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Effect of particle size and volume fraction on the tensile properties of Wood Ash Particles Reinforced Polypropylene (WARPP) composites

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Abstract
Wood ash is the residual powder left after combustion of wood, such as wood burnt in home fireplace or in an industrial power plant. Wood ash particles reinforced polypropylene (WARPP) composite samples were produced in an injection moulding machine. Three particle sizes of 0.25mm, 0.80mm and 1.40mm of the ash were choosen as representative particles for this reearch. The orthogonal array (matrix) of Taguchi experimental design was used to generate the factors combination used to produce the wood ash particles reinforced polypropylene composite samples. The samples produced were characterized using a mechanical testing method (tensile test). The tensile properties of the composite samples were tested using Hounsfield extensometer experimental rig. Results of the experimental data were then used to plot stress – strain curve which showed that the composites displayed magnesium or aluminum – like tensile behaviour. From the plots, it can be deduced that at 5% volume fraction, the tensile strength increased from 13.2N/mm^2 to 18.4N/mm^2 as the particle size increased from 0.25mm to 0.80mm, while there was a decrease from 18.4N/mm^2 to 11.2N/mm^2 as the particle size increased from 0.80mm to 1.40mm. At 35% volume fraction, the tensile strength increased fairly from 15.4N/mm^2 to 16.4N/mm^2 as the particle size increased from 0.25mm, 0.80mm to 1.40mm. At 60% volume fraction, the tensile strength increased from 13.2N/mm^2 to 16.6N/mm^2 and from 16.6N/mm^2 to 20.4N/mm^2 as the particle size increased from 0.25mm to 0.80mm and from 0.80mm to 1.40mm. The results show that the tensile constitutive behaviour of the WARPP composites is consistent with the typical heat treated steel (gray cast iron), magnesium, aluminum and copper.

Keywords: wood ash, polypropylene, particle size, volume fraction, tensile properties

Introduction

Ashes can be obtained from different materials, such as agricultural by-products like rice husk, industrial wastes, and animal dung. According to the U.S. Environmental Protection Agency (2010) [11], some industrial processes such as combustion, and metallurgical combustion sources generate ash particles in the range of 0.01 to 0.1 micrometer (µm). Wood ash is one of the by-products of such process. It is the residual powder left after combustion of wood, such as wood burnt in home fireplace or in an industrial power plant. Wood ash has long been recognized as a valuable substance, according to Sullivan (2013) [9], since wood ash is derived from plant material, it contains most of the essential nutrients the soil must supply for plant growth. He further stated that when wood is burnt, nitrogen and sulfur are lost as gases, while calcium, potassium, magnesium and trace element compounds remain.

Hume (2006) [4], and Naik *et al.* (2001) [5], observed that many studies have been conducted regarding the chemical composition of wood ash, with variable results, while some results quoted calcium carbonate as the major constituent of ash, others find none but calcium oxide instead. They further reported that some showed as much as twelve percent iron oxide, with elements such as calcium, magnesium, potassium and phosphorus.

Several factors are responsible for the variability in composition. They include:

1. Temperature of combustion: this has direct effect on decomposition of compound elements or substances. Conversion of carbonates, sulfides, etc. to oxides results in carbon, sulfur, carbonates or sulfides. Because of the dissociation to elemental state, some part of the conversion elements vapourize completely at wood combustion temperature;
2. The type of wood (whether hard or soft): age and growing environment of the wood stock imparts on the composition of the wood, and hence the ash;
3. Geographical location: this affects the composition of ash obtained from the wood in a particular location. Application of soil conditioner such as fertilizer affects the mineral composition of the soil.

Properties of ash are obtained from the physical and chemical tests, and the micro structural examination of the ash. Wood ash has the following physical properties: particle size distribution, bulk density and specific gravity. The chemical properties of ash are determined by the total chemical composition of the ash. The chemical composition includes the metallic elements in the ash, the chemical compounds (oxides) and the alkali. The concentration of metallic elements is important because it provides an insight into the potential leaching or wearing away characteristics of the product. The presence of oxides shows the existence of non-amorphous (crystalline) phase.

Polypropylene, a thermoplastic polymer, made by the polymerization of propylene monomers is obtained from the cracking of petroleum products. Polypropylene is the lightest of the common types of plastics. According to Shodhganga (2015) [8], other common types of plastics include polyethylene (LDPE and HDPE), polyvinyl chloride (PVC), and general purpose polystyrene (GPPS) and high impact polystyrene (HIPS). Polypropylene is harder than other common types of plastics, as it has a higher softening point, lower shrinkage and good processing properties. Polypropylene can be isotactic, syndiotactic and atactic in chain structure because of the arrangement of methyl (CH_3) group in the structure.

