduced ability of the composite material to effectively withstand impact forces.

From a scientific perspective, this trend can be attributed to several factors inherent to composite materials. Initially, adding fillers like wood powder to PP can reinforce the composite, enhancing stiffness and certain mechanical properties. However, as the filler content increases, it introduces challenges such as stress concentration around filler particles and limited polymer chain mobility. These factors collectively diminish the composite's ability to absorb and dissipate energy during impact, thereby lowering its impact strength [1,17].

Regarding the error analysis, the percentage errors range from 0.81% to 3.41%. These errors reflect the disparity between the impact strength values predicted by FEA and those observed in experimental tests. While discrepancies exist, they generally fall within an acceptable range (within ±5%). Such variations can arise due to variations in material properties or processing, and inherent variability in experimental testing procedures. These findings are consistent with those of Ferreira et al. [17].

The impact test results for wood powder/glass powder/PP composites are detailed in Table 2 and illustrated in Figure 5.

Table 2: Wood powder/Glass powder/ PP Composite Impact Results

Fibre Volume Fraction (υf %)	FEA-Results (MPa)	Experimental-Results (MPa)	Error %
0.000	105.361	107.40±15.52	1.90
10.000	100.36	102.44±0.18	-2.03
20.000	97.99	96.81±1.15	1.22
30.000	86.23	84.08±1.15	2.56
40.000	80.56	75.21±0.34	7.11
50.000	69.80	70.87±0..50	-1.51

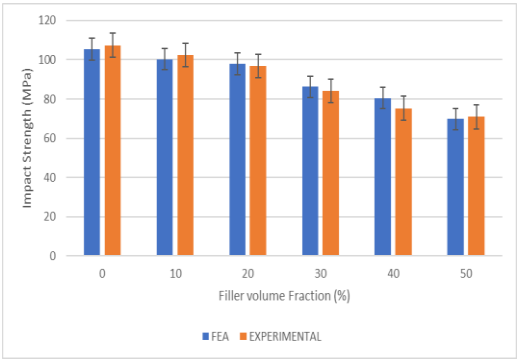

Figure 5: Plot of Impact strength results for wood powder/glass powder/ polypropylene composites.

The impact strength results shown in Table 2 and Figure 5, show a consistent trend as the filler volume fraction (% υf) increases from 0% to 50%. Both the Finite Element Analysis (FEA) predictions and experimental measurements indicate a gradual decrease in impact resistance. This trend suggests that higher concentrations of wood powder and glass powder fillers lead to a reduction in the composite material's ability to withstand impact forces effectively.

Scientifically, this decline in impact strength can be attributed to several factors inherent to composite materials. Initially, the addition of fillers like wood powder and glass powder enhances the

composite's stiffness and certain mechanical properties. However, as the filler content increases, it introduces challenges such as increased stress concentrations around filler particles and reduced polymer matrix ductility. These factors collectively impair the composite's ability to absorb and dissipate energy during impact events, resulting in lower impact strength values [17].

Regarding the comparison between FEA and experimental results, while there is generally good agreement in the overall impact strength trend with varying filler volume fractions, there are discrepancies in specific numerical values. The percentage error analysis reveals deviations ranging from -2.03% to 7.11%. This indicates that in some cases, the FEA predictions slightly overestimated or underestimated the experimental results. Such discrepancies can arise from various factors including variability in material properties, and uncertainties in experimental testing conditions. These findings are consistent with those of Ferreira et al. [17].

ANOVA analysis of the impact strength results is presented in Tables 3 and 4.

TABLE 3:SUMMARY

Groups	*Count*	*Sum*	*Average*	*Variance*
Mono(FEA)	6	507.07	84.51	244.28
Mono (Experimental)	6	502.13	83.69	271.56
Hybrid (FEA)	6	540.30	90.05	183.70
Hybrid(Experimental)	6	536.81	89.47	224.39

TABLE 4:ANOVA

Source of Variation	*SS*	*df*	*MS*	*F*	*P-value*	*F crit*
Between Groups	195.2	3	65.07	0.28	0.84	3.10
Within Groups	4620	20	230.98			
Total	4815	23				

The ANOVA analysis explores the impact strength differences across two types of composite materials: Mono composites, consisting of wood powder and polypropylene (PP), and Hybrid composites, which include both wood powder and glass powder in a PP matrix. Based on the ANOVA results, which compare impact strength between Mono (wood powder/PP) and Hybrid (wood powder/glass powder/PP) composites, there is no statistically significant difference in mean impact strength values between these groups. The analysis suggests that the observed differences are likely due to random variability rather than a systematic effect of composite type on impact strength.

Scanning Electron Microscopy

SEM analysis was conducted on both the fractured and surface sections of various composite samples, depicted in Figures 6-11. Examination of the SEM images reveals subtle differences in morphology among neat Polypropylene (Figures 6-7), wood powder/PP composites (Figures 10 & 11), and hybrid composites (Figures 8 & 9), all exhibiting rough surfaces. Specifically, SEM images of the wood powder/PP composites show clusters of wood powder, indicating inadequate dispersion that could potentially impact mechanical properties [23, 24]. Additionally, hollows and randomly dispersed powder particles and fibers are evident, suggesting weak adhesion between the PP matrix and wood powder, likely due to the hydrophilic nature of wood powder contrasting with the hydrophobic nature of PP.

In contrast, fracture surfaces of hybrid composites display roughness with numerous detachments or pull-outs from the polypropylene matrix. Importantly, in terms of surface morphology, hybrid composites exhibit fewer pull-outs compared to wood powder/PP composites, indicating better filler compaction within the polypropylene matrix of hybrid composites. This observation provides a plausible explanation for the superior mechanical properties observed in hybrid composites compared to wood/PP composites.

