Mechanical and Rheological Characterization of Efficient and Economical Structural Wood-Plastic Composite of Wood and PVC

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Summary: Light weight materials and specially composites are being investigated to replace metallic materials. In this research work, fiber reinforced composites are fabricated and investigated. Wood-Plastic Composites (WPCs) are developed which contain cellulosic fiberous-wood as fiber and Poly(vinylchloride) PVC thermoplastic as matrix materials. A compatibilizer was developed to make better adhesion between hydrophobic PVC matrix and hydrophilic wood fibers. Different grades of WPC were manufactured by varying the relative percentages of PVC and cellulosic wood fibers. WPC's were characterized mechanically and rheologically by employing Universal Testing Machine (UTM), impact tester, Melt Flow Index (MFI) tester and melting point apparatus. Tensile mechanical testing showed that mechanical strength was improved with increasing fiber concentration but to a limit of 50 weight % fibers. Rheological testing also illustrated that the tackiness increased by increasing fiber contents.

Keywords: Wood-Plastic Composite; PVC; Cellulosic fiber; Mechanical strength; Rheological characterization.

Introduction

Fiber reinforced composites (FRC's) are low density materials. Their high strength-to-weight and high modulus-to-weight ratios make them superior over metallic materials, so FRC's are replacing metals [1]. Lumber (wooden plank or beam) have been used in structures (decks, rails etc) for so long but its outdoor environmental degradation requires such materials which are inexpensive as wood but they can perform better than wood. So, introducing wood fibers to the plastics, the novel materials of Wood-Plastic Composites (WPC) could be developed. Economic considerations lead to different designs/formulations of WPC. Wood flour (cereal) is inexpensive as it is waste for furniture manufacturers. Increasing the inexpensive reinforcement in plastic matrix makes it economical as well as efficient. Wood-plastic composites are being used in structural applications because of their improved fatigue strength [2, 3]. In WPC, wood fibers bear the load which is dispersed by plastic resin. WPCs are considered as environment friendly green composite as wood composite can be recycled [4, 5]. Inexpensive WPC can be fabricated using recycled profiles of WPC with virgin wood and plastic resin. PVC and wood are used as matrix and reinforcement materials respectively. Effective utilization procedures of wood cellulosic fibrous material have been developed along with developments in the processing techniques of thermoplastic resins.

Wood flour is natural material and it contains 42-44% cellulose, 26-32% hemicelluloses, 18-29% lignin and minerals for soft & hard wood but percentages change with tree characteristics. Diameter of cellulose fiber is between 3-5µm. Cellulosic fibers improved structural properties of a composite with decreased contraction and coefficient of thermal expansion [6]. Furthermore, wood flour plays an important role in context that wood is inexpensive, abundant and sustainable.

Poly(vinyl chloride) PVC is thermoplastic material. PVC is selected as resin or matrix medium because PVC has higher flexural strength, flexural modulus and compressive strength as compared to High Density Polyethylene (HDPE) and Polypropylene (PP) [6]. Comparison of properties is given in Table-1.

Table-1: Comparing Mechanical & thermal properties of rigid PVC with HDPE and PP [6-10].

	Flexural strength (MPa)	Flexural modulus (MPa)	Compressive strength (MPa)	Coefficient of thermal expansion-contraction $x10^{-5}$ 1/°C
Rigid PVC	41-110	2413-4136	76	5-14
PP	41-48	1138-1724	46	4-13
HDPE	10	862-1654	32	4-13

PVC enjoys benefit of good chemical and weather properties [7, 8], along with mechanical tensile strength [9], tensile modulus [6], compressive strength [10] and thermal properties [6].

