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STUDIES ON MECHANICAL, ELECTRICAL AND PHYSICAL PROPERTIES OF RECYCLED HDPE REINFORCED WITH WOODFLOUR NANOCLAY/MICROCLAY

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Abstract
In this project we are using recycled HDPE along with woodflour as particulate filler and nanoclay/microclay to study the performance of these wood plastics composites. Recycled HDPE was melted-blended with wood flour (10%,20%&30%) using the twin screw compounding extruder. Also nano clay (1% and 3%) was added with recycled HDPE containing 30% wood flour and also microclay (1%, 3%, 5% and 7%) was added with nanoclay 3% containing recycled HDPE with 30% wood flour. PE-g-MAH was added as compatibilizer. The test specimens were prepared by injection molding machine as per ASTM standards. The mechanical, electrical, and physical properties were evaluated. The test results of wood plastics composites made with recycled HDPE indicated that the mechanical properties like tensile strength, tensile modulus, flexural strength, flexural modulus, and hardness are improved. The elongation at break and izod impact strength were lowered. Wood flour (30%) reinforced recycled HDPE has higher mechanical properties with optimum impact strength than other formulations with and without nanoclay/microclay. Electrical properties like volume resistivity, surface resistivity, arc resistance and dielectric strength were decreased as the wood flour content was increased. The melt flow index was decreased, density increased. The mechanical properties of wood recycled plastics composites were not affected or improved when nanoclay was used, when microclay was added then the tensile strength was little lowered and other mechanical properties were better than recycled HDPE but lower than wood plastic composites with and without nanoclay.
Keywords: recycled HDPE, wood flour, nanoclay, microclay, twin screw compounding, testing-mechanical, electrical, physical properties.

INTRODUCTION

Now a days the consumption of plastic materials has increased enormously due to their various advantages. The disposal of post-consumer plastic materials here and there in the form of carry bags, packaging films, boxes, etc. causes environmental pollution. Most of the waste plastic materials consist of a substantial amount of PE (polyethylene), PP (polypropylene) and less amount of PVC (poly(vinyl chloride)), PS (polystyrene), PET (poly(ethylene terephthalate)), etc. Recycling and reusing are one of the processes to reduce the environmental pollution caused by post-consumer plastic materials. The use of recycled plastic materials is restricted due to their lower mechanical properties due to degradation of polymer chain during usage. The properties can be improved if the waste plastics are combined with cellulosic materials. The nonconventional plant materials can be transformed into value added products by treating with waste plastics. The forest is rich in cellulose based resources. Eucalyptus is abundantly available in the forests. This plant is suitable for structural applications due to its good mechanical, dimensional and other properties. The saw dust obtained while cutting, polishing from this tree remains as bio-wastes. This saw dust can be utilized for producing composites to be used for various constructional purposes [1].

Wood plastic composites (WPCs) containing wood particulates or wood fibers dispersed in a matrix of polymers such as polyethylene, polypropylene or polyvinyl chloride are commercial products that are increasingly replacing lumber in construction applications, especially in residential decks. It might be recalled that the WPC industry grew out of the desire to recycle polyethylene, coming from applications such as grocery bags and milk containers, into useful products. As the volume of WPCs has grown over the years, the share of virgin polymers has exceeded that of reclaimed plastics in WPC manufacture [2].

Recycling helps conserving materials, but it is known to change the mechanical, physical, and chemical properties of the recycled plastics to some extent. As a result, public perceptions often view recycled plastics as a material of sub-par quality with low economic value. Many industries tend to shun away from recycled plastics because their functional performance may be affected during re-processing. Nevertheless, recycling is still deemed an economically viable mean to conserve and utilize fossil-based polymers in many countries. Deforestation and growing environmental concerns were the main driving forces for the launch of wood-plastic composites (WPC) as a natural wood substitute over two decades ago. Recycling reduces the raw material cost; it can help WPC manufacturers to gain competitive advantage on cost of raw materials while also conserving environment. Hence, recycling of WPC waste is deemed appropriate in terms of economy, utilization and management of natural resource and environment [3].

Wood as a natural renewable material of biological origin has several advantages in many applications ranging from structural, construction materials, furniture, and transport to raw materials for energy production, because of its very satisfactory mechanical and physical properties, its unique microscopic structure, its abundance, its relative ease of work and good visual effect. WPCs are expected to resemble wood in appearance and properties. This necessitates a wood content with a higher value being preferred for reasons of cost. However, high filler amounts result in high viscosity values that lead to processing difficulties, such as excessive extruder torque and the occurrence of melt fracture. Additionally, one observes a reduction in composite strength with increasing filler amount due to poor adhesion between the hydrophilic wood and the hydrophobic polymer. These problems are typically overcome using processing aids and coupling agents. Note, though, that different additive packages are required for different matrix polymers.