The commercial type of polypropylene mostly used is the isotactic type. Isotactic type has the highest crystallinity because the chains are packed closely together. Polymers crystallize methyl groups on both sides of the basic chain structure are regularly spaced and follow a particular order. The chemical composition or basic structure of polypropylene is shown in Figure 1 with its chemical formula as C_3H_6.

$$(- CH_2 - \underset{\substack{| \\ CH_3}}{CH} -)$$

Fig 1: Polypropylene basic structure

Most of the applications of wood ash had been to cement based brick moulding or construction materials, while some applications made to non-cement areas had been to thermoset polymers. In thermoplastic polymers, applications had been with wood fiber, wood chips, wood flour and wood sawdust. No application of wood ash particles has been made in thermoplastic polymer. Abdullahi (2006) [1], Naik, Kraus, and Kumar (2001) [5], and Goodman (1998) [3], applied wood ash particles in cement based construction materials as an admixture. Abdullahi (2006) [1], conducted his research on the use of wood ash particles for concrete making. Sanusi, Oyinlola, Akindapo, (2013) [7], investigated the influence of wood ash on the mechanical properties of fibre glass/ epoxy resin composite. Okunade (2008) [6], also investigated the effect of wood ash and saw dust admixture on the engineering compressive strength of burnt clay brick. In this work, wood ash particles reinforced polypropylene (WARPP) composites are studied to investigate the effect of particle size and volume fraction on the tensile properties of WARPP composites.

2. Materials and Methodology

2.1 Materials

Wood ash was prepared from approximately 10kg of wood saw dusts (Terminalia Superba). The saw dust from wood shavings was burnt by smouldering in an open space at ambient temperature for about 12hrs. The ash was collected at the end for sieve analysis. It was produced b the use of sieves of a mechanical vibrator for particles characterization. This characterization (sieve analysis) was conducted at the Soil and Materials laboratory of the Civil Engineering Department of Nnamdi Azikiwe University, Awka. The sieve analysis result is presented in Table 3 section 3, it was performed by using standard sieve sizes of ASTM #14 for 1.40mm, ASTM #20 for 0.80mm and ASTM #60 for 0.25mm.

The chemical analysis tests were conducted at the Dept. of Polymer Technology, Chemical and Leather Research, Ahmadu Bello Univ. Zaria. Result of the X – ray fluorescence analysis of wood ash is presented in Table 4 in section 3. Xanthos (2005) [12], noted that the wood ash contains some of the inorganic oxides that can be used as filler material for plastics. Polypropylene, the polymer resin used in the research was obtained from a resin chemical commercial outfit, Pokez chemicals at Onitsha. Polypropylene (PP) is a linear hydrocarbon polymer, a polyolefin or saturated polymer.

2.2 Samples preparation for tensile test

The polypropylene/ash particles composites were produced in an injection moulding machine. Three particles sizes of 0.25mm, 0.80mm and 1.40mm of the ash were selected as representative particles. The particle size 0.25mm is the least particle size, while particle sizes of 0.8mm and 1.40mm yielded more ash. These particle sizes were used in different volume fractions of 5%, 35% and 60%. To enhance the filler/matrix adhesion, a silane-based coupling agent, 3-aminopropyltrimethoxy silane was used. The filler material was treated with the silane based coupling agent before adding to the polypropylene for composite production. The silane based coupling agent was added without dilution and sprayed to the filler while stirring. The treated filler (wood ash particles) were measured out in three volume fractions of 5%, 35% and 60%. The polymer material, polypropylene was also measured out according to corresponding masses. The measured quantities were mixed and injection moulded. Table 1 shows the masses of wood ash and PP obtained by the application of rule of mixture equations which were used to calculate the volume of materials.

