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Mechanical Properties of Highly Filled PVC/Wood-Flour Composites

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ABSTRACT: The present article evaluates the effect of variation of WF on the tensile strength, modulus, and elongation at break. The pin-bearing properties usually correlate well with tensile properties. Tensile strength and pin-bearing strength decrease but tensile modulus increases on addition of WF. The tensile strength, pin-bearing strength, and mode of pin-bearing failure also depends on the void content. Increase in void content has a detrimental effect on all the mechanical properties. Wide variation is observed in impact strength because of unpredictable void content due to use of non-vented extrusion process. TGA indicates insignificant effect of WF on thermal stability of composites. Infrared spectroscopy indicates some interaction between PVC and WF.

KEY WORDS: polymer–matrix composites, mechanical properties, statistical properties extrusion, thermogravimetric analysis, FTIR.

INTRODUCTION

INCORPORATION OF WOOD flour (WF) and wood fiber as reinforcement in thermoplastic matrix has been gaining popularity in the last few years. WF and wood fiber are available in abundance and at low cost. Wood plastic composites (WPC), as they are called, offer advantages of high stiffness-to-weight ratio and low cost over conventional reinforcements, which justify their use [1,2]. Various thermoplastic matrix materials used include polyethylene (PE), polypropylene (PP), and polyvinyl chloride (PVC). PE, which has the major market share, is used in decking applications. PP is predominantly used in automobile industry while PVC is used in furniture applications [3]. PVC, which has been used as matrix material in the present experimental work, is commonly used for furniture applications because of its good mechanical properties, chemical resistance and water resistance, low flammability, and low cost. Also, PVC-based wood composites can be cut, sawed, screwed, and nailed like standard wood materials using conventional tools [4]. One of the major issues with PVC/WF composites, like other WPCs, is the incompatibility between the hydrophilic WF and hydrophobic plastic matrix. This leads to poor interfacial adhesion, leading to poor dispersion and reduction in mechanical properties. Various compatibilizers have been used for increasing the interfacial adhesion between the matrix material and WF, leading to enhancement in mechanical

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properties [5]. Hristov et al. [5,6] used maleated PP as a compatibilizer for PP/WF composites. Maleated PP improved the adhesion between the matrix and the fibers, forming an interfacial layer [5]. This caused increase in the impact strength and transition from brittle to semi-brittle failure. Shah and Matuana. [7] used chitin and chitosan as coupling agent for PVC/WF composites. Addition of chitin and chitosan enhanced the flexural strength, flexural modulus, and storage modulus. Ge et al. [8] used a silane coupling agent for PVC/WF composites with WF content varying from 10 to 50 phr. Mechanical and thermal properties along with morphology of PVC/WF composites were studied. Tensile strength and strain at break was reduced on addition of WF [8]. Copper-Amine treated WF was used in PVC/WF composites [4]. Un-notched impact strength, flexural strength, and flexural toughness were enhanced as compared to unmodified composites. Djidjelli et al. [9] investigated the effect of addition of 10–30 phr WF to PVC on the mechanical and thermal properties. Stress and strain at break were reduced on addition of WF but the change in T_g is insignificant [9]. Sombatsompop et al. [10] studied the effect of addition of untreated sawdust on mechanical properties. Mechanical properties of the composites reduced up to addition of 16.7% of sawdust, after which they almost remained constant. In the present experimental work untreated WF up to 300 phr has been added to the matrix. Tensile, impact, and pin-bearing properties have been studied. Fourier transformed infrared (FTIR) spectroscopy has been used to assess interaction between PVC and WF. Thermogravimetric analysis (TGA) has been carried out to study the effect of WF on thermal properties.