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Thermoplastic matrix and wood fibers are combined with compatabilizer/coupling agent to fabricate Wood-Plastic Composite (WPC). The synergetic effect gives enhanced mechanical and environmental properties than individual materials of thermoplastic matrix and wood fibers. But the composite formed of thermoplastic matrix and wood flour without coupling agent results in poor mechanical performance [11]. This is because of poor interactions of hydrophobic polymer matrix (saturated carbon chains) and hydrophilic wood fibers (cellulosic chains of repeating glucose molecules). So, tri-component coupling agent was prepared to improve adhesion. In which one was base component and two others were attached on either sides. One of side component made bond (covalent bonding, Hydrogen bonding, and Vander-Waal's weak forces) with hydrophobic wood flour and other bonds to hydrophilic PVC polymer matrix. Coupling agent/compatiblizer was synthesized through melt mixing of Poly(styrene) (PS), Maleic anhydride (MAH) and Poly(methyl methacrylate) PMMA.

Composite was also fabricated through melt mixing of PVC, wood flour and coupling agent using internal mixer. Inclusions of coupling agent to the composite not only modify surface properties (morphology) [12] but flow behavior (rheology) and structural (mechanical) properties were also modified. Different composites have been prepared by varying relative percentages of PVC and wood. Processing conditions, amount of compatibilizer and additives were kept constant. Studied mechanical and rheological properties of different composite grades are presented in following research work.

Experimental

Materials

Wire and cable grade Poly(vinyl chloride) was purchased from Chemicals Company Limited (Thailand), wood fiber was collected as waste of furniture manufacturers and sieved to get fibers of < 50 µm mesh size, Poly(styrene) PS, Poly(methyl methacylate) PMMA and Maleic anhydride (MAH) were used as provided by Akzo Nobel ®. Ethylene propylene diene monomer EPDM, clay, short glass fibers and amines were also purchased from Akzo Nobel ®. Table-2 enlists materials used and their role in composite. Liquid amine was added during melt mixing.

Processing

Preparation of coupling agent

Chemicals were used as received and stored under controlled conditions (moisture-chemical free environment). Poly(styrene) PS, Poly(methyl methacrylate) PMMA and maleic anhydride MAH were mixed in melt form in banbury internal mixer (Brabender® Plasticoder).

Temperature control was very important because the shearing action of rotors also produced heat along with built-in heaters. The temperature was adjusted in accordance to glass transition temperatures as given in Table-3. These three materials were mixed in two banbury-shaped rotors/screws at process conditions given in Table-4. Constituents were mixed for 4 minutes so that all three components can make bond with each other. The melt form of coupling agent is separated and cooled to room temperature for later use in composite fabrication.

Fabrication of Wood-Plastic Composite

For composite fabrication, wet impregnation technique was employed [6]. Banbury Internal mixer (Brabender® Plasticoder) was used for composite fabrication. The process conditions are shown in Table-4.

The temperature run was very crucial. It should not increase as high as to degrade wood-fibers. High temperature was controlled by adjusting rpm. Even at that temperature of 160°C the longer retention or process time degraded the wood contents. So, rpm and later time inclusion of wood-flour to the melt was devised to minimize degradation. First composite was manufactured as *Wood-PVC Composite-Without Coupling Agent (WPVCC-WCA).* Each composite contained PVC, wood and additives on weight basis as followed in Table-5

Composite weight = 40 grams

PVC & wood = 80% of 40 grams = 32 grams

Additives*= 20% of 40 grams = 8 grams

*additives include fixed 4 g of C.A**, 1 g of EPDM, 1 g Clay, 1 g SGF and 1 g of amines

** C.A (Coupling Agent), EPDM (Ethylene Propylene Diene Monomer), SGF (Short Glass Fiber)

The recipe of different grades of composite is presented in Table-5

Note: *WPVCC (Wood PVC Composite)*, PVC (Polyvinylchloride)

Processing methodology

At first plasticoder was switched on and temperature was adjusted. Heaters started heating up the walls of mold and the rotors up to fixed temperature. It takes maximum of 5 minutes to gain steady state process conditions. So, after that, at designated rpm the plastic material of PVC was poured. When PVC was melt-like softened then the additives were added. For later time inclusion wood flour was added after 1 minute of operation. Wood flour has low density of 0.19-0.22 g/cm^3, so, higher volumes cannot be added at once. Therefore wood flour was added at regular intervals. For complete impregnation of wood flour internal mixing apparatus ran for one minute after addition of wood flour. The melt mix composites were cooled to room temperature and maintained under controlled conditions to avoid environment effects such as water absorption.