A major issue in achieving true reinforcement for wood plastic composites is the inherent incompatibility between the hydrophilic fibers and the hydrophobic polymers which results in poor adhesion and therefore in poor ability to transfer stress from the matrix to the reinforcing fillers. Several investigators have explored the capability of additives to enhance adhesion and thereby improve properties, such as the tensile and flexural strengths of these composite materials. The coupling agent is chosen to form chemical bonds between the cellulose chains in the fiber and the polymer matrix. There has been a lot of research over the past decades on different types of coupling agents to improve the adhesion between the wood and the plastic. The most used coupling agents are maleated polyolefins. However, in the past, neither chemical nor physical methods have significantly improved the interfacial adhesion and thus improve the mechanical properties of WPCs [4].

Composite industries are seeking more environmentally friendly materials for their products. There is an increasing interest in biodegradable renewable composites. The combination of interesting mechanical and physical properties together with their sustainable character has triggered various activities around ‘green composites’. In the other words, green chemistry is the chemical products and processes that reduce or eliminate the use and generation of hazardous substances. A significant goal of green chemistry must be to maximize the efficiency of use of raw materials and to minimize the creation of waste [5].

Plastic waste is one of the major components of global municipal solid waste and, as such, it presents a promising raw material source for WPCs, especially because of the large volume and low cost of this material. By using recycled plastics rather than virgin resin, these wood/recycled plastic composites provide an additional market for recycled plastics, thereby helping to reduce the burden of waste disposal in landfills. Hence, the development of new value-added products using wood waste and recycled plastic is assuming greater importance. The utilization of recycled plastic for the manufacture of WPCs are cheaper than competitive materials, including those made of virgin wood fillers. WPCs can also be fabricated using wood fillers, such as from sawdust (wood flour) from sawmills, providing an additional usage for recycled wood fillers and thus further reducing waste in landfills. Composites containing recycled plastics and wood fillers offer interesting combinations of properties. The mechanical properties of WPCs containing recycled plastics were statistically like those of composites made from virgin plastics. This result is a strong impetus to expand the use of recycled plastics in the manufacture of WPCs. The use of plastic and wood wastes seems inevitable, and the present opportunities are promising [6].

Wood flour is cheaper filler having good hardness and abundantly available from saw industries. Wood flour has lower density. The general characteristics are excellent mouldability, good shock resistance with desirable electrical properties. Also, it has good dimensional stability and waterproof properties. The moulded products can be easily machined. Wood flour is a natural polymer obtained from trees. The trees contain about 50% cellulose which is bio- degradable. Hence the wood flour filled recycled HDPE composites will also be bio-degradable and eco-friendly. Hence, our interest in this present study is the recycled HDPE reinforced with wood flour by using PE-g-MAH as a compatibilizer and evaluate their mechanical, electrical, and physical properties. The PE-g-MAH has reactive maleic anhydride/acid groups on HDPE of the compatibilizer which will react with –OH groups of Cellulosic / Woodflour Polymer molecules and permanent chemical bond forms. Also, the PE part of compatibilizer will be miscible with recycled HDPE polymer chains by cohesively. Finally, this improves the compatibility and reinforcement of wood flour with recycled HDPE. Therefore, the mechanical stress transfer will be better than in virgin HDPE and recycled HDPE. The main objective is to reduce the cost by adding the recycled HDPE and wood flour and to study their mechanical, electrical, physical properties. Polymer composites are physical mixtures of two or more polymers with/without chemical bonding between them. The objective of the polymer composites is practically achieving commercially viable product through either unique properties or reduce the cost of the raw materials [7].

Nanocomposite with layered silicate nanoclays as an in-situ reinforcement has been extensively investigated in recent years. Nanocomposite improves mechanical, thermal, optical, and physiochemical properties remarkably compared to conventional composites. However, far less is known regarding wood polymer nanocomposites [8-10]. Also, in this study we used combination of nanoclay and microclay in optimum composition of 3% nanoclay. Thus 1%, 3%, 5% and 7% of microclay are added to wood flour reinforced R. HDPE composites which is cheaper than nanoclay.

EXPERIMENTAL

Materials

The recycled HDPE material was injection grade, and the melt flow index of this material is 3.74g/10 min, density 0.955 g/cc obtained from Polyelectro, Chennai. The wood flour was obtained from Uniply decor limited in Chennai. Compatibilizer PE-g-MAH of density 0.954 g/cc obtained from Dupont, Delhi. The glycerol and silicone oil were obtained from Ram Chandra Trading Corporation, Chennai. The modified MMT nanoclay (SE 3000) was obtained from Ultrananotech Pvt Ltd, Bangalore. The microclay was obtained from Supreme Chemicals, Chennai.

High speed mixing

The high-speed mixer was used for the Mixing of Wood flour filler in Recycled HDPE as given below after the following paragraph. Compositions were thoroughly mixed with wood flour. The R. HDPE and wood flour are immiscibility nature, so the 2% PE-g-MAH used as the compatibilizer. 1% Glycerol and 1% silicone oil was used as a sticking agent for wood flour with recycled HDPE granules, otherwise while mixing the wood flour with recycled HDPE, wood flour was getting separated from R. HDPE granules. These raw materials were fed into the high-speed mixer and the materials were mixed at the speed of 100 rpm to get proper mix.