MATERIAL

PVC: 57 GE R01 grade of suspension PVC manufactured by M/s Reliance India limited, India was used. The grade used, though suitable for injection molding, has been used for extrusion, as WF content being quite high, the required melt viscosity of PVC is considerably low. Wood is primarily composed of hollow, elongate, spindle-shaped cells that are arranged parallel to each other along the trunk of a tree. When lumber and other products are cut from the tree, the characteristics of these fibrous cells and their arrangement affect such properties as strength and shrinkage [11]. Wood comprises primarily four structural components, which include cellulose (approximately 45–50% by weight), hemicelluloses (approximately 20–25%), lignin (approximately 20–30%), and extractives (approximately 5–10%). Cellulose, the major component, constitutes approximately 50% of wood substance by weight. It is a high molecular weight linear polymer, consisting of chains of one to more than four beta-linked glucose monomers with degree of polymerization 5000–10,000. Most of the cell wall cellulose is crystalline. Hemicelluloses have a lower degree of polymerization (150–200) and are relatively straight or branched. Cellulose and hemicelluloses contain free hydroxyl groups that lend wood its inherent hygroscopic property. Lignin constitutes 23–33% of the wood substance in softwoods and 16–25% in hardwoods. Lignin is a large, amorphous polymer consisting of varying ratios of the phenyl propane precursors linked mainly by two or three ether bonds and the rest by C–C bonds. Extractives, also known as extraneous materials, are extractable with neutral organic solvents and water. In general, the extractives are any compound that do not belong to the classes of cellulose, hemicellulose, or lignin [12]. The WF used in this project is from the same variety of wood commercially available in India as *Ghana Teak*, generically *Tactonna Grandis*. WF sieved from 90 mesh size and dried for 8 h at 120°C was used for this experimental work. Tensile strength and modulus of the *Ghana teak* wood were found to be 73 and 9563 MPa, respectively, in fiber direction.

PROCESSING

The WF loading in the experimental work is up to 300% of PVC. Such a high percentage of loading poses problems of poor dispersion of WF in the matrix. Also, WF, with its high bulk factor, will require considerably high power for compaction. The effective density of the composite is a function of compounding of the WF with the polymer and the compaction force during processing. The effective density that can be achieved for WF in a wood/polymer composite after compaction is as high as 1.40 g/cc. [13]. Thus, processing of these composites is an energy intensive process. In the present experimental work, PVC was formulated using a high-speed mixer. WF was added to formulated PVC and the dry blend was compounded using two-roll mill. The hide was reduced in size using a scrap granulator. The granulated material was extruded in the form of sheet using twin-screw extruder attachment of Plasticorder Brabender, model number AEV 651. In the absence of compatibilizers between constituents, the extent of bonding between the constituents is marginal and consequently the different constituents remain separate phases and the resulting strength becomes a function of void content of the composite, which in turn depends on the compaction force during the extrusion. Void content in general will reduce the tensile strength. The nature of the composite and the process, however, will invariably introduce void content and hence it is important to have void content as one of the variables affecting the strength.

EXPERIMENTAL WORK

Void content was calculated using the formula $v_v = \rho_c - \rho/\rho_c$, where v_v = volume fraction of voids, ρ_c = theoretical density, and ρ = actual density experimentally measured. The density was measured using the formula $\rho_c = 1/(w_1/\rho_1) + (w_2/\rho_2)$, where ρ_c = density of the composite, ρ_1 and ρ_2 = density of PVC and WF, and w_1, w_2 = weight fraction of PVC and WF. Limiting density of wood is taken as 1.54 g/cc and density of formulated PVC was measured as 1.399 g/cc. PVC has considerable small particulate size compared to WF (Figure 1). WF is not spherical in shape but has acicular nature with one dimension larger than the other (Figure 2). During the process of extrusion, the dimension in the direction of extrusion becomes even larger due to orientation and compaction force giving it a flake-like structure. Flake-like structure of WF is the principle reason as to why such high loading of WF is possible. FTIR was carried out on a SHIMADZU FTIR-8400 spectrometer. Thermogravimetric analysis was carried out using Toshvin DTG60-H, under nitrogen atmosphere. The temperature range was 30–400°C at the heating rate of 5°C/min). Type IV samples as per ASTM D-638 were used for tensile testing on Instron 4204 machine with a 10 kN load cell and strain rate of 5 mm/min. Pin-bearing strength was obtained by tension testing a pin-loaded hole in a flat specimen on Instron 4204 machine with strain rate of 5 mm/min (Figures 3 and 4). Bearing strength was calculated as per ASTM D 953. The Izod Impact test as per ASTM D256-97, test method A, was carried out using the ZWICK 5102 impact testing machine. The Izod specimens were cut using profile cutting machine with high speed milling cutter to avoid the micro cracks.