Grinding

The material from plasticoder was in form of small lumps. For further testing it should be in small particle size, for which it was grounded to obtain fine composite material. Grinder had sieve at exit which allowed particles with size less than 100 mesh size.

Table-2 Materials and their functionality in composite

Material	Function in composite
PVC (Polyvinyl chloride)	Matrix
Wood flour	Reinforcement
PS (Polystyrene)	Tri-component
PMMA(Polymethyl methacrylate)	compatibilizer/ coupling agent
MAH (Maleic anhydride)	
EPDM (Ethylene propylene diene monomer)	Plasticizer
Clay	Filler
SGF (Short glass fibers)	Reinforcing Filler
Amine	Heat stabilizer

Table-3: Glass transition and melting temperatures of PS, PMMA, MAH.

Constituents	Tg [°C] (Glass Transition Temperature)	Melting Point [°C]
PS	104	240
PMMA	105	160
MAH	40	53

Table-4 Manufacturing conditions of 1-Composite and 2-Tri-component coupling agent

	Temperature [°C]	Time [min]	rpm*
1	160	4	35
2	100	4	40

**rpm: revolutions per minute*

Testing and Analysis

Mechanical Testing

Standard dumbbell-shaped structures [9] were molded for each of specimen at 180^{0}C using laboratory press (GIBITRE Instruments Srl, Italy). The standard dumbbell structures were conditioned at 20^{0}C ±2 for 48 hrs. Tensile properties were measured via Universal Testing Machine (TIRA test 2810 A.S.T Gmbh Dresdon, Germany). Tensile tests were performed according to ASTM standard D 638. Tensile properties were found with 0.5 mm/min rate of loading. Thickness, gauge width and gauge length of dumbbell specimens were 4 mm, 7 mm and 30 mm respectively as shown in Fig. 1.

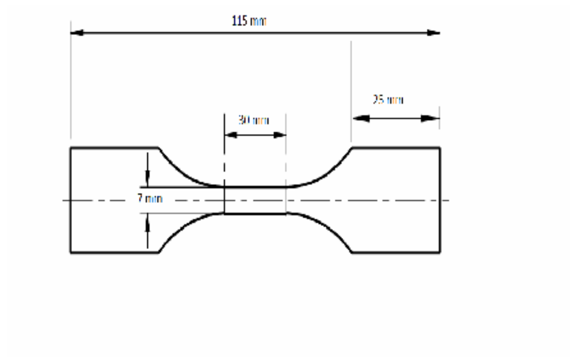

Fig. 1: Dimensions of dumbbell specimen for tensile testing of composite material.

Impact strength was measured through Drop Weight Tubular Impact tester (Sheen Instruments Ltd, England). Test specimens were prepared at 180^{0}C by laboratory press (GIBITRE Instruments Srl, Italy). ASTM D7136 was followed for impact testing.

Rheological Testing

Process-ability of wood-plastic composite was determined by melt flow index (MFI). MFI was measured by melt flow index tester (KARG Industietechnik, Germany). Composite material of 5 grams was filled in barrel of melt flow index tester. Temperature was set at 165^{0}C. Tests were performed in accordance with ASTM D 1238. MFI was calculated by using equation (1) at load of 20 kg.

$$MFI= (600 * W) / T \quad (1)$$

W = average weight of material extruded between standard markings on piston

T = time of material extrusion
600= constant for 10 minutes

Table-5: Recipes of different composites.