Table 1 Composition of samples

S.No	Identity code	Formulation ratio
1	WF0%	R. HDPE(100%):wood flour(0%)
2	WF10%	R. HDPE(90%):wood flour(10%)
3	WF20%	R. HDPE(80%):wood flour(20%)
4	WF30%	R. HDPE(70%):wood flour(30%)
5	NC1%	R. HDPE(70%):wood flour(30%):nanoclay(1%)
6	NC3%	R. HDPE(70%):wood flour(30%):nanoclay(3%)
7	MC1%	R. HDPE(70%):wood flour(30%):nanoclay(3%):microclay(1%)
8	MC3%	R. HDPE(70%):wood flour(30%):nanoclay(3%):microclay(3%)
9	MC5%	R. HDPE(70%):wood flour(30%):nanoclay(3%):microclay(5%)
10	MC7%	R. HDPE(70%):wood flour(30%):nanoclay(3%):microclay(7%)

Twin screw compounding

The high-speed mixed materials were pre-dried for 4hrs at 80^{o}C in oven. The twin screw compounding extruder was used for the direct dry blending [10] of wood flour filler with recycled HDPE. Compositions were thoroughly mixed with wood flour. The R. HDPE and wood flour are immiscibility in nature, so the 2% PE-g-MAH used as the compatibilizer. 1% glycerol and 1% silicone oil were used as a sticking agent for wood flour with recycled HDPE granules. The raw materials mixed with high-speed mixer were fed into the hopper of a twin-screw compounding extruder. In the twin screw extruder, the wood flour and the compatibilizer was blended using the following temperature profile as shown below and screw speed was 100 rpm. The extrude blends material was passed through a cooling water trough and then cut into granules which are used for making test specimens.

Zone temperature of Twin Screw Extruder

Zone	1	2	3	4	5	6	Die Zone
Temperature(^{o}C)	175	180	190	200	200	210	220

Specimen preparation and testing

Test specimen was made as per ASTM D [11] standards by injection moulding machine (Electronica) in CIPET, Chennai. The temperature range in the barrel was 190^{o}C to 260^{o}C with an injection pressure of 60 to 90 bar. The testing was done as per ASTM standards for the following properties.

Mechanical testing

Tensile strength (ASTM D 638), tensile modulus (ASTM D 638), elongation at breaking point (ASTM D 638), Flexural strength (ASTM D 790), Flexural modulus (ASTM D 790), izod impact strength (ASTM D 256) and hardness.

Electrical Properties

Volume resistivity (ASTM D 257), surface resistivity (ASTM D 257), arc resistance (ASTM D 495), dielectric strength (ASTM D 149).

Thermal Properties

Melt flow index (ASTM D 1238).

Physical Properties

Density (ASTM D 792), water absorption (ASTM D 570), shrinkage (ASTM D 955).

The tensile strength and elongation test was done using a UTM (Universal Testing Machine) Lloyd, LR l00k (UK) as per ASTM D638 Type I specimen, using 50 mm/min test speed. The flexural strength test was done as per ASTM D790, using the same UTM, with flexure fixture. The test speed is 1.4 mm/min. The test specimen size 127x12.7x3.2 mm. The izod impact test is done by using an impactometer, Ats Faar m/c (Italy) as per ASTM D256 Standards. Hardness test was done as per using a Digital Rockwell hardness tester (FIE Rockwell Hardness tester, India) with impact/flexural specimens.

MFI test was carried out using a MFI tester (Lloyd Instruments, UK) at 200 Deg C/2.16 Kg. HDT test was carried out as per ASTM D 648 standards using a HDT machine (Ceast, Italy) at 0.46 MPa**.** Density was done as per ASTM D 792 with a small 10x10x3.2mm specimen cut from any impact / flexure specimens. Water absorption was done using 50mm dia disc with thickness 3.2 mm as per ASTM D570 standard. DSC was carried out using Netzsu equipment at Mogappair, Chennai, with 100 mg sample. Heating rate was 20°C/min. Scanning electron microscope (SEM) was taken after gold coating on a small fractured test specimen after applying vacuum drying for 4 h. SEM m/c of Carel Zeiss (UK) Model EVO MA15 was used. FT–IR was recorded with Perkin-Elmer Spectrophotometer. The properties are reported in the Table I to IV and figures 1 to 5. The test specimens were conditioned as per ASTM D 618 at 23±2^{o}C and 50±5 % relative humidity for 24 hrs.

RESULTS AND DISCUSSION

During the twin screw compounding, the recycled HDPE material is reinforced with wood flour material. The improvement in adhesion among plastic materials as well as wood fillers is very important for obtaining better composites. The adhesion can be improved using a suitable compatibilizer. The compatibilizer will be such that it can be able to interact with hydrophilic wood fillers and hydrophobic polymers and at the same time will improve the interfacial adhesion among different thermoplastic materials. In this composite, 2% PE-g-MAH used as a compatibilizer respectively. 1% Glycerol and 1% Silicone oil was used as a sticking agent, the Recycled HDPE and wood flour are not degraded but changed the Pale Yellow colour to brown colour when added the wood flour.