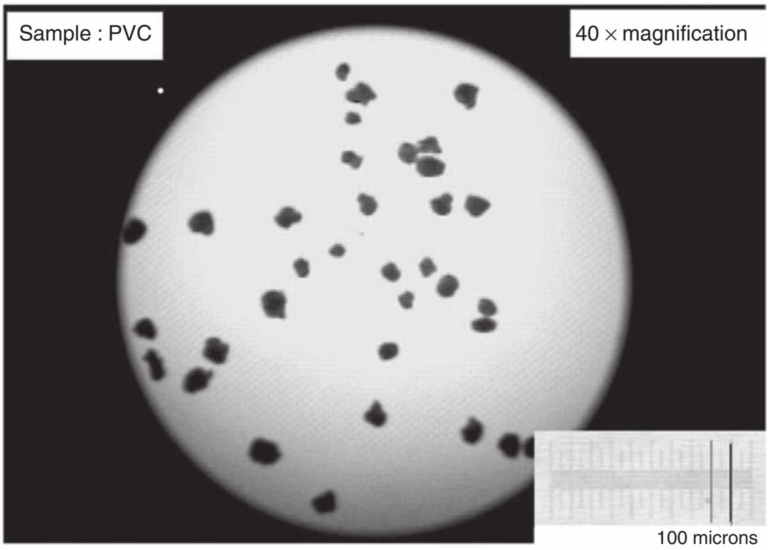


Figure 1. *Micrograph for PVC.*

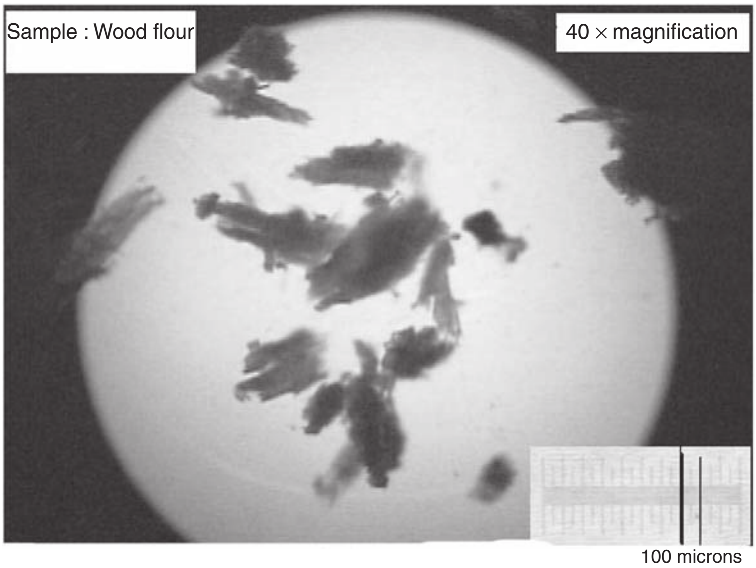
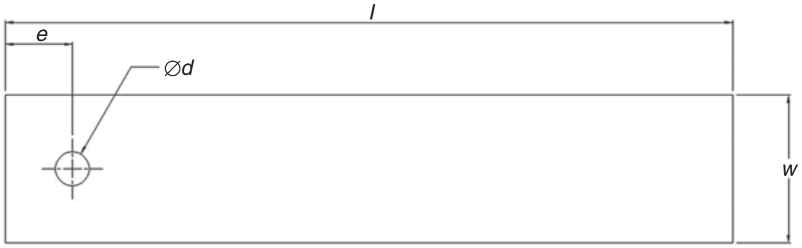
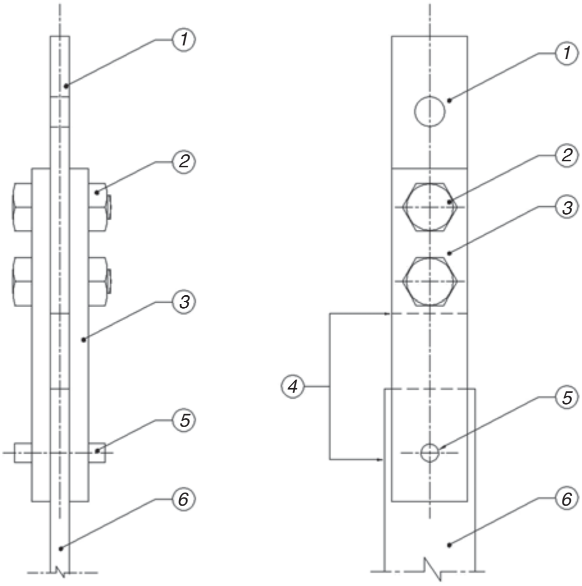