Composite	PVC (gms)wt %	Wood (gms)wt %	C.A+EPDM (gms) wt %	Clay+SGF (gms)wt %	Amines (gm)wt %	Total (gms) Total wt %
WPVCC-1	24	8	4+1	1+1	1	40
	60	20	10+2.5	2.5+2.5	2.5	100
WPVCC-2	20	12	4+1	1+1	1	40
	50	30	10+2.5	2.5+2.5	2.5	100
WPVCC-3	16	16	4+1	1+1	1	40
	40	40	10+2.5	2.5+2.5	2.5	100
WPVCC-4	12	20	4+1	1+1	1	40
	30	50	10+2.5	2.5+2.5	2.5	100
WPVCC-5	8	24	4+1	1+1	1	40
	20	60	10+2.5	2.5+2.5	2.5	100

Note: WPVCC (Wood PVC Composite), PVC (Polyvinylchloride), C.A (Coupling Agent), EPDM (Ethylene Propylene Diene Monomer), SGF (Short Glass Fiber)

Results and Discussion

Tensile Test Result

The wood-PVC composite containing 30% wood was fabricated without coupling agent and tested accordingly. Poor mechanical properties achieved were in accordance with the literature [12-16]. These results could be visualized by tensile test. Fig. 2 and Table-6 summarize the tensile test results of coupling agent effect.

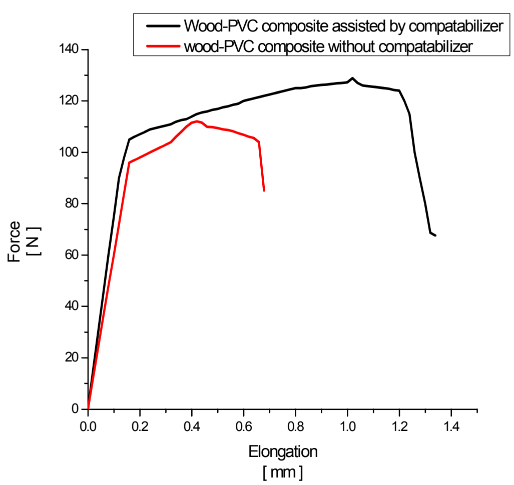

Fig. 2: Tensile test graphs of WPVCC-WCA and WPVCC-1.

Table-6 Effect of coupling agent by comparison of properties

Sample	Maximum Force(N)	Tensile Strength (MPa)	elongation at break (mm)
WPVCC-WCA	112.04	3.46	0.73
WPVCC-1	128.94	4.15	1.33

In presence of coupling agent better mechanical properties were achieved [16]. It is clear that tensile strength and elongation of composite were improved due to bonding nature of coupling agent. Usually the mechanical integrity is provided by reinforcement and dimensional elasticity by matrix. But in absence of coupling agent neither wood reinforcement imparted mechanical aid (low tensile strength) nor PVC matrix provided dimensional flexibility (low elongation). The low properties of WPVCC-WCA are attributed to the poor interactions between hydrophilic wood fibers and hydrophobic thermoplastics [12, 14-16]. Results indicate that the maximum force (N), overall extension at maximum force (%), elongation at maximum force (mm) and overall extension at sample break (%) were better when coupling agent was added. The above experimental results and discussion concluded that coupling agent modified adhesion of matrix and reinforcement resulting in improved structural properties.

All fabricated composites were tested and results are shown in Table-7. For different concentration of wood fiber in PVC matrix different strength and modulus were obtained. Tensile strength and tensile modulus for composites are presented in Fig. 3.

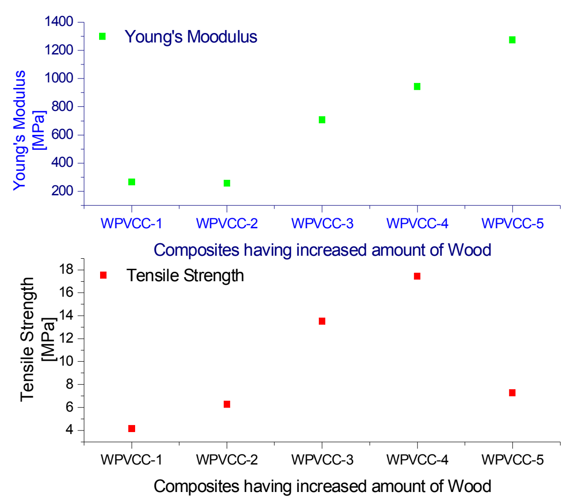

Fig. 3: Tensile strength and Young's modulus of the fabricated composites.

