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# Mechanical Properties of Iron Ore Tailings Filled-Polypropylene Composites

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## ABSTRACT

Iron ore tailings filled polypropylene (PP) composites were produced using the compo-indirect squeeze casting (C-ISC) process. Particle sizes 150, 212 and 300 µm where considered for different volume fractions of 5% to 30% at intervals of 5%. The tensile and impact behavior of the produced composites were investigated, experimentally, by carrying out uniaxial tensile and izod impact tests to obtain tensile strength, elongation at break, modulus of elasticity and impact strength. Empirical data were compared with results obtained from models proposed by Nielsen, Bigg and Einstein. The experimental results show that elongation at break for iron ore tailings filled PP reduces with increasing 150 µm particle size. Tensile strength reduces with increasing filler. The Bigg equation exhibited improved predictability with decreasing particle size of filler in PP; while the Einstein equation which assumes poor adhesion gives the best prediction of modulus of elasticity with increasing particle size in PP. Izod impact strength decreases with particle size but increases with increasing volume content of iron ore tailings from 5% to 25% for each particle size considered.

**Keywords:** Compo-Indirect Squeeze Casting; Iron Ore Tailings; Polypropylene; Izod; Composite

## 1. Introduction

Particle reinforced plastics composites (PRPCs) are composites to which fillers (discrete particles) have been added to modify or improve the properties of the matrix and/or replace some of the matrix volume with a less expensive material. Common applications of PRPCs include structural materials in construction, packaging, automobile tyres, medicine, etc. Determination of effective properties of composites is an essential problem in many engineering applications [1,2]. These properties are influenced by the size, shape, properties and spatial distributions of the reinforcement [1,3,4].

Among the various studies carried out with particle filled PP worth mentioning, are works by Maiti and Mahapatro [5,6] on the tensile and impact behavior of nickel-powder-filled PP and $CaCO_3$ filled PP composites. It was discovered that the addition of nickel-powder causes decrease in tensile modulus, tensile strength and elongation-at-break with increasing filler. In the case of the addition of $CaCO_3$, tensile modulus increased while tensile strength and elongation-at-break decreased with increasing filler. Izod impact strength for the composites at first increased up to a critical filler content, beyond which the value decreased inappreciably. In a related work, Tavman [7], performed tensile test on aluminum powder reinforced high-density polyethylene composites. The empirical data obtained were compared with theoretical findings from the Nielsen's, Bigg's and Einstein's mathematical models. He discovered that Einstein's model explains the experimental results below 12% volume content of aluminum particles quite well.

This present work intends to investigate, experimentally, the tensile and impact behavior of PP filled with iron ore tailings. Empirical data obtained from the tensile test will be compared with the Nielsen's, Bigg's and Einstein's models. The effect of particle size which was not considered in Tavman's work will also be considered.

## 2. Theory

Among the challenges which particle reinforced plastics composites (PRPCs) present is the complexity of their mechanical behavior, particularly during plastic deformation. This makes it difficult to predict performance analytically and hence leads to conservative designs and extensive test programmes [8].

The tensile behavior 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

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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 [9], if both constituent materials have material properties of the same order of magnitude or if 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 [3] 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 [4,9].

According to Nielsen [10], 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:

$$\varepsilon_c = \varepsilon_p \left(1 - \phi^{1/2}\right) \quad (1)$$

where $\varepsilon_c$ is the elongation at break of the composite, $\varepsilon_p$ is the elongation at break of the unfilled polymer while $\phi$ is the percentage volume fraction of the filler.

Bigg [11] 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:

$$\sigma_c = \sigma_p \left(1 - b\left(\phi^{2/3}\right)\right) \quad (2)$$

where $\sigma_c$ is the tensile strength of the composite, $\sigma_p$ 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 [12] 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:

$$E_c = E_p \left(1 + 2.5\phi\right) \quad (3)$$

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

$$E_c = E_p \left(1 + \phi\right) \quad (4)$$

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

## 3. Experimental

### 3.1. Materials

The matrix used is commercial polypropylene with a density of 0.91 Mg/m$^3$ in pellet form produced by Lotte Daesan Petrochemical Corporation under the brand name "Séetec". The filler is iron ore tailings in particle form and approximately irregular in shape with particle sizes 150, 212 and 300 µm from iron ore beneficiation plant in Itakpe, Kogi State in the Middle belt region of Nigeria.