Figure 2. *Micrograph for WF.*



d=Diameter
e=Distance between center of pin hole & dimension of specimen
w=Width
l=Length

Figure 3. *Pin-bearing test sample.*



1-Hardened spacer plate
2-Steel bolts 6.3 mm
3-Hardened side plate
4-Extensometer span
5-Hardened side pin
6-Test specimen

Figure 4. *Fixture for pin-bearing test.*

RESULTS AND DISCUSSION

Infrared Spectroscopy

As revealed by FTIR, the peak at 1724 cm^{-1} in formulated PVC is due to carbonyl group present in the plasticizer and lubricants used during PVC compounding. Slight peak shifting from 1724 to 1726 cm^{-1} is observed on addition of 25% WF to the matrix. Further, on addition of 60% WF, the peak is observed at 1730 cm^{-1}, for 100% WF at 1730 cm^{-1}, 1726 cm^{-1} for 150% WF, and 1734 cm^{-1} for 250% WF. Proton donor–acceptor interaction between PVC and lignin chains have been reported [8]. These interactions occur between –OH and C=O group of lignin and α-H of PVC. With increase in WF percentage, there is increase in lignin content, which leads to more interaction.

Void Content

On addition of WF to PVC, void content increases. WF is hygroscopic and possesses high bulk factor. It is difficult to flow. During processing, the polymer melts and wets the WF. As WF content increases, its wettability decreases. This leads to poor dispersion and increase in void content. Also, voids are caused due to evaporation of the moisture present in WF during extrusion. Extrusion up to 300% of WF could be carried out, albeit, with difficulty. However, extrusion beyond this percentage of WF content could not be carried out as the PVC acting as binder was simply insufficient to hold the WF together. The increase in void content is almost linear (Table 1). The extrusion power is linearly proportional to product of output and the die head pressure. The die head pressure, or the pressure at which extrusion screw works, is related to the compaction force experienced by the material. Void content is inversely proportional to the die head pressure or the pressure at which extrusion screw works. Up to certain percentage, void content increases, as WF is dispersed in phase with PVC matrix after which PVC and WF percentages become close to each other. Further increase in the WF will increase the void content, as PVC becomes a minor component in the WF matrix and dispersion becomes difficult. With 300 WF, due to considerable reduction in binder, i.e. PVC, the extrusion could be carried out at high power and low output causing high compaction and hence void content recorded was low. WF is hydrophilic in nature due to presence of the –OH group. PVC is hydrophobic in nature. During processing, when the polymer is in the fluid form, it wets the WF surface and penetrates the porous structure of wood particles, which renders the WF surface hydrophobic [12]. This affects the moisture absorption of the composites and in turn the void content.

Thermo Gravimetric Analysis

Hemicellulose in wood begins to degrade at 225°C and is completely degraded by 325°C. Cellulose is more stable until 370°C. Only lignin remains stable beyond this temperature [14]. PVC undergoes dehydrochlorination to form polyene sequences between 250°C and 350°C [8,15]. Figure 5 shows the weight loss curves for the composites. Formulated PVC, along with all the composites, shows a single degradation stage in the temperature range 30–400°C. Onset temperature of degradation calculated from the degradation curves is presented in Table 1. The onset of degradation for formulated PVC was found to be 240°C. Addition of WF to PVC marginally decreased the onset temperature

Table 1. Formulations and test results.