Table 1: Tensile test mould design for volume fraction and masses of filler used in WARPP composite samples production

V_f (%)	$v_f = V_f \, x \, 9{,}144 \, (mm)^3$	$m_f (g)$	$v_m = \frac{1 - V_f}{V_f} x \, V_f \, (mm)^3$	$m_m = v_m \, x \, 9.05 \, x10^{-4} g / mm^3$	V_c
5	457 .2	0.497	8,686 .8	7.86	9,144
10	914 .4	0.995	8,229 .6	7.45	9,144
15	1,371 .6	1.492	7,772 ,4	7.03	9,144
20	1,828 .8	1.989	7,315 .2	6.62	9,144
25	2,286 .0	2.486	6,858 .0	6.21	9,144
30	2,743 .2	2.984	6,400 .8	5.79	9,144
35	3.200 .4	3.481	5,943 .6	5.38	9,144
40	3,657 .6	3.978	5,486 .4	4.97	9,144
45	4,114 .8	4.476	5,029 .2	4.55	9,144
50	4,572 .0	4.973	4,572 .0	4.14	9,144
55	5,029 .2	5.470	4,114 .8	3.72	9,144
60	5,486 .4	5.967	3,657 .6	3.31	9,144

Where V_f% = volume fraction of filler.

v_f= actual volume of filler with respect to mould cavity and approximately equal to the volume fraction for that cavity.

v_m = volume of matrix material.

V_c = volume of composite related to the mould cavity and approximately equal to the volume of mould cavity.

m_f and m_m are computed for various volume fraction and for composite.

Multi – cavity mould was used to produce five samples for the tensile test. The dimension of the tensile test samples was selected according to ASTM D638 method. Rectangular sample produced with dimensions of 15cm x 1.91cm x 0.32cm for tensile test is presented in Figures 2.

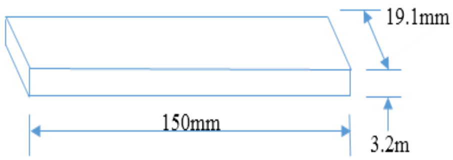

Fig 2: Diagram of specimen for tensile test.

2.3 Determination of the Tensile Strength of WARRP Composites

A universal testing machine, Monsanto Swindon was used to conduct tensile tests. The mean values of stresses and strains were obtained from the experimental primary data of the tensile tests of WARPP samples. The mean values of stress and strain presented in Table 2 were used to plot the stress – strain curves of Figures 3 to 8.

Table 2: Mean values of stress and strain of experimental results of tensile tests of WARPP samples

Sample filler particle size (mm)	Volume fraction (%)	Mean stress (N/mm^2)	Mean strain
0.25	5	0.00	0.00
		5.4165	0.0047
		9.1943	0.0138
		11.667	0.0335
		12.917	0.0604
		13.195	0.0927
		13.195	0.1240
0.25	35	0.00	0.00
		3.8889	0.0060
		8.7499	0.0121
		13.195	0.0272
		15.278	0.0573
		15.278	0.0814
		15.278	0.1086
0.25	60	0.00	0.00
		4.722	0.0073
		8.750	0.0181
		10.972	0.0431
		12.222	0.0776
		12.917	0.1138
		13.195	0.1595
0.80	5	0.00	0.00
		7.687	0.0051
		13.1944	0.0108
		17.0833	0.0263
		18.3333	0.0513
		18.4772	0.0749
		18.4772	0.1052
Sample filler particle size (mm)	Volume fraction (%)	Mean stress (N/mm^2	Mean strain
0.80	35	0.00	0.00
		4.8054	0.0047
		10.6943	0.0135
		14.665	0.0340
		15.1387	0.0664
		15.2775	0.0987
		15.2775	0.1289
0.80	60	0.00	0.00
		6.3889	0.0086
		11.8055	0.0190
		15.8333	0.0427
		16.6667	0.0715
		16.6667	0.1056
		16.6667	0.1319
1.40	5	0.00	0.00
		4.3888	0.0056
		8.4167	0.0162
		10.4167	0.0444
		10.9722	0.0806
		11.1111	0.1172
		11.1111	0.1401
1.40	35	0.00	0.00
		6.3870	0.0060
		11.1111	0.0018
		14.1670	0.0421
		15.8330	0.0776
		16.5280	0.1185
		16.5280	0.1651
1.40	60	0.00	0.00
		6.9400	0.0065
		13.8889	0.0127
		18.7500	0.0237
		20.8300	0.0453
		20.8300	0.0905
		20.8300	0.1293

3. Results and Discussion

Table 3 depicts the result of sieve analysis with the column data explained below the table.