Mechanical properties

The mechanical properties of recycled HDPE reinforced with wood flour blends are shown in Table 2 and 3. The impact strength and elongation at break were decreased. The hardness, tensile sttrength, tensile modulus, flexural strength, flexural modulus of the recycled HDPE reinforced with wood flour was increased when wood flour content was increased and little lowered when nanoclay/microclay incorporated. Figure 1, 2 and 3. was represents about impact strength and hardness, tensile strength, and flexural modulus of these composites. The nanoclay/microclay may be soft materials than wood flour. Wood flour acts as reinforcement and the nanoclay not intercalated with R. HDPE/wood flour matrices, when microclay was used still the strength, modulus was lowered. Impact strength also not improved.

When compared with virgin HDPE, the recycled HDPE will have lower tensile strength since of lower molecular weights, lower flexural modulus, and higher impact strength. But the cost of recycled HDPE is lower by about 50% of virgin HDPE [12].

The wood flour reinforced recycled HDPE is having higher tensile strength, flexural modulus than that of virgin HDPE as well as recycled HDPE and slightly lower impact strength than that of virgin HDPE and recycled HDPE since hardness will be higher (Table 2).

Table 2. Mechanical properties of wood flour filled recycled HDPE.

S.No	Properties	Unit	Test Results			
			R.HDPE100%	R.HDPE+wood flour		
				WF10 %	WF20 %	WF30 %
1	hardness	Shore D	65	67	68	70
2	izod impact strength	kJ/m^2	49.70	10.52	6.30	5.34
3	tensile strength	MPa	24.07	26.17	28.12	31.24
4	tensile modulus	MPa	1222	1414	1558	2143
5	elongation at break	%	114.51	44.89	16.55	8.79
6	flexural strength	N/mm^2	20.05	23.73	28.22	30.88
7	flexural modulus	N/mm^2	395	571	636	1072

ELECTRICAL PROPERTIES

The electrical properties of recycled HDPE reinforced with wood flour blend are shown in Table 4 and 5. The volume resistivity and surface resistivity of these composites was reduced when wood flour content increased. The arc resistance was decreased while added with wood flour and slightly increased when added with nanoclay/microclay. The dielectric strength when added with wood flour it was reduced and the increased when added with nanoclay/microclay. These is due to wood flour has hygroscopic nature.

Figure 4 and 5 was represents about volume resistivity and arc resistance of these composites. Higher arc resistance and dielectric strength were achieved when nanoclay/microclay was added R. HDPE/30% wood flour. The arc resistance and dielectric strength values are higher for microclay filled composites than that of nanoclay filled composites. Wood flour may be easily catching fibre than nanoclay/microclay.

Table 3. Mechanical properties of R.HDPE+30% wood flour with nanoclay/microclay.

S.No	Properties	Unit	Test Results						
			R.HDPE + 30%WF	R.HDPE + 30% WF		R.HDPE + 30% WF + 3% NC			
				NC 1%	NC 3%	MC 1%	MC 3%	MC 5%	MC 7%
1	hardness	Shore D	70	68	67	68	67	66	66
2	izod impact strength	kJ/m^2	5.34	4.93	5.10	4.33	4.17	4.11	4.75
3	tensile strength	MPa	31.24	23.59	24.83	23.02	19.98	18.43	17.49
4	tensile modulus	MPa	2143	2001	2131	2171	1971	1925	1788
5	elongation at break	%	8.79	7.16	6.17	6.59	6.23	6.01	8.89
6	flexural strength	N/mm^2	30.87	24.61	23.19	28.38	26.86	24.21	24.21
7	flexural modulus	N/mm^2	1072	774	740	938	879	832	746

