ISSN 0511-5728
The West Indian Journal of Engineering
Vol.35, No.1, July 2012, pp.51-59

Tensile Properties of Iron Ore Tailings Filled Epoxy Composites

Segun M. Adedayo [a] and Modupe A. Onitiri [b Ψ]

[a] Department of Mechanical Engineering, University of Ilorin, Nigeria; E-mail: amsegun@unilorin.edu.ng

[b] Department of Mechanical Engineering, University of Lagos, Nigeria; E-mail: monitiri@unilag.edu.ng

[Ψ] *Corresponding Author*

(Received 20 September 2011; Revised 23 January 2012; Accepted 07 February 2012)

Abstract: *Iron ore tailings reinforced epoxy composite (ITR-EC) is produced by reinforcing epoxy with iron ore tailings, which is the waste material derived from the beneficiation of iron ore. Two particle sizes, namely; 150 µm and 300 µm of varying percentage volume 0 to 30% at intervals of 5% were considered. Prior to this, particle size analysis over the range of -63 µm and +2000 µm in 8 different mesh sizes and chemical tests were carried out on the iron ore tailings. A uniaxial tensile test was carried out on the ITR-EC produced to obtain stress-strain curves from which tensile yield, tensile strength and Young's modulus curves with varying volume content of iron ore tailings and particle size were generated. Empirical data from the tensile test were compared with the Nielsen's, Bigg's and Einstein's equations. It was discovered that 30% volume content of 300 µm iron ore tailings gave the maximum Young's modulus; 4.83% greater than that for pure epoxy. Addition of 300 µm iron ore tailings causes an increase in yield strength with increasing percentage volume content of iron ore tailings but reduced yield strength when compared with 150 µm ITR-EC.*

Keywords: *Compo-indirect squeeze casting, Compo-casting, Iron ore tailings, Epoxy, Composite, Particle size*

1. Introduction

Most modern design requires materials with unusual combination of properties that cannot be met by conventional metal alloys, ceramics and polymeric materials; hence, the need for the development and use of composites. Given the vast range of materials that may be considered as composites, the broad range of uses for which they may be designed for and their wide range of properties, it is possible to generalise the properties of composite materials as follows; high strength, high stiffness, high fatigue and creep resistance, low density, low creep and low coefficients of thermal expansion (CTE) (Smith, 2003; Zweben, 2008; Flinn and Trojan, 1990). The properties of composites are a function of the properties of the constituent phases, their relative amounts, and the geometry of the dispersed particle (such as the particle shape, the particle size, distribution, and orientation).

In the case of adoption of plastics as an alternative to metals, the plastic material is expected to exhibit mechanical characteristics such as stiffness, toughness, abrasion resistance, dimensional and thermal stability at ambient and high-temperature. To achieve these properties, reinforcing materials such as fibers, short fibers or particles are added to the plastics matrix to produce plastics matrix composites (PMCs). These composites have become one of the new competitive materials in engineering and are increasingly found in structural and industrial applications because of their high tensile strength and stiffness and low density (Ouyang, 2005; Ning, 2005; Srinivasan et al., 2007).

The interaction between matrix and particles (i.e. interface behaviour) is an important factor which influences the mechanical properties of particulate composites (Sohn et al., 2003). Maiti and Singh (1986) surmised that the matrix (especially plastics matrix) and reinforcing particles, in the absence of coupling or dispersion agents, experience formation of voids in the matrix and poor adhesion between matrix and reinforcing particles with increased particle size. Voids, air pockets in the matrix, are harmful because the particles passing through the void are not supported by the matrix. Addition of coupling agent, also known as a bonding agent or binder, provides a flexible layer at the interface between particles and matrix that will improve their adhesion and reduce the number of voids trapped in the material (Wall et al., 2003).

An important work on particle reinforced thermosetting plastics worth mentioning is the work by Ku et al. (2008) on the tensile properties of phenol formaldehyde reinforced with Environspheres SLG. They discovered that the composite with 10% SLG produces the highest yield, tensile strength and Young's modulus. In an investigation by Singla and Chawla (2010) on the mechanical properties of epoxy resin – fly ash composite, it was discovered that compressive strength of the composite increased with increasing fly-ash. In another investigation by Sapuan et al. (2003) on the tensile properties of epoxy reinforced with coconut shell particles, they discovered that tensile properties increased with increasing filler content. Nakamura et al. (1992) discovered that epoxy resin filled with angular-shaped silica shows increased tensile strength with decrease in particle size.