Table 3: Sieve Analysis of Wood Ash

Sieve size (mm)	Weight of basin, w_1 (g)	Weight of basin + ash, w_2 (g)	Weight of ash retained, w_3 (g)	Percentage weight of ash retained (%)	Cummu. % weight of ash retained (%)	% weight of ash passing (%)
2	151.30	185.69	34.39	11.46	11.46	88.54
1.6	151.30	155.67	4.37	1.46	12.92	87.08
1.4	151.30	232.86	81.56	27.19	40.11	59.89
0.8	151.30	259.32	108.02	36.01	76.12	23.88
0.4	151.30	201.32	50.02	16.67	92.79	7.21
0.25	151.30	167.03	15.51	5.17	97.96	2.04
Pan (tray)	151.30	157.65	6.13	2.04	100	0.00
Total			300			

Column 1 of Table 3 shows the particle sizes retained by different sieve sizes;
Column 2 shows the weight of empty basin (g);
Column 3 shows the weight of the basin plus the mass of ash retained for different particle sizes;
Column 4 contains the weight of ash retained for different particle sizes, which when added together gave a total mass of ash (300 g);
Column 5 shows the percentage of weight of ash retained for different particle sizes e.g. $\frac{34.39\ g}{300\ g} \times 100 = 11.46\ \%$
Column 6 shows the cummulative percentage of weight of ash retained (e.g. for particle size of 1.6mm, cumulative % of weight of ash is 11.46% + 1.46% = 12.92 %); while,
Column 7 shows the percentage of weight of ash that passed different sieve sizes for different particle sizes (e.g. for particle size of 2mm, weight of ash retained was 34.39g, then weight that passed is 300g – 34.39g = 265.61g. ∴ 265.61g / 300g x 100 = 88.54%).

3.2 X – Ray Fluorescence Analysis

The result of the x – ray fluorescence analysis is presented in Table 4. The result of this research is presented along with the result of the work of Sanusi, Oyinlola, Akindapo, (2013) [7], for comparison.

Table 4a: Result of X – Ray Fluorescence Analysis of Wood Ash

Compound oxides	% composition
* Silicon Dioxide, SiO_2	13.9
Titanium Oxide, TiO_2	0.76
* Sulphur (vi) oxide, SO_3	10.5
Iron Oxide, Fe_2O_3	4.31
* Calcium Oxide, CaO	45.2
* Manganese Oxide, MnO	1
* Rubidium Oxide, Rb_2O	0.055
Manganese Oxide, MnO	2.97
* Strontium Oxide, SrO	0.1
* Indium (iii) Oxide, In_2O_3	3.6
Copper (ii) Oxide, CuO	0.083
* Potassium Oxide, K_2O	15.1
Zinc Oxide, ZnO	2.24
* Europium (iii) Oxide, Eu_2O_3	0.07
* Barium Oxide, BaO	0.09

Table 4b: Result of chemical composition of wood ash analyzed by Sanusi, Oyinlola, Akindapo, (2013) [7]

Compound Oxide	Concentration %
Calcium Oxide, CaO	78.50
Potassium Oxide, K_2O	8.79
Silicon Dioxide, SiO_2	7.20
Sulphur (vi) Oxide, SO_3	1.10
Titanium Oxide, TiO_2	0.31
Manganese Oxide, MnO	0.09
Iron Oxide, Fe_2O_3	1.27
Zinc Oxide, ZnO	0.06
Yttrium Oxide, Y_2O_3	1.20
Barium Oxide, BaO	1.00
Cerium Oxide, CeO_2	0.04
Europium (iii) Oxide, Eu_2O_3	0.05
Rhenium Oxide, Re_2O_7	0.30

On comparative basis the chemical compositions of the analyzed wood ash has similar chemical compound but in varying compositions with the wood ash analyzed for application in Fibre glass/Epoxy resin composite (Sanusi, Oyinlola, Akindapo, 2013) [7]. The three major compositions in the wood ash for application in Fibre glass/Epoxy resin composite are: SiO_2 (7.2%), K_2O (8.79%) and CaO (78.5%). The wood ash applied in this work has CaO (45.2%), K_2O (15.1% and SiO_2 (13.9%) as its major compositions.

From the analysis, the wood ash contains some of the inorganic oxides that are used as filler material for plastics according to Xanthos (2005) [12]. They are the oxides of metals that are not transition metallic elements. From Table 4, the asterisked compounds which include: silicon dioxide (SiO_2), Sulphur (vi) oxide (SO_3), Calcium Oxide (CaO), Magnesium oxide (MgO), Rubidium Oxide (Rb_2O), Strontium oxide (SrO), Indium (iii) oxide (In_2O_3), Potassium Oxide (K_2O), Europium (iii) oxide and Barium Oxide (BaO) are the inorganic oxides. These oxides form 89.615% of the total oxides present in the ash. It is known that transition metallic elements ions easily change their oxidation states thus enabling them to act as catalysts.