Sr. no.	Formulation code	PVC	WF	Onset temperature (°C) from TGA	Void content (%)	Ultimate breaking strength (MPa)	Young's modulus (MPa)	Strain at failure (%)	Bearing strength at 4% hole deformation (MPa)	Maximum strength (MPa)	Type of failure
1	PVC	100	0	240	2.75	37.5	2820	4.50	13.9	76.1	Bearing to tensile
2	25 WF	100	25	238	3.51	31.9	2768	1.75	8.9	76.0	Cleavage to tensile
3	66 WF	100	66	235	5.85	27.5	3909	1.07	20.8	71.7	Cleavage to tensile
4	100 WF	100	100	236	7.28	27.4	4980	0.68	24.9	63.3	Tensile
5	150 WF	100	150	235	7.02	24.9	6084	0.55	27.3	48.3	Tensile
6	233 WF	100	233	236	8.68	20.2	6246	0.53	32.8	46.3	Tensile
7	300 WF	100	300	–	5.15	19.2	6331	0.32	20.1	26.5	Tensile

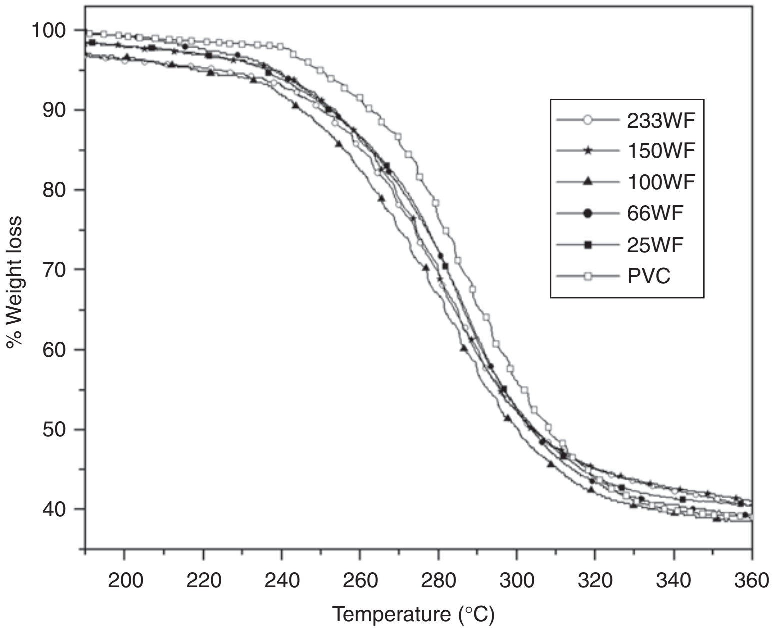


Figure 5. *TG spectra for all composites.*

to 236°C for 233 WF. This can be attributed to lower decomposition temperature of hemicellulose. In general, TGA indicates insignificant effect of WF on thermal stability of composites.

Tensile Strength

Formulated PVC shows ductile failure with tensile strength 37.5 MPa and 4.5% strain to failure (Table 1). On addition of WF, the failure mode immediately shifts from ductile to brittle (Figure 6). This is due to the limitations imposed on the molecular movement by the WF. The limiting loading of WF works out to be 300% of PVC and at 300% WF, the composite yields a value of 24.3 MPa as ultimate breaking strength, and strain at failure was as low as 0.0032. It is interesting to note that strain at failure drops down immediately to a low value with addition of 25% WF (Figure 7). Variation in stress and strain at failure follows a linear pattern. This is largely in agreement with observations of several other authors [8,9,10] Decrease in the tensile strength is attributed to poor interfacial adhesion between the hydrophobic PVC and hydrophilic WF. The modulus shows corresponding steady increase with increase in WF content (Table 1). Increase in the modulus is due to the restraining effect of filler on the polymer molecules.

Pin-bearing Property

Formulated PVC shows bearing failure with 76 MPa pin-bearing strength and 1.5 mm pin hole deformation. As there is no filler present in formulated PVC, there is no chain stiffening and the chains can take up all possible configurations on application of stress.