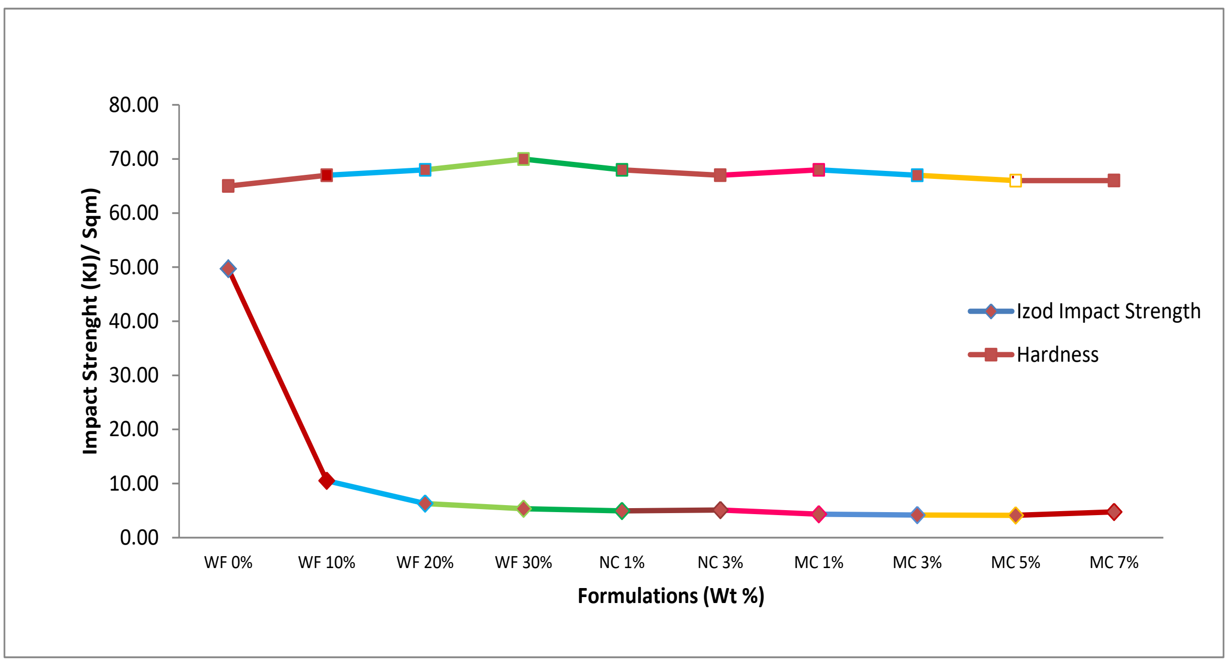

Figure 1. Impact strength and hardness of R.HDPE/wood flour/nanoclay/microclay composites.

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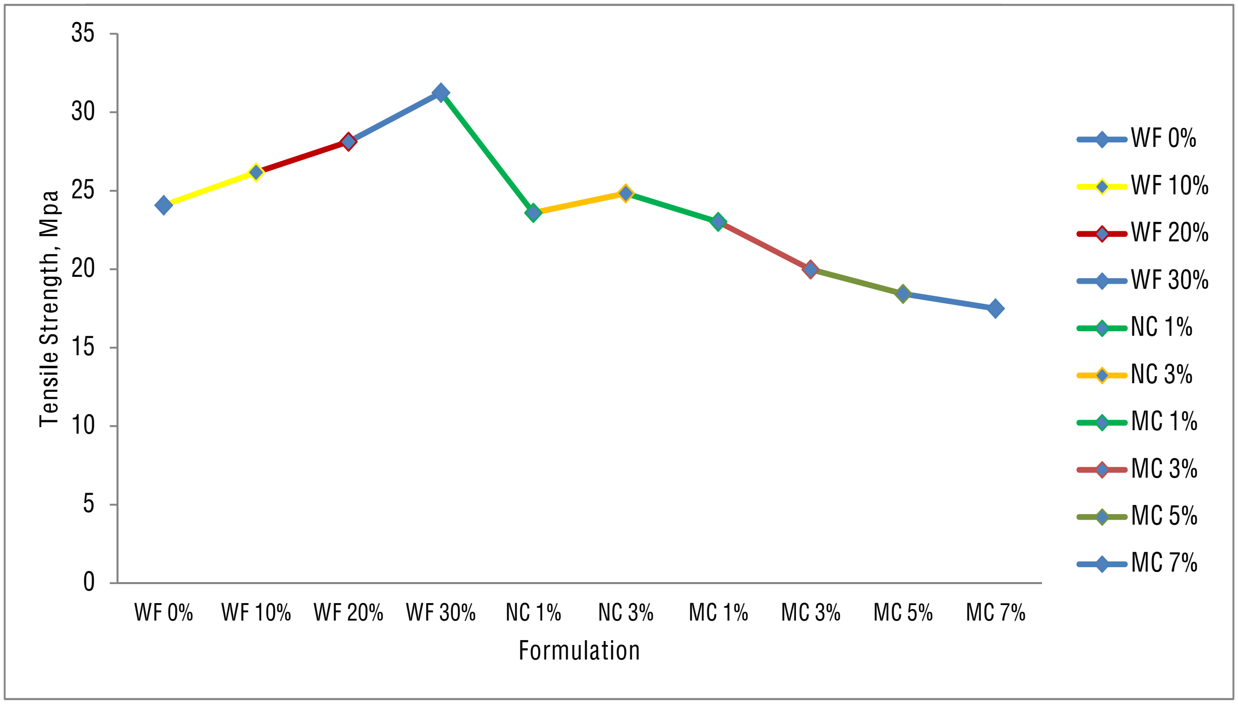