3.3 Stress – Strain Plots of WARPP Sample Composites

Figures X1 to Z2 show the stress – strain plots of the WARPP composites. It is clear from the plots that the tensile strengths exhibit the behaviour of magnesium, aluminum or gray cast iron material. The plots did not show distinctive yield points (i.e. the lowest stress at which there is a marked increase in strain without corresponding increase in stress). However, the yield point of the plots can be determined based on how Ugural (2004) [10] defined it. He stated that it is usual to use the yield strength at the point where a line at 0.2% offset of strain is drawn parallel to the initial slope at point 0 (origin) of the curve to define it.

Table 1a - Stress - strain WARPP sample plot @ 0.25mm PS, 5% VF.							
Average Strain	0.00	0.0047	0.0138	0.0335	0.0604	0.0927	0.124
Average Stress	0.00	5.4165	9.1943	11.6667	12.9166	13.1945	13.1945

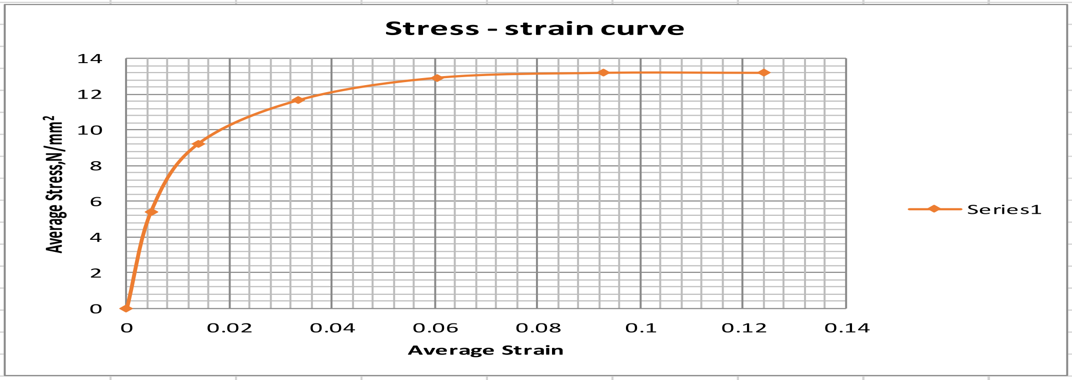

Table 4a: Stress - strain WARPP sample plot @ 0.80 PS, 5% VF							
Average Strain	0.00	0.0051	0.0108	0.0263	0.0513	0.0749	0.1052
Average Stress	0.00	7.6387	13.1944	17.0833	18.3333	18.4772	18.4772

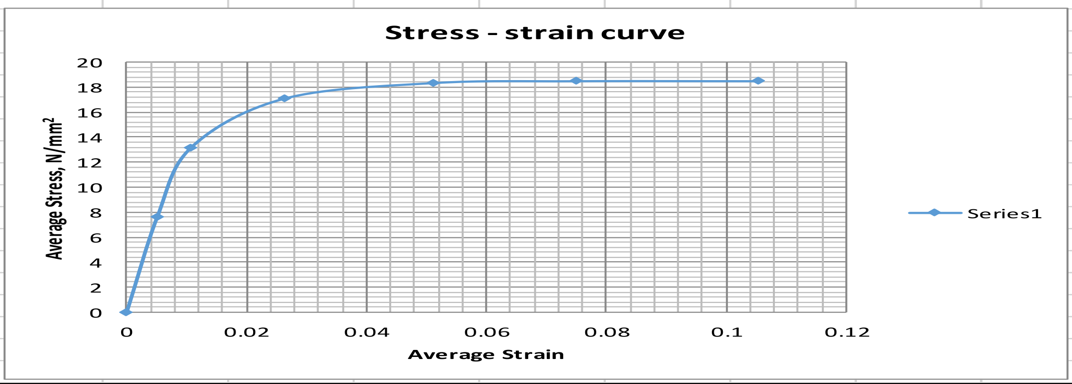

Fig 3: Stress – strain WARPP sample plots for 0.25mm& 0.80mm PS @ 5% VF

Table 7a - Stress - strain WARPP sample plot @ 1.40mm PS, 5% VF.							
Average Strain	0.00	0.0056	0.0162	0.0444	0.0806	0.1172	0.1401
Average Stress	0.00	4.3888	8.4167	10.4167	10.9722	11.1111	11.1111