In this present work, tensile tests will be carried out on epoxy reinforced with iron ore tailings of varying percentage volume and particle size to determine the effect of the filler on the yield strength, tensile strength and Young's modulus of the epoxy composite produced. Efforts will also be made to determine the optimum percentage volume of iron ore tailings used in the composite. Empirical data obtained from the tensile test will be compared with the Nielsen's (1996), Bigg's (1979) and Einstein's (1905) models.

2. Epoxy and Iron ore tailings:

Epoxy is a copolymer formed from two different chemicals referred to as resin and hardener. The resin consists of monomers or short chain polymers with an epoxide group at either end while the hardener consists of polyamine monomers. When these compounds are mixed together, the amine groups react with the epoxide groups to form a covalent bond so that the resulting polymer is heavily cross linked, and is thus rigid and strong. Epoxies are one of the most widely used thermosetting resin system (others are polyesters and vinyl esters) used in structural composites; collectively they account for 90% of all thermosetting resin used (SP Systems, 1998).

Iron ore tailings are the waste generated during the beneficiation of iron ore. The beneficiation process reduces the solid impurities physically bonded to the iron ore, hence, producing an ore of greater iron composition. The process of improving the percentage iron content of the ore leads to production of large quantities of tailings (Adepoju and Olaleye, 2001; Olubambi and Potgieter, 2005) which are dumped on site as wastes. As part of efforts to put this waste into judicious use, its influence on the tensile properties of epoxy, when added as filler to form composites, is to be investigated.

3. Theory

Among the challenges which particle reinforced plastics composites (PRPC) present is the complexity of their mechanical behaviour, particularly during plastic deformation. This makes it difficult to predict performance analytically and hence leads to conservative designs and extensive test programmes (McCarthy and Wiggeraad, 2001).

The tensile behaviour of rigid particle reinforced composites is influenced by the particle size, filler concentration, filler surface treatment, matrix and filler properties, superimposed pressure, and the rate of strain. It is well established that the fracture of particulate composites is associated with interfacial debonding between the matrix and particles, particle cracking, and the ductile plastic failure in the matrix depending on the relative stiffness and strength of the two constituent materials and the interface strength. According to Nie (2005), if either constituent materials have material properties of the same order of magnitude or the strength of particle is low, particle cracking can occur. On the other hand, if the embedded particles are much stiffer and stronger than the matrix, matrix cracking (or cavity formation) and particle/matrix interface debonding become the major damage modes.

Ravichandran and Liu (1995) presented a schematic of a possible damage mode for a two-phase spherical particle reinforced composite (in perfect adhesion) subjected to tension. According to them, upon loading at a critical strain level the matrix deforms more than the filler particle (interfacial debonding) where formation of cavity for well-bonded particles occurs. Tensile strength and modulus drastically decrease after debonding takes place, and there is a large increase in volume (dilation) as elongation continues (Nie, 2005; Kwon et al., 1998).

According to Nielsen (1996), the elongation to break of a system filled with particles of approximately spherical shaped particles and assuming perfect adhesion can be predicted by equation (1) below;

$$\varepsilon_c = \varepsilon_p \left(1 - \phi^{1/3}\right) \qquad \text{....Eq.(1)}$$

where ε_c is the elongation at break of the composite, ε_p is the elongation at break of the unfilled polymer while ϕ is the percentage volume fraction of the filler.

Bigg (1979) proposed a model which states that, for a case of no adhesion between the polymer matrix and the filler, the tensile strength of the composite may be expressed as;

$$\sigma_c = \sigma_p \left(1 - b\left(\phi^{2/3}\right)\right) \qquad \text{....Eq.(2)}$$

where σ_c is the tensile strength of the composite, σ_p is the tensile strength of the polymer matrix while b is a constant which accounts for the adhesion quality between the matrix and filler. b = 1.21 implies the extreme case of poor adhesion, hence, a lower b value e.g. b = 1.1 implies better adhesion.

Einstein (1905) proposed two equations which are valid only at low concentration. The first assumes that with perfect adhesion between the filler and the polymer matrix the elastic modulus can be expressed as;

$$E_c = E_p (1 + 2.5\phi) \qquad \text{....Eq.(3)}$$

while the second assumes that with poor adhesion between the filler and the polymer matrix the elastic modulus can be expressed as;

$$E_c = E_p (1 + \phi) \qquad \text{....Eq.(4)}$$

where E_c is the elastic modulus for the composite while E_p is the elastic modulus for the polymer matrix.