Figure 2. Tensile Strength of R.HDPE/wood flour/nanoclay/microclay composites

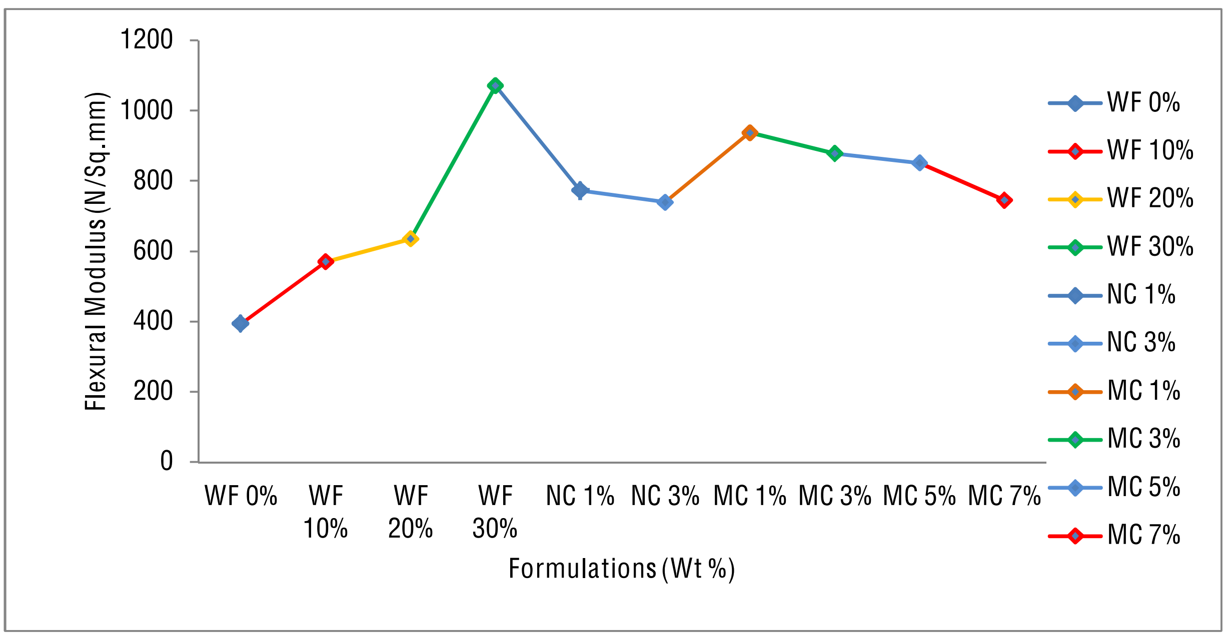

Figure 3. Flexural modulus of R.HDPE/wood flour/nanoclay/microclay composites

Table 4. Electrical properties of wood flour filled recycled HDPE.

S.No	Properties	Unit	Test Results			
			R.HDPE100%	R.HDPE+Wood flour		
				WF10%	WF20%	WF30%
1	volume resistivity	Ohm-cm	30.07×10^{15}	7.23×10^{15}	18.96×10^{14}	33.96×10^{13}
2	surface resistivity	Ohms	53.7×10^{14}	48.33×10^{14}	38.66×10^{14}	21.46×10^{14}
3	arc resistance	Seconds	256	164	148	139
4	dielectric strength	kV/mm	14.47	14.39	13.98	13.30

PHYSICAL PROPERTIES

The Physical properties of recycled HDPE reinforced with wood flour are shown in Table 6 and 7. Melt flow index was reduced when added the wood flour since wood flour is having very high molecular weight. The density was increased than the virgin HDPE. The shrinkage of these composites was decreased due to wood flour content and little increased when added with nanoclay/microclay. The water absorption was slightly increased than virgin HDPE. Because the wood flour has polar nature and it's a hygroscopic material. It can absorb the water. Figure 6 was representing about MFI and density of these composites. When the nanoclay was added the water absorption was little lowered. But when microclay was added it was further decreased. However, all the values are higher than 0.2% and in the range of 0.3 – 0.4%. Hence the R. HDPE wood flour reinforced nanoclay/microclay composites to be pre-dried before processing.

Table 5. Electrical properties of R.HDPE+30% wood flour with nanoclay/microclay

S.No	Properties	Unit	Test Results						
			R.HDPE + 30%WF	R.HDPE + 30% WF		R.HDPE + 30% WF + 3% NC			
				NC 1%	NC 3%	MC 1%	MC 3%	MC 5%	MC 7%
1	volume resistivity	Ohm-cm	33.96×10^{13}	6.57×10^{13}	7.40×10^{13}	1.95×10^{10}	1.96×10^{10}	1.98×10^{10}	2.13×10^{10}
2	surface resistivity	Ohms	21.4×10^{14}	21.4×10^{14}	21.4×10^{14}	21.4×10^{12}	21.4×10^{12}	21.5×10^{12}	21.5×10^{12}
3	arc resistance	Seconds	139	156	155	186	182	174	162
4	dielectric strength	kV/mm	13.30	14.64	14.62	16.08	15.18	14.82	14.74