Table-7 Results of Tensile testing of WPVCC 1-5

Sample	Tensile Strength (MPa)	Max. Force(N)	Overall extension at max. force (%)	Elongation at max. force (mm)	Breaking force (N)	Stress at break (MPa)	Overall extension at sample break (%)	Elongation at sample break (mm)	Young's Modulus (GPa)
WPVCC-1	4.15	128.94	1.56	1.02	68.79	2.21	2.04	1.33	0.27
WPVCC-2	6.26	194.27	2.44	1.68	157.72	5.08	2.80	1.93	0.26
WPVCC-3	13.51	438.90	1.91	1.42	438.58	13.50	1.94	1.44	0.71
WPVCC-4	17.44	567.41	1.85	1.26	566.44	17.41	1.86	1.27	0.94
WPVCC-5	7.26	188.56	0.57	0.44	169.64	6.53	0.60	0.46	1.27

Effect of Increasing Wood Fibers Concentration on Tensile Strength

Tensile strength was improved with increasing the concentration of fibers but up to 50 wt %. After which the tensile strength of WPVCC dropped gradually. The maximum tensile strength of 17.44 MPa was achieved for composite containing 50 wt % wood and 30 wt % PVC matrix as shown in Fig. 4. The ability of composite, to withstand higher stresses, comes from the increased amount of fibers (load carrying material in composite). This 50% fiber's concentration is justifiable for matrix to hold them tightly to give better properties as shown in Table-7. But increasing fibers to 60% and above is not acceptable because poor material strength was observed. This is because of increased surface area of fibers which cannot be impregnated or bonded by decreased amount of PVC matrix, and agglomerates of fibers yield lower properties. So we can conclude that better composite was developed at justifiable higher loadings of wood fibers.

Effect of Wood Fibers Contents on Tensile Modulus

The tensile modulus was increased with increasing concentration of wood fibers. As fiber contents increased, the ability of material to bear the load increased. Increment of rigid fibrous material gave stiff composite. Now resultant composite has the capability to absorb high level stresses. Increasing inflexible wood and decreasing flexible PVC plastic matrix also yielded the decreased elongation. Consequently overall ratio of stress to strain (the modulus) increased. Even though WPVCC-5 (wood-PVC 60/20) has highest modulus (1.274 GPa), but its lower tensile strength (7.26 MPa) knocks it out for design considerations.

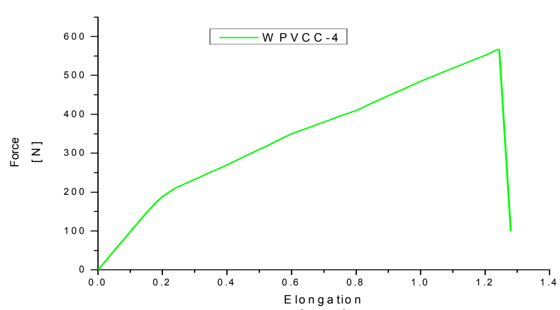

Fig. 4: Wood/PVC composite containing 50% wood.

The stress strain curve for the most efficient but inexpensive WPVCC-4 containing 50% wood and 30% PVC matrix is discussed here. This composite bore maximum force of 567.41 N which was highest among other formulations. Ultimate tensile strength was 17.44 MPa. Likewise higher wood inclusions resulted in decreased elongation of 0.44 mm than 1.26 mm, 1.42 mm, 1.68 mm respectively for WPVCC-4, WPVCC-3 and WPVCC-2.

The necking behavior lasted for very short interval. Then, material fractured right after the ultimate tensile strength. The material did not show long necking time due to the material brittleness that increased with increasing concentration of rigid wood fiber.