4. Experimental Method

4.1 Materials

The matrix used is commercial epoxy in liquid form under brand name "virgin epoxy" while the filler is iron ore tailings in particle form and irregular in shape with particle sizes ranging between 150 and 300 µm. The iron ore tailings were obtained from the iron ore beneficiation plant in Kogi State, North Central, Nigeria. The volume mix ratio adopted in this work is from 5 to 30% at intervals of 5%. Composites produced from the addition of iron ore tailings contents above 30% in epoxy disintegrated easily on handling.

4.2 Chemical composition of iron ore tailings

The following tests were carried out to determine the chemical composition of the tailings (see Table 1).

4.3 Iron ore tailings preparation

The iron ore tailings were dried at room temperature 30±2°C and 50 ± 5% relative humidity for a minimum of 40 hours (ASTM D 618; ASTM E 171; ASTM E 41). The different particle sizes were generated using standard ASTM laboratory sieves (Adepoju and Olaleye, 2001; Olubambi and Potgieter, 2005). After vibrating, the sieve arrangement was dismantled and the tailings deposited in each sieve were weighed and recorded (see Table 2).

Table 1. Test methods to determine iron ore tailings constituents

S/No.	Constituent	Test method
1	pH	Glass-electrode pH meter (Philips meter) model PW9504
2	Moisture content, organic carbon and total organic matter	Titration method (Walkey-Black method)
3	Metals	Atomic Absorption Spectrophotometer (AAS) Perkin Elmer Analyst 200 using air-acetylene flame

Table 2. Particle size analysis of iron ore tailings

Sieve Size Range (µm)	Normal Aperture size (µm)	Weight Retained (g)	Weight Retained (%)	Cumulative weight retained (%)	Cumulative passing (%)
> 2000	2000	0.35	0.18	0.18	99.82
< 2000 > 1180	1180	6.01	3.08	3.26	96.74
< 1180 > 600	600	48.01	24.63	27.88	72.12
< 600 > 425	425	30.4	15.59	43.48	56.52
< 425 > 300	300	31.02	15.91	59.39	40.61
< 300 > 212	212	25.22	12.94	72.32	27.68
< 212 > 150	150	17.84	9.15	81.47	18.53
< 150 > 63	63	25.54	13.10	94.57	5.43
< 63	-63	10.57	5.42	100.00	0.00
	Total	194.96	100		

The particle volume fraction was calculated using the relationship (Tavman, 1996) in Equation (5) below:

$$\phi = \frac{\varphi}{\varphi + (1-\varphi).\frac{\rho_{par}}{\rho_{mat}}} \qquad \text{....Eq.(5)}$$

where, ϕ = volume fraction of particle, φ = weight fraction of particle, ρ_{mat} = density of matrix, and ρ_{par} = density of particle

The weight fraction of particle, φ, was determined using a OHAUS digital scale with an accuracy of 0.01g. The density of the particle, ρ_{par}, was measured at room temperature based on the Archimedes principle with water as the immersion medium (Wang, 2003). ρ_{par} was calculated from Equation (6) below;

$$\rho_{par} = \rho_{wat}\left(\frac{D}{M-S}\right) \qquad \text{....Eq.(6)}$$

where ρ_{par} = density of particle, ρ_{wat} = density of water, D = dry mass of particle, S = mass of particle suspended in water, and M = mass of particle saturated with water.

4.4 Specimen production

The compo-casting (CC) process was used to produce the ITR-EC tensile test specimens using the ITR-EC tensile specimen production rig (see Figures 1 and 2). The ITR-EC tensile specimen production rig consists of a wooden base, a plastic cavity strip, a wooden top, two thread bolts and two locking nuts. The base and top were made from 4mm thick sherry oak wood while the plastic cavity was made from polyurethane. Polyvinyl chloride (PVC) films were introduced between the layers to prevent the cast from sticking to the wooden top and base. One and three parts of epichlohydrin (hardener) and epoxy, respectively, were poured into a clean plastic container and stirred thoroughly with a wooden palate.

An appropriate ratio of iron ore tailings was then added and stirred thoroughly so as to obtain a perfect mix and remove air.

The PVC film was fixed between the wooden base and the cavity strip. The mix was then poured into this arrangement. The second PVC film was placed on the filled cavity followed by the wooden top. The arrangement was then clamped using the two threaded bolts and locking nuts. The cast was allowed to cure for 24 hours before the rig was dismantled and the cast removed. This procedure was carried out for different mix ratios.