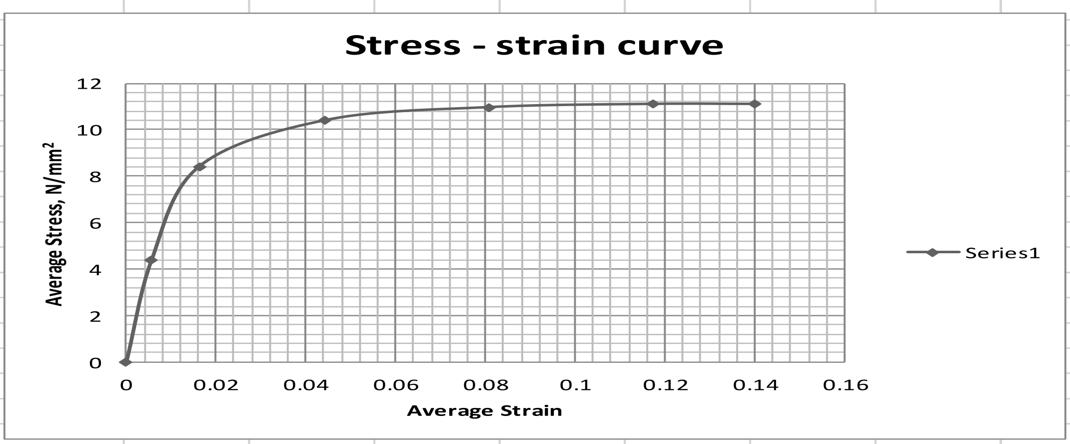

Fig 4: Stress – strain WARPP sample plot for 1.40mm PS @ 5% VF

Table 2a - Stress strain WARPP sample plot @ 0.25mm PS, 35% VF.

Average Strain	0.00	0.006	0.0121	0.0272	0.0573	0.0814	0.1086
Average Stress	0.00	3.8889	8.7499	13.1945	15.2774	15.2774	15.2774

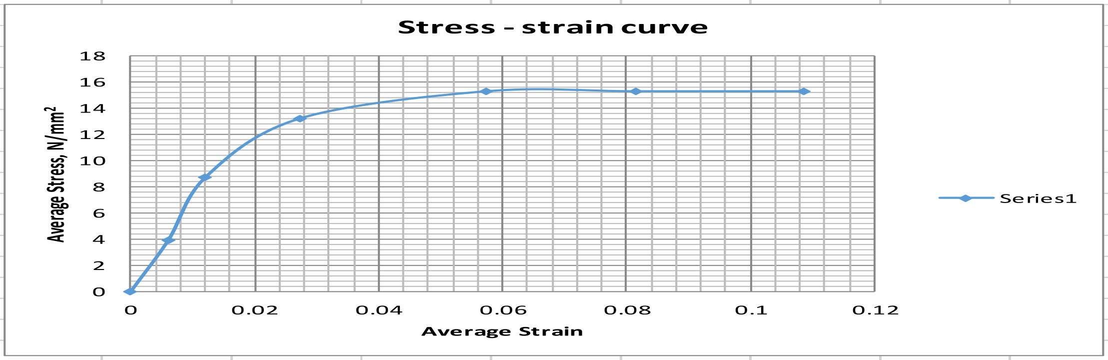

Table 5a - Stress - strain WARPP sample plot @ 0.80mm PS, 35% VF.

Average Strain	0.00	0.0047	0.0135	0.034	0.0664	0.0987	0.1289
Average Stress	0.00	4.8054	10.6943	14.6665	15.1387	15.2775	15.2775

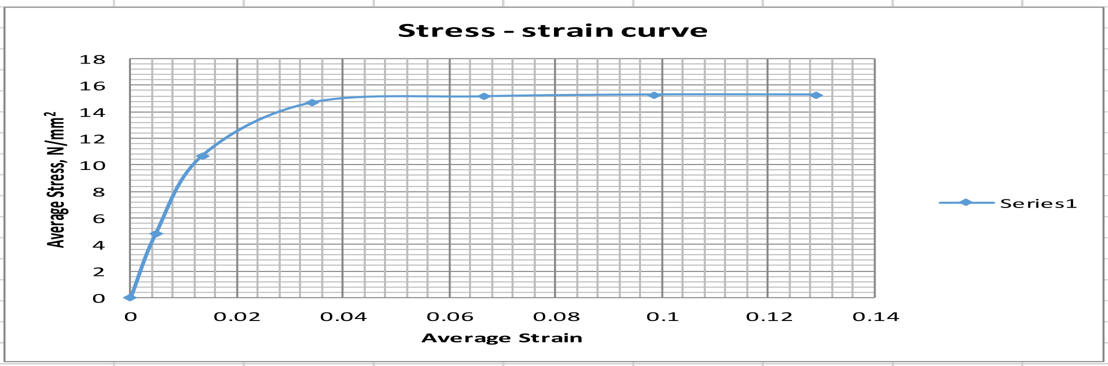

Fig 5: Stress – Strain WARPP sample plots for 0.25mm and 0.80mm PS @ 35% VF

Table 8a - Stress - strain WARPP sample plot @ 1.40mm PS, 35% VF.