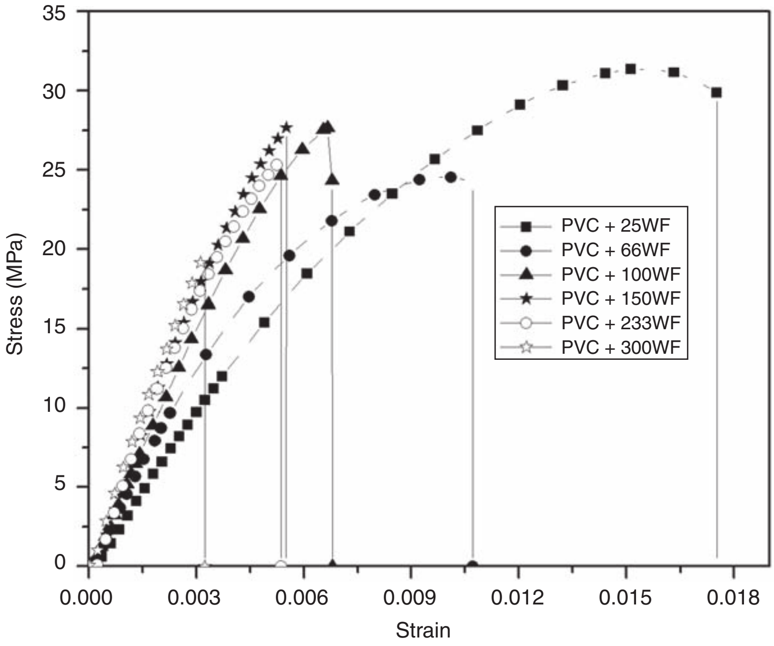


Figure 6. *Stress–strain curves for all composites.*

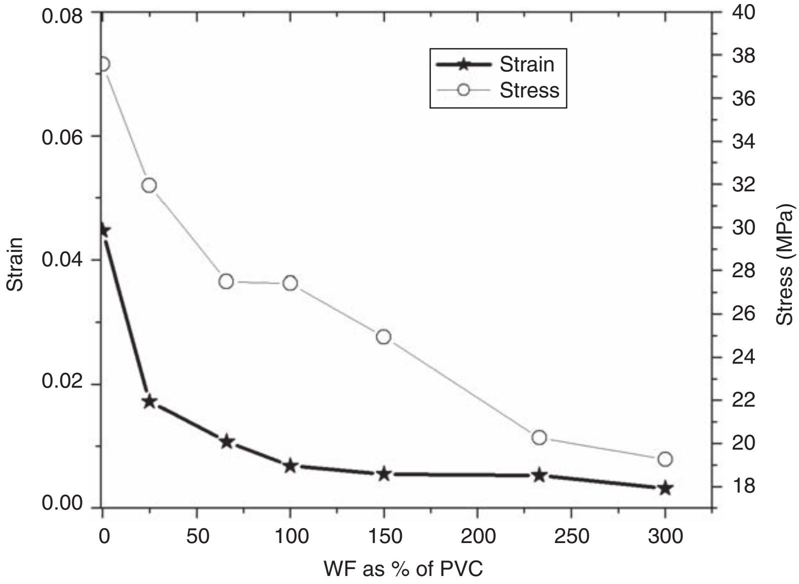


Figure 7. *Effect of WF content on stress and strain at break.*

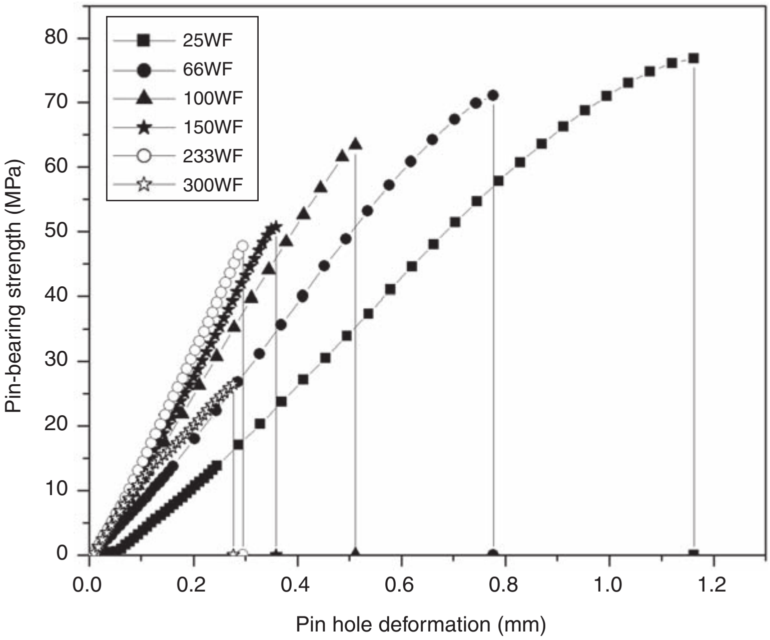


Figure 8. *Pin-bearing strength vs. pin hole deformation for all composites.*

The tensile modulus of PVC is low (Table 1). This results into greater deformation of the pinhole before complete failure. Bearing strength, i.e., strength at 4% hole deformation, increases with increase in WF content due to immobilization of chains (Figure 8). As WF content increases, the degree of immobilization also increases [16]. Therefore, to attain the same deformation, force required is higher. This results in higher bearing strength. The maximum bearing strength remains almost constant up to 66% loading (Table 1). Decrease in the maximum strength on further WF loading is observed. This can be attributed to the fact that, up to 66% WF loading, there is enough binder material for complete wetting. This ensures effective stress transfer. Further addition of WF leads to decrease in the maximum strength due to insufficient binder. At least four modes of failure are possible with pin-bearing test. Mode of failure for pin-bearing tests depends on the geometry and inherent material properties [17]. The geometry of sample has not been varied. Therefore, the type of failure is only dependent on the inherent material property. Composites with directional properties show cleavage failure. Cleavage failure occurs due to little or almost nil transverse orientation of the filler. This mode of failure is an indicator of the possible filler orientation in the extrusion direction. This also is an indication of orthotropy of the material or the difference in modulus in longitudinal and transverse direction. It can be safely concluded that the transverse modulus of the composite showing cleavage failure will be lower than the longitudinal modulus. From