Figure 1. ITR-EC tensile test specimen production rig

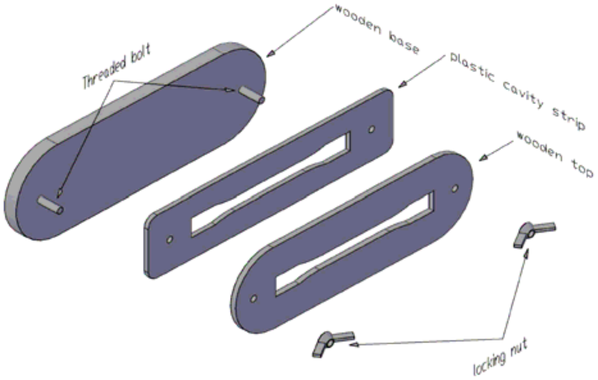

Figure 2. Components of ITR-EC tensile test specimen production rig

4.5 Tensile test

The tensile test was carried out as specified in (ASTM D 638) with a test speed of 1.00 mm/min under standard laboratory atmosphere (ASTM E 171; ASTM E 41) on an Instrom 3369 testing machine. Prior to testing, the tensile test specimens were conditioned at room temperature 30°C ± 2°C and 50 ± 5% relative humidity for a minimum of 40 hours (ASTM D 618; ASTM E 171; ASTM E 41; Onitiri and Adeniyi, 2003). The tensile test machine and procedure were highlighted in a previous work (Onitiri and Adeniyi, 2003). Five specimens were tested for each particle size and corresponding iron ore tailings volume ratio considered.

5. Results and Discussions

5.1 Iron ore tailings composition and size analysis

Table 3 shows the result obtained from the chemical test carried out on iron ore tailings. It can be seen that silicon oxide is the largest constituent of the iron ore tailings. This is similar to the trend recorded by Olubambi and Potgieter (2005) for bulk iron ore from the same source. The sharp drop in Fe_2O_3 from 30.88% in Olubambi and Potgieter (2005)'s work to 0.23% could be attributed to the beneficiation process which causes iron oxide to decrease and increase in bulk iron ore and iron ore tailings, respectively.

The graph of percentage weight retained against aperture size is presented in Figure 3. It can be seen that particle sizes in the range 1180 to 2000 μm form the highest constituent of iron ore tailings while the least was recorded for those greater than 2000 μm.

Table 3. Chemical composition of Itakpe iron ore tailings

S/No.		Composition (%)
1	Moisture	0.1487
2	Total organic carbon	0.6442
3	Total organic matter	0.7598
4	Fe_2O_3	0.2312
5	Copper	0.0061
6	Zinc	0.0018
7	Nickel	0.0013
8	Sodium	0.0051
9	Silicon oxide	61.4771
10	Calcium oxide	11.8924
11	Potassium	0.0012
12	Magnesium oxide	4.1640
13	Chromium	0.0017
14	Cadmium	1.65E-06
15	Magnesium	0.0025
16	Aluminum oxide	20.6630

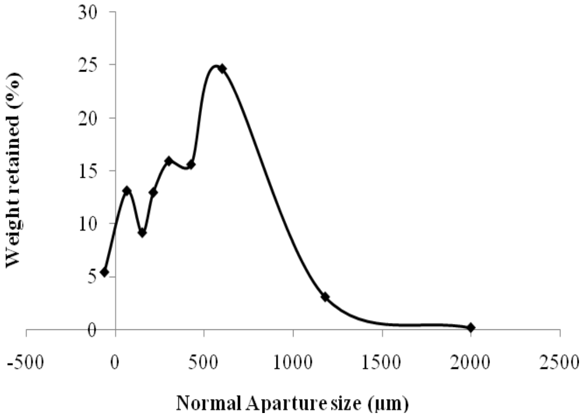

Figure 3. Cumulative passing versus normal aparture size

5.2 Tensile test results

Stress-strain results for 150, 212 and 300μm particle size iron ore tailings reinforced epoxy composite (ITR-EC) are presented in Tables 4 and 5.

Figures 4 and 5 show the stress-strain curves for 150μm and 300μm iron ore tailings reinforced epoxy composite (ITR-EC) with varying volume content of iron ore tailings, respectively. 150 μm ITR-ECs exhibit decreasing plastic deformation at low stress with increasing % volume content of iron ore tailings up to 25%. This could be attributed to the fact that increased volume content of iron ore tailings leads to greater particle contact and reduction in inter-particle space to be occupied by the matrix. This in turn culminates into increased particle-matrix debonding and reduction in ductility. 300 μm ITR-EC exhibits better plastic deformation at low stress for all % volume content when compared with pure epoxy. The curves experience convergence at low strain (i.e., below 3%) while divergence increases with increasing strain.