Average Strain	0.00	0.00598	0.01766	0.04214	0.07762	0.1185	0.16508
Average Stress	0.00	6.387	11.11	14.167	15.833	16.528	16.528

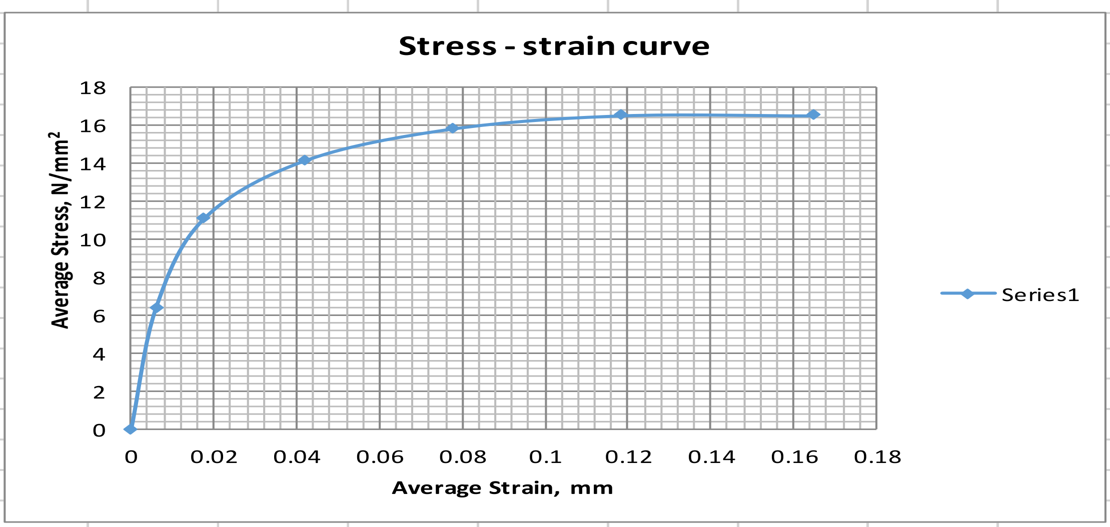

Fig 6: Stress – strain WARPP sample plot for 1.40mm PS @ 35% VF

Table 3a - Stress -strain WARPP sample plot @ 0.25 PS, 60% VF.							
Average Strain	0.00	0.0073	0.0181	0.0431	0.0776	0.1138	0.1595
Average Stress	0.00	4.722	8.75	10.972	12.222	12.917	13.195

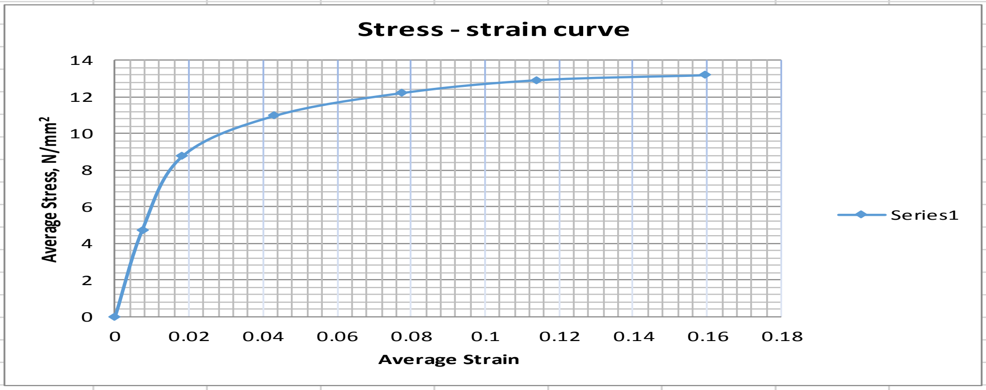

Table 6a - tress - strain WARPP sample plot @ 0.80mm PS, 60% VF.							
Average Strain	0.00	0.0086	0.019	0.0427	0.0715	0.1056	0.1319
Average Stress	0.00	6.3889	11.8055	15.8333	16.6667	16.6667	16.6667