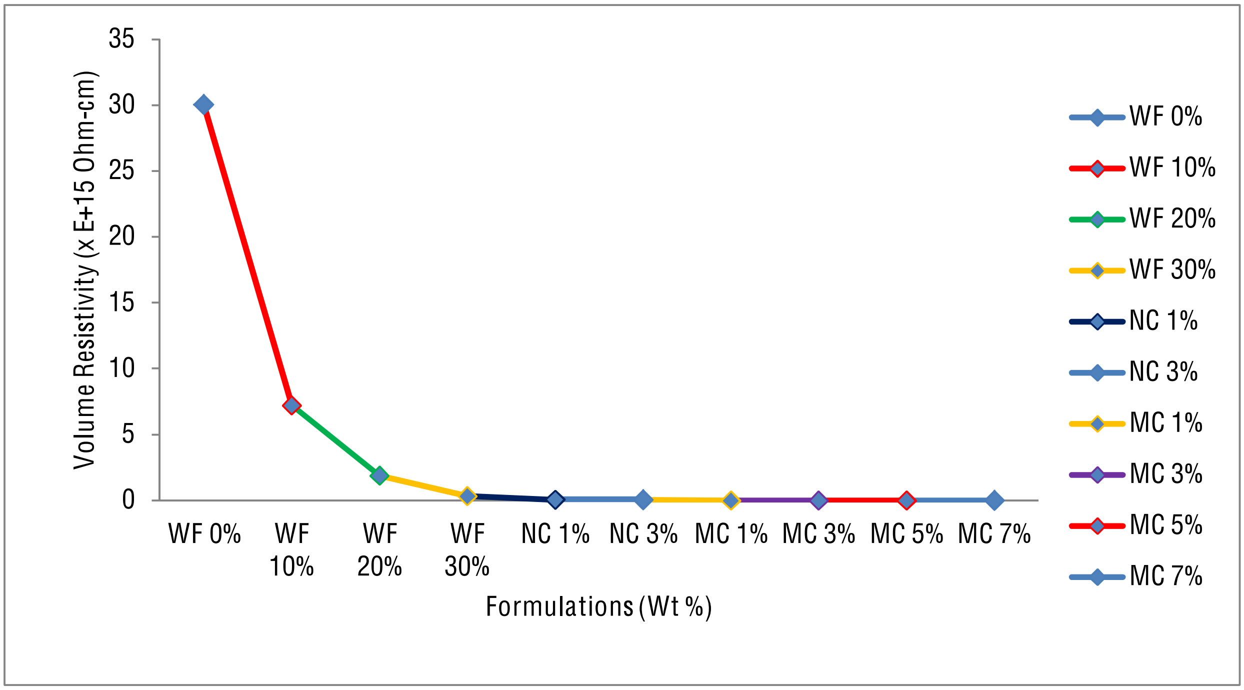

Figure 4. Volume resistivity of R.HDPE/wood flour/nanoclay/microclay composites.

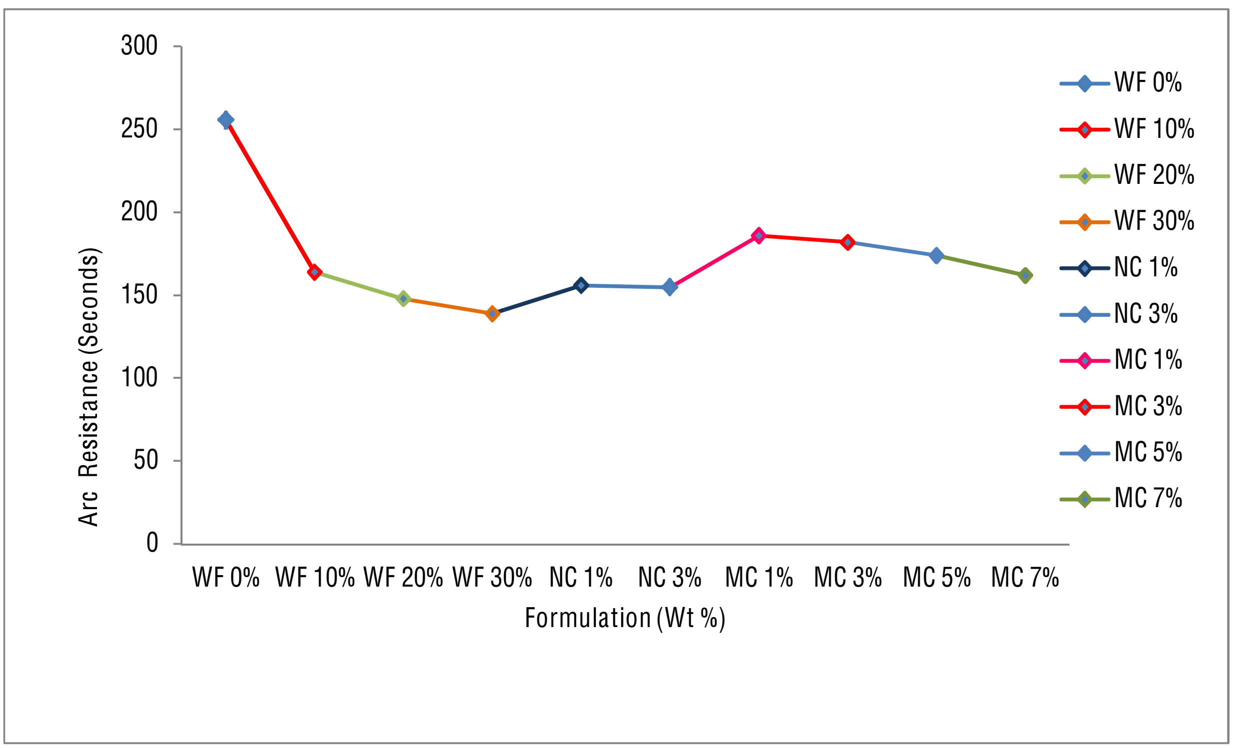

Figure 5. Arc resistance of R.HDPE/wood flour/nanoclay/microclay composites.

Table 6. Physical properties of wood flour filled recycled HDPE.