Table 4. Stress-strain results for 150 μm particle size ITR-EC

	Volume ratio of iron ore tailings (%)							**Min.**	**Mean**	**Max.**
	0	**5**	**10**	**15**	**20**	**25**	**30**			
Stress at yield (MPa)	*5.00*	2.00	2.00	2.00	0.50	3.00	2.50	0.50	2.43	5.00
Stress at ultimate point (MPa)	*40.00*	15.00	32.00	36.00	30.00	20.00	25.00	15.00	28.29	40.00
Stress at fracture (MPa)	*40.00*	15.00	32.00	36.00	30.00	20.00	25.00	15.00	28.29	40.00
Strain at yield(%)	*1.31*	1.50	2.20	1.87	1.50	1.24	1.28	1.24	1.56	2.20
	+0.32 -0.32	*+0.10 -0.10*	*+0.30 -0.20*	*+1.13 -0.42*	*+0.20 -0.10*	*+0.56 -0.44*	*+0.28 -0.28*			
Strain at ultimate point (%)	*4.45*	4.35	7.30	8.35	5.38	2.81	3.78	2.81	5.20	8.35
	+1.60 -1.60	*+0.20 -0.10*	*+0.07 -0.13*	*+2.79 -0.69*	*+0.30 -0.30*	*+0.50 -0.5*	*+0.03 -0.03*			
Strain at fracture (%)	*4.45*	4.35	7.30	8.35	5.38	2.81	3.78	2.81	5.20	8.35
	+1.60 -1.60	*+0.20 -0.10*	*+0.07 -0.13*	*+2.79 -0.69*	*+0.30 -0.30*	*+0.50 -0.5*	*+0.03 -0.03*			

Table 5. Stress-strain results for 300 μm particle size ITR-EC

	Volume ratio of iron ore tailings (%)							**Min.**	**Mean**	**Max.**
	0	**5**	**10**	**15**	**20**	**25**	**30**			
Stress at yield (MPa)	*5.00*	0.30	0.20	0.50	0.50	0.50	1.00	0.20	1.14	5.00
Stress at ultimate point (MPa)	*40.00*	34.00	32.00	38.00	32.00	24.00	32.00	24.00	33.14	40.00
Stress at fracture (MPa)	*40.00*	34.00	32.00	38.00	32.00	24.00	32.00	24.00	33.14	40.00
Strain at yield(%)	*1.31*	1.75	2.00	1.70	1.30	1.80	2.06	1.30	1.70	2.06
	+0.32 -0.32	*+0.10 -0.10*	*+0.51 -0.33*	*+0.21 -0.33*	*+0.70 -0.70*	*+0.20 -0.20*	*+0.10 -0.10*			
Strain at ultimate point (%)	*4.45*	5.70	5.50	6.25	4.98	4.75	4.75	4.45	5.20	6.25
	+1.60 -1.60	*+0.35 -0.35*	*+0.35 -0.35*	*+0.23 -0.23*	*+0.25 -0.25*	*+0.37 -0.28*	*+0.18 -0.18*			
Strain at fracture (%)	*4.45*	5.70	5.50	6.25	4.98	4.75	4.75	4.45	5.20	6.25
	+1.60 -1.60	*+0.35 -0.35*	*+0.35 -0.35*	*+0.23 -0.23*	*+0.25 -0.25*	*+0.37 -0.28*	*+0.18 -0.18*			

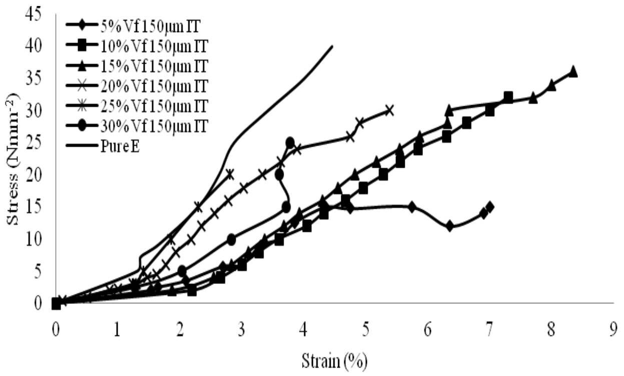

Figure 4. Stress-strain curves for ITR-ECs with iron ore tailings particle sizes 150 μm and volume content 5-30%

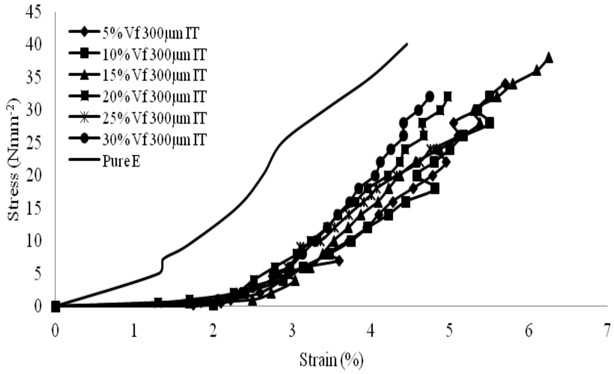

Figure 5. Stress-strain curves for ITR-ECs with iron ore tailings particle sizes 300 μm and volume content 5-30%

The yield strength, tensile strength and Young's modulus of ITR-EC with varying volume content of iron ore tailings and particle size obtained from Figures 4 and 5 are presented in Figures 6, 7 and 8, respectively.