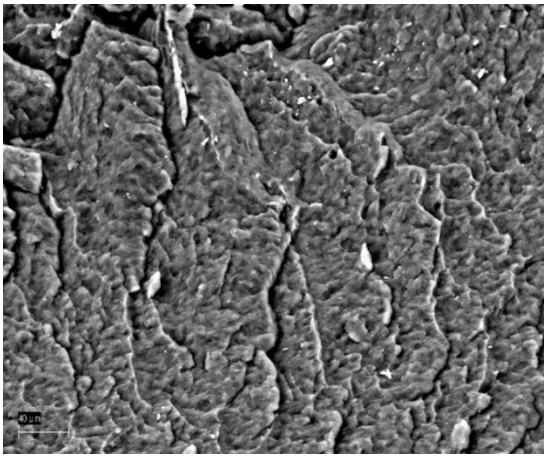

Figure 6: Neat Polypropylene (Fractured sample)

Conclusions

The impact toughness of wood powder/PP composites and wood powder/glass powder/ PP composites decreases with increase in filler additions.

The impact toughness of the hybrid composites was higher than those of the mono composites.

SEM images of the wood powder/PP composites show clusters of wood powder, indicating inadequate dispersion that potentially impacted mechanical properties.

In terms of surface morphology, hybrid composites exhibited fewer pull-outs compared to wood powder/PP composites, indicating better filler compaction within the polypropylene matrix.

From the dynamic scanning calorimetry, it was noted that the glass transition temperature of neat polypropylene was -20ºC, while those of wood powder/PP composites and wood powder/glass powder/ PP composites were in the range 10ºC to 60ºC.

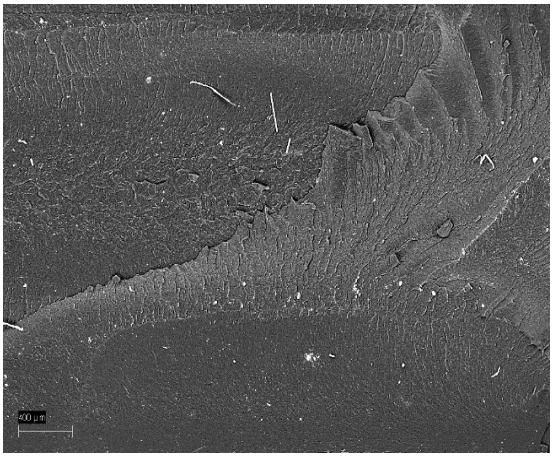

Figure 7: Neat polypropylene (Fractured sample)

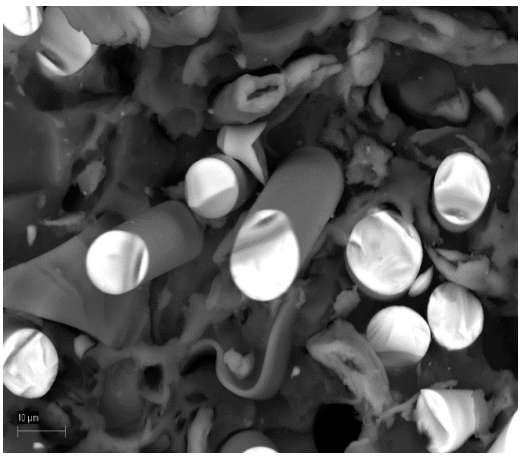

Figure 8: Hybrid composite (Fractured sample)

Declarations

Availability of data and Material

Every piece of data produced in this investigation is encompassed within this published article

Competing Interests

There is none to be declared

Funding

There is none to be declared

Figure 9: Hybrid composite (Surface sample)

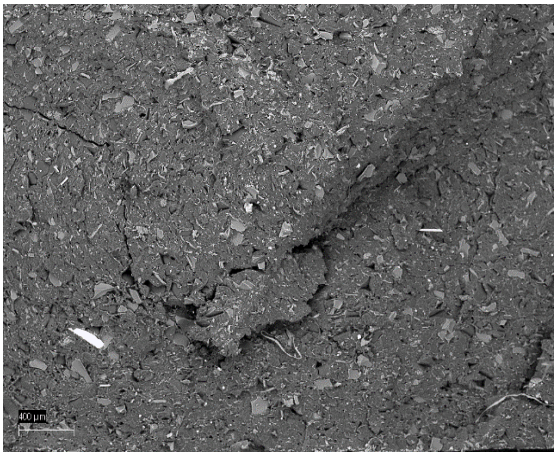

Figure 10: Wood powder/PP composite (Surface sample)

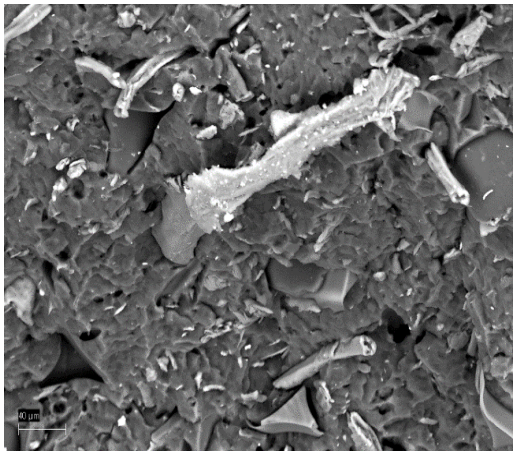

Figure 11: wood powder/PP composite (Fracture sample)

Authors' Contribution

Each author made an equal contribution to the accomplishment of this publication.

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