### 3.2. Iron Ore Tailings Preparation

The iron ore tailings was dried at room temperature 30˚C ± 2˚C and 50% ± 5% relative humidity for a minimum of 40 hours prior to testing [13,14]. The different particle sizes were generated using standard ASTM laboratory sieves [15,16].

### 3.3. Production of ITR-PPC Tensile Test Specimens

The dimensions of the ITR-PPC tensile test specimens are in conformity with BS 18 specimen specification [17]. The ITR-PPC tensile test specimens are dumb-bell shaped with circular cross section and 64 mm long.

The compo-indirect squeeze casting (C-ISC) process was used to produce the ITR-PPC tensile test specimens. Five specimens were produced for each mix ratio 5% to 30% at intervals of 5% for the three particle sizes considered.

The particle volume fraction was calculated for the ITR-PPC using the relationship [7]:

$$\phi = \frac{\varphi}{\varphi + \left(1 - \varphi\right) \cdot \dfrac{\rho_{par}}{\rho_{mat}}} \quad (5)$$

where, $\varphi$ is the weight fraction of particle, $\rho_{mat}$ is the density of matrix, and $\rho_{par}$ is the density of particle.

The weight fraction of particle, $\varphi$, was determined using a OHAUS digital scale with an accuracy of 0.01 g. The density of the particle, $\rho_{par}$, was measured at room temperature based on the Archimedes principle with water as the immersion medium. $\rho_{par}$ was calculated from [18]

$$\rho_{par} = \rho_{wat} \left(\frac{D}{M - S}\right) \quad (6)$$

where, $\rho_{wat}$ is the density of water, *D* is the dry mass of particle, *S* is the mass of particle suspended in water, *M* is the mass of particle saturated with water.

For the C-ISC process, required quantity of PP was poured into the crucible and placed on the heating element in the melting chamber. The chamber was covered using the transparent screen (with the stirrer already fixed to it). The temperature monitoring and control unit was plugged to the AC mains and switched on. The dial, initially at zero, was set to 170˚C and the PP melted. This temperature produces a gelatinous state which is the preferred condition for further processing in the production rig. Then appropriate quantity of iron ore tailings was added to the melt and stirred thoroughly to obtain a good blend. The melt was stirred to obtain a good mix and even distribution of heat. Hasty addition of the tailings could lead to increased melting time which could burn the PP.

Prior to melting the PP and adding the iron ore tailings, the metal mould and barrel were preheated to temperatures of 100˚C - 120˚C and 170˚C, respectively. The temperature of the barrel was kept at the same temperature as the melt to allow for easy flow of the molten mix. Mould temperature above 120˚C produced specimens that were brittle and flaky with irregular geometry. After the required mix had been achieved, the plunger is pulled out of the barrel and the mix poured into the barrel through the funnel at a steady continuous rate to prevent turbulence which could lead to air pockets developing in the cast. The plunger is then pushed into the barrel at a steady rate of approximately 3.75 mm/s and pressure of 27 kN/mm$^2$. After two hours the mould was dismantled to remove the cast. This procedure was carried out for different particle sizes and corresponding volume content of iron ore tailings.

### 3.4. Tensile Test

The tensile test was carried out as specified in ASTM D 638 at a test speed was 1.30 mm/min [19]. The tensile test was carried out under standard laboratory atmosphere [13,14] 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 [14,15,20].

### 3.5. Izod Impact Test

The impact test was carried out as specified in ASTM D 256 under standard laboratory atmosphere on an Avery-Denison 6705/U series impact testing machine [21]. Prior to testing, the impact test specimens were conditioned at room temperature 30˚C ± 2˚C and 50% ± 5% relative humidity for a minimum of 40 hours [14,15].

## 4. Results and Discussion

**Tables 1-3** and **Figures 1-3** and **5** were obtained from the uniaxial tensile test carried out on the produced composites. **Tables 1-3** show the tensile stress-strain results for 150, 212 and 300 µm particle size iron ore tailings reinforced polypropylene composites (ITR-PPC) while **Figure 1** shows the stress-strain curves for pure polypropylene (PP) and PP reinforced with 5% and 15% iron ore tailings.