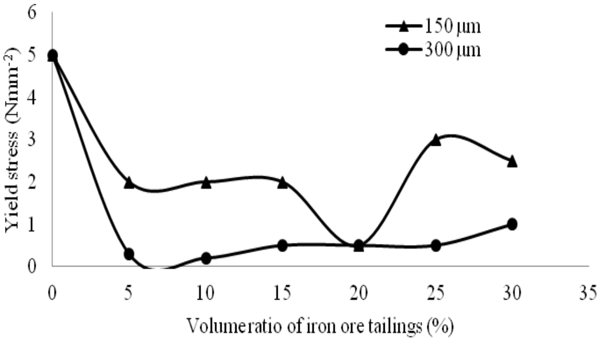

Figure 6. Yield stress of ITR-EC with varying volume content of iron ore tailings and particle size

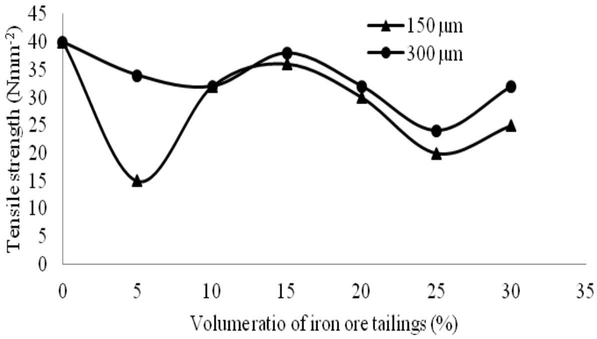

Figure 7. Tensile strength of ITR-EC with varying volume content of iron ore tailings and particle size

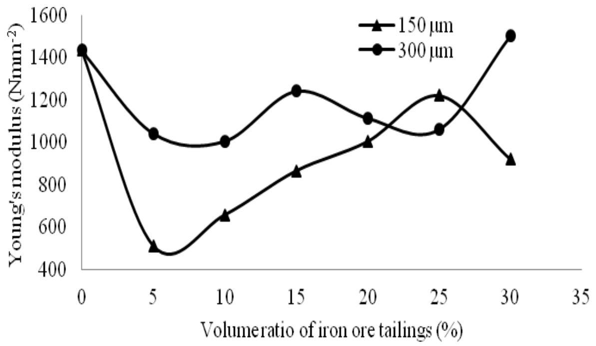

Figure 8. Young's modulus of ITR-EC with varying volume content of iron ore tailings and particle size

In Figure 6, it can be seen that ITR-ECs experienced a sharp drop in yield stress (2 Nmm^{-2}) at 5%. 150μm ITR-EC exhibits stable yield stress from 5-15%, a drop to 0.5 Nmm^{-2} at 20% (which is equal to yield stress for 300μm ITR-EC at 20%). 300 μm ITR-EC experienced a gradual increase in yield stress, after the initial sharp drop at 5% with increasing volume ratio. A stable yield stress of 0.5 Nmm^{-2} was recorded from 15-25%.

Figure 7 shows that 150 and 300 μm ITR-ECs exhibit lower tensile strength for all volume content considered when compared with 40 Nmm^{-2} for pure epoxy. Ku et al. (2008) concluded that maximum tensile strength for the phenol formaldehyde composite exhibited maximum tensile strength at about 10%, it was also discovered that maximum tensile strength for 150 and 300 μm ITR-EC is achievable at 15%. It is interesting to note that 150 and 300 μm ITR-EC show similar tensile strength of 32 Nmm^{-2} at 10%.

Figure 8 shows that after the initial drop at 5% for 150 μm ITR-EC, increase in Young's modulus with % volume content of iron ore tailings till 25% was observed. 300 μm ITR-EC on the other hand, experienced a fluctuating trend in Young's modulus with increasing % volume content of iron ore tailings; with the greatest value of 1502.77 Nmm^{-2} at 30%. This trend shows that the reduction of percentage volume addition of iron ore tailings in epoxy reduces the modulus of elasticity of the composite. Low percentage volume inclusion of iron ore tailings to epoxy acts like the inclusion of impurities to pure epoxy due to the heterogeneous nature of the filler.