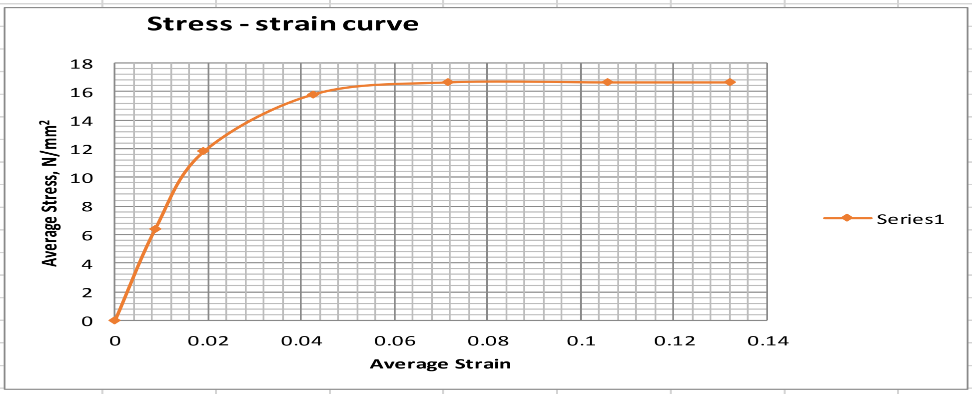

Fig 7: Stress – Strain WARPP sample plots for 0.25mm & 0.80mm PS @ 60% VF

Table 9a - Stress - strain WARPP sample plot @ 1.40mm PS, 60% VF.							
Average Strain	0.00	0.0065	0.0129	0.0237	0.0453	0.0905	0.1293
Average Stress	0.00	6.94	13.89	18.75	20.83	20.83	20.83

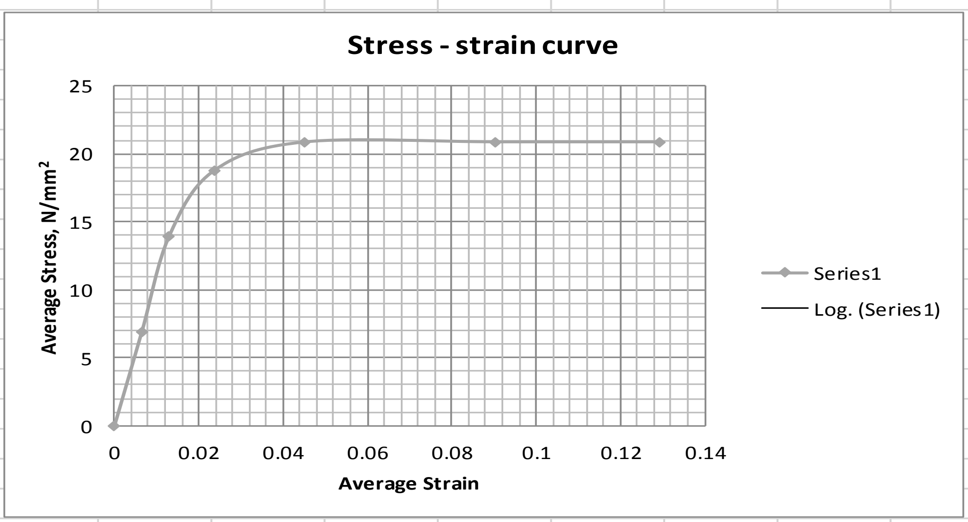

Fig 8: Stress – Strain WARPP sample plot for 1.40mm PS @ 60% VF.

4. Conclusion

From the plots, it can be deduced that at 5% volume fraction, the tensile strength increased from 13.2N/mm^2 to 18.4N/mm^2 as the particle size increased from 0.25mm to 0.80mm. At 35% volume fraction the tensile strength fairly increased from 15.4N/mm^2 to 16.4N/mm^2 as the particle size increased from 0.25mm, 0.80mm to 1.40mm. At 60% volume fraction the tensile strength increased from 13.2N/mm^2 to 20.4N/mm^2 as the particle size increased from 0.25mm to 1.40mm. The results of the plots show that the tensile constitutive behaviour of the WARPP composites is consistent with the typical heat treated steel (gray cast iron), magnesium, aluminum and copper, which is in line with the research finding of Ugural (2004) [10].

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