It can be seen in **Table 1** that inclusion of 150 µm particle size in PP causes reduction in yield, ultimate and fracture stress and strain compared with pure PP (see **Figure 1**). 15% ITR-PPC exhibited the highest strain value at ultimate and fracture point while 5% ITR-PPC has the highest stress at ultimate and fracture point for all mix ratio considered. **Table 2** shows that the addition of 212 µm particle size in PP leads to drop in tensile properties of the composites when compared with pure PP. ITR-PPC exhibited the highest rigidity and plastic deformation with the addition of 5% iron ore tailings. **Table 3** shows that ITR-PPC experiences reduced yield

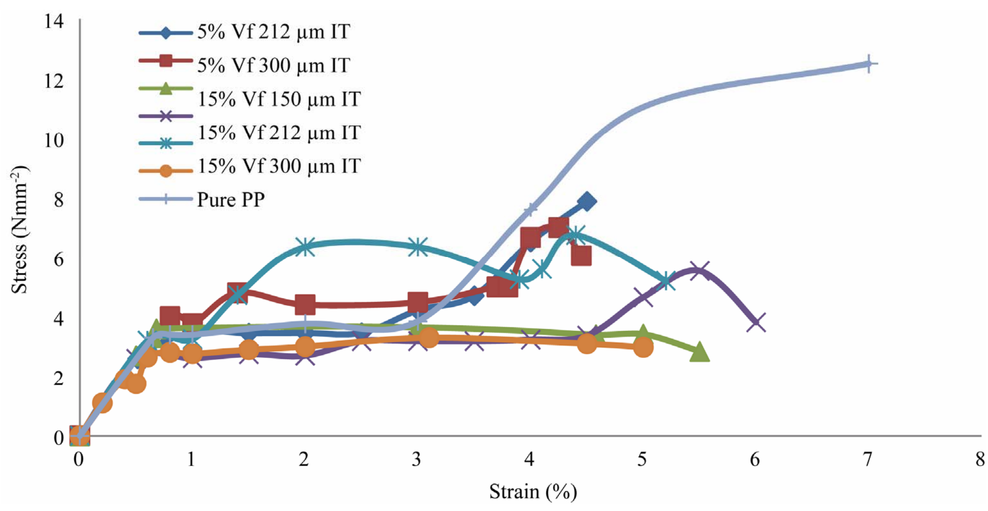


**Figure 1. Stress-strain curves of pure PP and ITR-PP composites with particle sizes 150 µm, 212 µm and 300 µm at volume content 5% and 15% of iron ore tailings.**

 

**Table 1. Stress-strain results (tensile) for 150 μm particle size ITR-PPC.**

| | Volume ratio of iron ore tailings (%) | | | | | | | | | | | | | | Min. | Mean | Max. | S.D |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | | 5 | | 10 | | 15 | | 20 | | 25 | | 30 | | | | | |
| Stress at yield (MPa) | 3.40 | +0.10<br>−0.10 | 2.60 | +1.20<br>−1.41 | 2.42 | +0.58<br>−1.12 | 2.59 | +0.91<br>−0.32 | 3.63 | +0.87<br>−0.23 | 2.95 | +0.5<br>−0.23 | 2.95 | +1.55<br>−1.55 | 2.42 | 3.42 | 3.63 | 0.69 |
| Stress at ultimate point (MPa) | 12.05 | +1.05<br>−1.05 | 7.38 | +1.38<br>−1.38 | 6.84 | +0.96<br>−0.84 | 5.33 | +0.17<br>−0.13 | 5.92 | +1.08<br>−1.08 | 5.05 | +3.51<br>−1.29 | 5.25 | +0.75<br>−0.75 | 5.05 | 7.97 | 12.50 | 2.08 |
| Stress at fracture (MPa) | 12.05 | +1.05<br>−1.05 | 7.30 | +0.00<br>−0.00 | 5.70 | +4.30<br>−4.30 | 3.87 | +0.37<br>−0.37 | 5.92 | +0.00<br>−0.00 | 2.56 | +0.13<br>−0.13 | 2.55 | +0.25<br>−0.25 | 2.55 | 6.66 | 12.50 | 2.45 |
| Strain at yield (%) | 1.00 | | 0.51 | | 0.50 | | 0.50 | | 0.80 | | 1.00 | | 0.60 | | 0.50 | 0.82 | 1.00 | 0.25 |
| Strain at ultimate point (%) | 7.00 | | 4.50 | | 4.00 | | 5.50 | | 5.00 | | 3.70 | | 3.60 | | 3.60 | 5.55 | 7.00 | 1.08 |
| Strain at fracture (%) | 7.00 | | 4.50 | | 4.50 | | 6.10 | | 5.00 | | 5.00 | | 5.00 | | 4.50 | 6.18 | 7.00 | 1.06 |