The elongation at break versus volume fraction of 150 and 300 μm iron ore tailings in epoxy is presented in Figures 9 and 10, respectively. It can also be seen that the Nielsen's (1996) model gives a poor representation for all ITR-ECs considered. This could be attributed to the fact that the composite produced has irregular shaped particles as filler with no binding agent contrary to the Nielsen model which assumes a spherical shaped particle and perfect adhesion.

Figures 11 and 12 show the result of tensile strength versus volume of 150 and 300 μm iron ore tailings in epoxy, respectively. It may be seen that Bigg's (1979) model does not give an exact presentation of the tensile strength. Approximate representation of tensile strength by Bigg's model can be obtained for composites produced from 150 μm iron ore tailings due to better dispersion of experimental data especially between 10 and 30% volume content.

The modulus of elasticity versus volume of 150 and 300 μm iron ore tailings in epoxy are presented in Figure 13 and 14, respectively. The experimental results are compared with values calculated from the Einstein equations. It can be seen that Einstein equations which assume perfect adhesion give poor prediction of modulus of elasticity compared with the Einstein model which assumes poor adhesion when both are compared with the modulus of elasticity. This trend could be attributed to the fact that the epoxy and filler have poor adhesion due to the absence of a binding agent. Predictability of the model seems to improve with increased particle size for Einstein model that assumes poor adhesion between particles and polymer.