S.No	Properties	Unit	Test Results			
			R.HDPE 100%	R.HDPE+Wood flour		
				WF10%	WF20%	WF30%
1	MFI at 10 kg & 190°C	g/10min	3.734	2.998	2.698	3.318
2	density	g/cc	0.955	0.979	1.015	1.054
3	shrinkage	%	3.047	3.01	2.409	2.38
4	water absorption	%	0.044	0.137	0.319	0.420

Table 7. Physical properties of R. HDPE+30% wood flour with nanoclay/microclay

S.No	Properties	Unit	Test Results						
			R.HDPE + 30%WF	R.HDPE + 30% WF		R.HDPE + 30% WF + 3% NC			
				NC 1%	NC 3%	MC 1%	MC 3%	MC 5%	MC 7%
1	MFI at 10 kg & 190ºC	g/10min	3.318	3.465	3.255	3.245	3.352	3.475	3.521
2	density	g/cc	1.054	1.055	1.056	1.050	1.051	1.052	1.054
3	shrinkage	%	2.38	2.27	2.03	2.18	1.99	1.87	1.43
4	water absorption	%	0.420	0.394	0.383	0.376	0.351	0.319	0.261

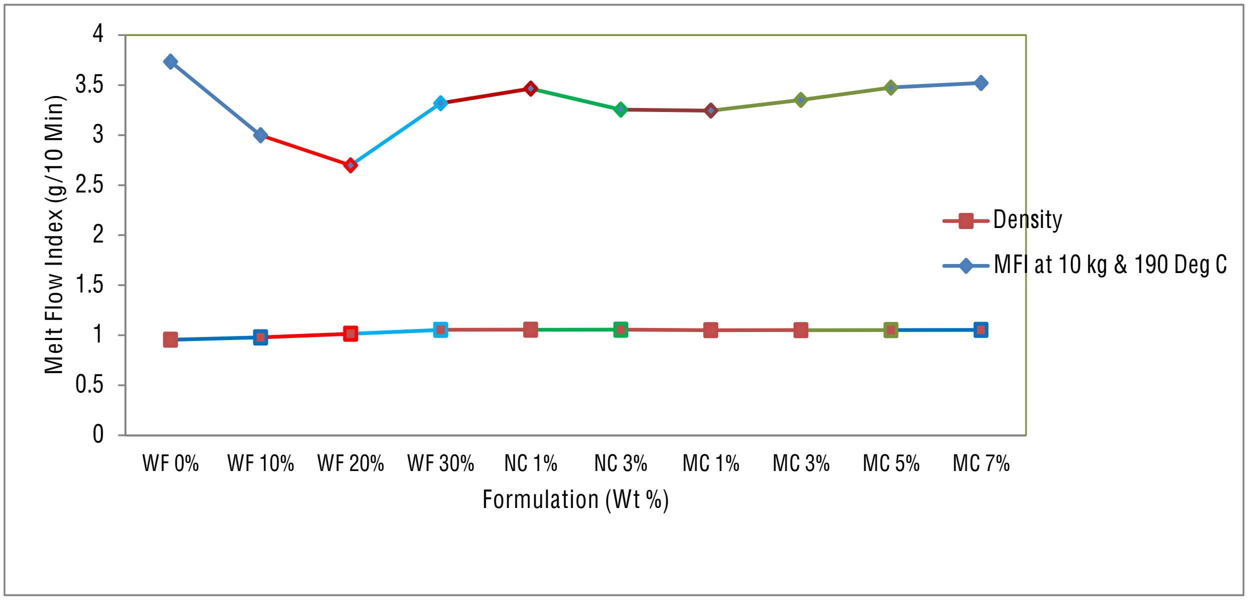

Figure 6. MFI and density of R. HDPE/wood flour/nanoclay/microclay composites

CONCLUSION

The recycled HDPE reinforced with wood flour was having increasing hardness but decreased the impact while increasing the filler content. The tensile modulus, flexural strength and flexural modulus were increased while increasing the filler content and slightly lowered when added with nanoclay/microclay. The elongation at break of the recylcled HDPE/wood flour/nanoclay/microclay composites was decreased when compared to unfilled reycled HDPE. The volume resistivity, surface resistivity, arc resistance was lowered when reinforced with wood flour and nanoclay/nicroclay respectively. Dielectric strength was increased when compared with unfilled recycled HDPE. The melt flow index and shrinkage were decreased when reinforced with wood flour and then getting decreased while added with nanoclay/microclay. Density and water absorption was increased while increasing to the fillers. The wood flour can decrease cost of matrix material. The PE-g-MAH used for compatibility purpose and miscibility in composites. Therefore, the properties are not much more improved and not affected when nanoclay/microclay were used. The nanoclay WPC are having better tensile properties than with microclay added in nanoclay WPC, but the flexural modulus in NC/MC has better values than nanoclay WPC but lower than R. HDPE. Cheaper products can be made with optimum mechanical properties with adequate electrical insulation and physical properties using recycled HDPE reinforced with wood flour composites are bio-degradable and hence eco-friendly. Therefore, the composites can be best suited and adequate for many buildings, construction, automotive interior, and exterior applications.

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