**Table 2. Stress-strain results (tensile) for 212 μm particle size ITR-PPC.**

| | Volume ratio of iron ore tailings (%) | | | | | | | | | | | | | | Min. | Mean | Max. | S.D |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | | 5 | | 10 | | 15 | | 20 | | 25 | | 30 | | | | | |
| Stress at yield (MPa) | 3.40 | +0.10<br>−0.10 | 2.90 | +1.10<br>−1.10 | 1.50 | +0.57<br>−1.73 | 1.37 | +1.33<br>−0.27 | 2.20 | +0.80<br>−0.70 | 0.76 | +0.01<br>−0.01 | 0.98 | +1.02<br>−0.73 | 0.76 | 2.18 | 3.40 | 0.92 |
| Stress at ultimate point (MPa) | 12.05 | +1.05<br>−1.05 | 4.30 | +1.33<br>−1.33 | 4.13 | +0.13<br>−0.13 | 5.23 | +0.97<br>−0.98 | 4.13 | +0.27<br>−0.13 | 4.40 | +0.60<br>−1.40 | 3.77 | +0.27<br>−0.63 | 3.77 | 6.33 | 12.50 | 1.85 |
| Stress at fracture (MPa) | 12.05 | +1.05<br>−1.05 | 4.50 | +1.33<br>−1.33 | 1.70 | +0.00<br>−0.00 | 4.48 | +0.25<br>−0.25 | 1.49 | +0.41<br>−0.33 | 2.10 | +0.40<br>−0.40 | 2.75 | +0.05<br>−0.05 | 1.48 | 4.85 | 12.50 | 2.13 |
| Strain at yield (%) | 1.00 | | 0.80 | | 0.10 | | 0.55 | | 0.60 | | 0.45 | | 0.50 | | 0.10 | 0.67 | 1.00 | 0.27 |
| Strain at ultimate point (%) | 7.00 | | 4.30 | | 7.00 | | 3.00 | | 3.00 | | 2.00 | | 2.00 | | 2.00 | 4.72 | 7.00 | 1.92 |
| Strain at fracture (%) | 7.00 | | 4.50 | | 4.00 | | 4.30 | | 4.00 | | 4.60 | | 3.00 | | 3.00 | 5.23 | 7.00 | 1.02 |

**Table 3. Stress-strain results (tensile) for 300 μm particle size ITR-PPC.**

| | Volume ratio of iron ore tailings (%) | | | | | | | | | | | | | | Min. | Mean | Max. | S.D |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | 0 | | 5 | | 10 | | 15 | | 20 | | 25 | | 30 | | | | | |
| Stress at yield (MPa) | 3.40 | +0.10<br>−0.10 | 3.45 | +0.15<br>−0.15 | 2.30 | +0.49<br>−0.50 | 1.75 | +0.90<br>−0.75 | 2.07 | +0.43<br>−0.28 | 1.60 | +1.00<br>−0.50 | 1.25 | +0.25<br>−0.25 | 1.25 | 2.64 | 3.45 | 0.90 |
| Stress at ultimate point (MPa) | 12.05 | +1.05<br>−1.05 | 3.42 | +0.98<br>−0.92 | 5.30 | +0.30<br>−0.30 | 3.25 | +0.25<br>−0.25 | 3.04 | +0.41<br>−0.41 | 4.66 | +0.21<br>−0.21 | 2.80 | +0.10<br>−0.10 | 2.80 | 5.75 | 12.50 | 2.05 |
| Stress at fracture (MPa) | 12.05 | +1.05<br>−1.05 | 2.84 | +0.98<br>−0.92 | 5.30 | +0.30<br>−0.30 | 2.98 | +0.38<br>−0.38 | 3.04 | +0.41<br>−0.41 | 3.50 | +0.00<br>−0.00 | 2.80 | +0.10<br>−0.10 | 2.80 | 5.42 | 12.50 | 2.02 |
| Strain at yield (%) | 1.00 | | 0.67 | | 0.01 | | 0.50 | | 0.50 | | 0.50 | | 0.30 | | 0.01 | 0.58 | 1.00 | 0.27 |
| Strain at ultimate point (%) | 7.00 | | 5.00 | | 6.00 | | 3.10 | | 4.50 | | 6.00 | | 1.21 | | 1.21 | 5.47 | 7.00 | 2.05 |
| Strain at fracture (%) | 7.00 | | 5.45 | | 6.00 | | 5.00 | | 4.50 | | 7.00 | | 1.21 | | 1.21 | 6.03 | 7.00 | 1.98 |

 

stress with increasing percentage volume of iron ore tailings at a standard deviation of 0.9.