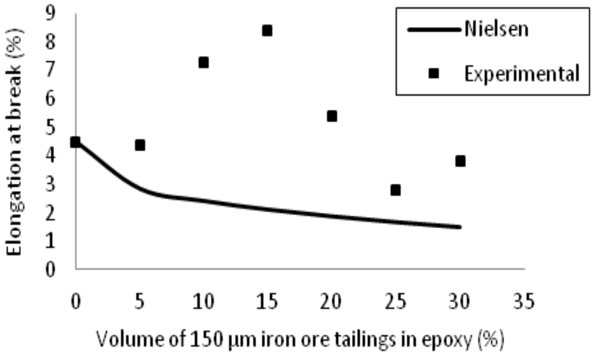

Figure 9. Elongation at break versus volume of 150 µm iron tailings in epoxy

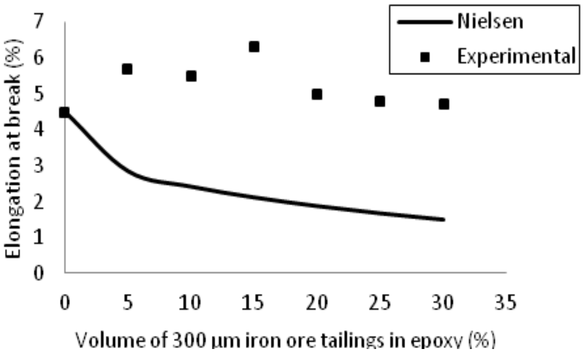

Figure 10. Elongation at break versus volume of 300 µm iron tailings in epoxy

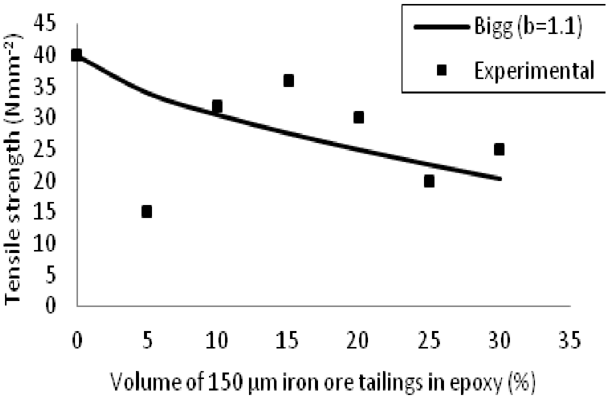

Figure 11. Tensile strength versus volume of 150 µm iron tailings in epoxy

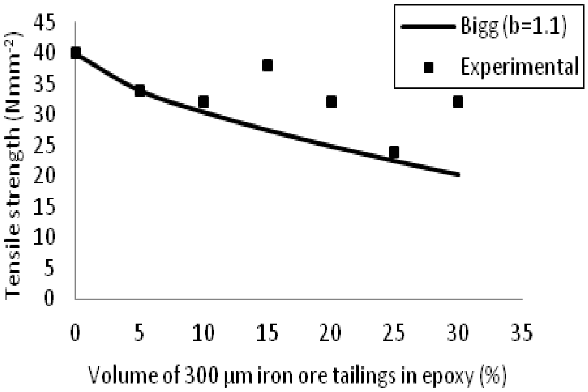

Figure 12. Tensile strength versus volume of 300 µm iron tailings in epoxy

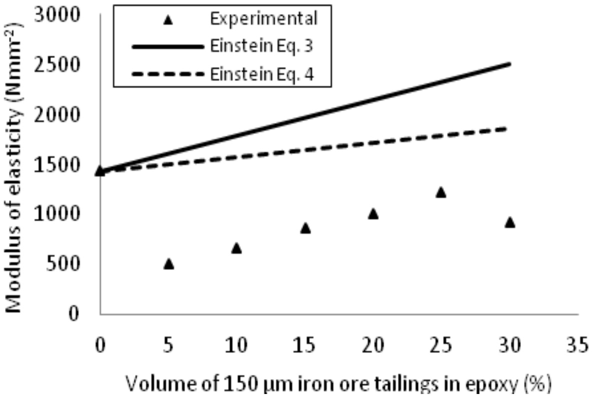

Figure 13. Modulus of elasticity versus volume of 150 µm iron tailings in epoxy

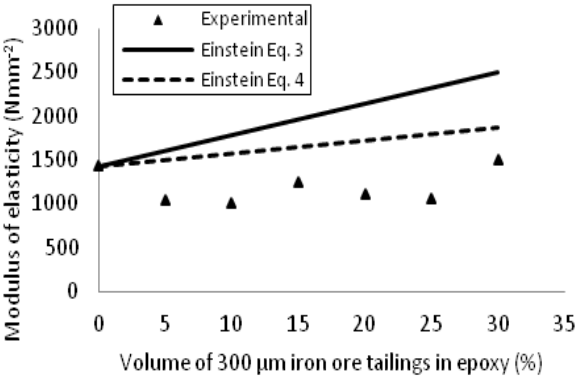

Figure 14. Modulus of elasticity versus volume of 300 µm iron tailings in epoxy

6. Conclusion

The 30 % volume content of 300 µm particle size of iron ore tailings is the better combination that can be added to epoxy to give maximum Young's modulus. Though tensile strength for ITR-ECs produced is lower when compared with pure epoxy, better tensile strength with increased particle size was recorded for all % volume content considered. On the other hand, better yield strength was recorded at reduced particle size and increasing the % volume content. Contrary to Nakamura et al. (1992)'s work which shows increased tensile strength with decreased particle size, it was observed that tensile strength reduces with decreased particle size. This could be attributed to the fact that Nakamura et al. (1992) used a homogeneous filler (i.e. angular shaped silica), whereas this present investigation used a heterogeneous filler (see Table 3) which could have a significant effect on the behaviour of the composite produced.

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Authors' Biographical Notes:

Segun Mathew Adedayo *is presently an Associate Professor of Mechanical Engineering with The University of Ilorin, Nigeria, where he had been lecturing for over 22years. Dr. Adedayo holds a B.Eng. and an M.Eng. (Production) Degree in Department of Mechanical Engineering from Ahmadu Bello University, Nigeria. He also has a Ph.D. in Mechanical Engineering from The University of Ilorin, Nigeria. Dr. Adedayo worked as a Gas Equipment Maintenance Engineer at Ajaokuta Steel Company, Nigeria and as a Trainee Engineer at Zaparostal plant, U.S.S.R. He also taught at Kaduna Polytechnic, Nigeria. Dr. Adedayo's specific areas of interest include Residual Stresses in Structures, Mechanical Testing of Engineering Materials, Service Quality and Reliability of Weldments, Welding operations, Metal Forming, Mechanical Vibration, Casting and Engineering Design.*

Modupe Adeoye Onitiri *is presently a Lecturer at the Department of Mechanical Engineering, University of Lagos,* *Nigeria. He holds a B.Eng. and an M.Eng. (Production) Degree in Mechanical Engineering from The University of Ilorin, Nigeria. He had worked as an Industrial Trainee in OSO/NGL Department, Mobil Producing Nigeria Unlimited (now known as ExxonMobil Nigeria Unlimited). Currently, he is a PhD candidate in Department of Mechanical Engineering, University of Ilorin, Nigeria, working on development of particle reinforced plastics composites, mechanical testing and finite element modelling of their mechanical properties. His specific areas of interest include Design and Construction of Composite Material Production Equipment, Development of Plastics Composites, Recycling of Wastes, Mechanical Testing of Materials and Finite Element Modelling.*

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