bearing failure observed in PVC, the failure mode shifts to cleavage with addition of WF content up to 66% and then shifts to pure tensile failure (Table 1). The result is in agreement with the tensile testing result where the strain to failure reduces from 4.5% in case of PVC to 1.07% in case of 66% WF loading, and then suddenly drops with further addition of WF, indicating brittleness. The modulus also shows corresponding increase with change in mode of failure.

Table 2. Impact strength.

Sr. no.	Formulation code	Impact strength in J/m (Weibull distribution)	Shape parameter	Location parameter	Variance
1	PVC	31.34	6.91	35.39	5.26
2	25 WF	28.47	5.90	30.62	5.50
3	66 WF	30.63	13.57	31.77	2.75
4	100 WF	27.09	9.24	28.48	3.49
5	150 WF	33.01	1.79	37.14	3.12
6	233 WF	23.58	11.33	24.57	2.51
7	300 WF	19.15	10.07	20.05	2.27

Impact Strength

Variance of the readings of impact test is large as can be seen from the lower values of shape parameter, α of Weibull distribution (Table 2). This can be attributed to the fact that void content shows wide variation in the extrusion process. The variation of void content could be due to use of non-vented extruder. With increase in WF content in PVC, impact strength is expected to reduce. This is because WF, which is filler, acts as an impurity in the matrix. In absence of a coupling agent stress transfer between matrix and filler does not take place, resulting in decrease in impact strength. The results obtained showed strong dependence on void content along with WF content.

CONCLUSIONS

The results of tensile and pin-bearing tests show strong dependence on WF content. Impact results, however, show wide variation, which can be attributed to inconsistency in the void content during extrusion process. Even in absence of coupling agents, interaction between WF and PVC is seen in the FTIR spectra. TGA results indicate insignificant effect of WF content on the thermal stability.

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