Impact Testing

The impact strength of material is its ability to absorb energy from dropped weight before fracture or crack propagation. Before testing the sheets were conditioned for 48 hours at ambient temperature of 20^0C ±2 in controlled conditions. Tubular Drop Weight Impact Tester was used according to ASTM D3029 to calculate impact strength. The standard heights are recorded at which falling weight ruptured/ cracked the composite sheets. The energy released from drop weight is calculated from equation 2.

$$E = mgh - l \quad (2)$$

where E is energy (J) or impact strength, m mass of weight (kg), g is gravitational acceleration (m/s^2), h is height (m), l are frictional losses (J).

Results from impact testing are given in Table-8.

Table-8 The results from impact tester

Sample	Rapture at height (in)	Impact Strength (J)
WPVCC-1	5	1.24
WPVCC-2	4	0.99
WPVCC-3	3	0.75
WPVCC-4	2	0.49
WPVCC-5	2	0.49

The increase in wood content decreased the impact strength. The results were in accordance with the literature [2]. Ductile polymer matrix incorporates better impact strength where weight ratio of polymer matrix to wood is higher. The increasing amounts of discontinuous fibers allow the crack initiation upon stress application making the composite material more vulnerable to impact loadings [2]. Moreover at higher wood concentration the ductility of PVC matrix cannot take part in stress shifting mechanism as de-bonding occurs at fiber-matrix interface resulting poor impact properties. So, increasing fibers to 50 wt % resulted in 66% decreased impact strength. Therefore low impact strength was achieved while keeping the wood at higher level to design inexpensive wood plastic composite.

Rheological Studies

The Melt Flow Indices (MFI) is given in Table-9 calculated at specified conditions. MFI was measured at a fixed temperature of 165°C and constant stress of 2.76MPa (free falling constant area piston with load of 20 kg). During processes of extrusion and injection molding process, the same parameters are kept constant. So, MFI readings are valuable to determine processing conditions of fabrication technique. Decreasing melt flow index was due to increased fiber contents. As MFI is the flow rate of melt with respect to 10 min under constant applied load. MFI describes easiness of material flow Thus, calculated melt flow indices can be used to design the processing conditions (rotor speed, barrel temperature, screw geometry, feeding rate) for fabrication method.

Table-9 Melt Flow Indices of composite samples.

Sample	MFI (at temperature of 165 °C, Load of 20kg)
WPVCC-1	1.54
WPVCC-2	1.30
WPVCC-3	1.01
WPVCC-4	0.89
WPVCC-5	0.76

Conclusion

Conventional pure wood degrades environmentally and wood-plastic composites are replacing the pure wood structures. In current research, the researchers selected PVC as matrix material because of superior PVC properties than HDPE and PP plastics. Natural wood fibers being abundant and sustainable were selected as reinforcement for wood-plastic composite. Using low cost raw materials, the economical wood-plastic composites were fabricated. But costs can further be reduced by increasing concentrations of inexpensive wood fibers compared to other costly synthetic glass, carbon fibers. Research work was done to increase the wood flour contents in composite while achieving best mechanical performance. Conventionally wood flour is used up to 40% but 50% wood contents will make composites more economical. Additionally the composite containing 50% wood performed best among other formulations when characterized mechanically. The suggested PVC-wood proportion resulted in highest mechanical strength and desirable stiffness (modulus) along with preferable percent elongation. Wood plastic composites containing high PVC concentrations performed better under impact loads. Impact strength value was decreased with decreasing ductile PVC. Rheological studies revealed that designing the conditions of fabrication process needs diligent attention and proper consideration was made when composites of higher wood fiber (higher viscosity) were processed. The high viscosity material requires more time to flow but at high temperatures the wood fibers degraded, so, delayed inclusion of fibers to plastic matrix was proposed and employed. Better mechanical properties for high wood contents were made possible by strengthening the poor interactions of hydrophobic PVC and hydrophilic wood. Novel coupling agent was developed comprising Poly(styrene), Poly(methyl methacrylate) and maleic anhydride. Better bonding of ingredients by coupling agent and other additives led to formulate the high-quality performance composite. Concluding that, by proper modifications the most economical and efficient wood-PVC composite can be formulated.

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