The elongation at break versus volume fraction of 150, 212 and 300 μm iron ore tailings at varying volume content in PP curves are presented in **Figure 2**. It can be seen that the Nielsen model gives poor representation with increasing percentage volume of iron ore tailings from 15% in the case of 212 μm fillers. This shows that the weak adhesion between particles and matrix of ITR-PPC, due to absence of binding agents, becomes significant at high volume concentration of fillers for Neilsen model which assumes perfect adhesion.

**Figure 3** shows the tensile strength versus volume of 150, 212 and 300 μm iron ore tailings in PP curves. Bigg's model gives poor representation of the tensile strength for all particle sizes. The relationship between experimental tensile strength and the Bigg's model seem to improve with decreasing particle size. This could be attributed to the fact that perfect adhesion, in the absence of binding agents, improves with decreasing particle size. Large particle size creates greater filler surface area to be covered by the matrix and thinner inter particle space to occupy with increasing volume concentration of fillers (see **Figure 4**).

The modulus of elasticity versus volume of 150, 212 and 300 μm iron ore tailings at varying volume content in PP curves are presented in **Figure 5**. The experimental results are compared with values calculated from the Einstein equations. Einstein equation which assumes perfect adhesion between fillers and the polymer shows

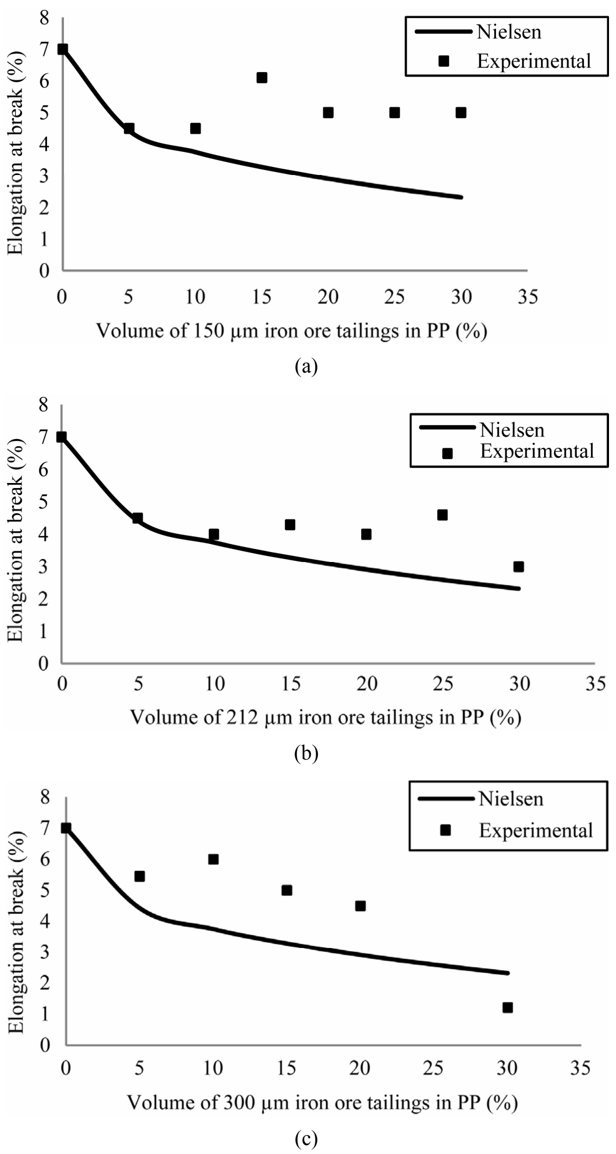


**Figure 2. Elongation at break versus volume of (a) 150 μm; (b) 212 μm; (c) 300 μm iron ore tailings in PP.**

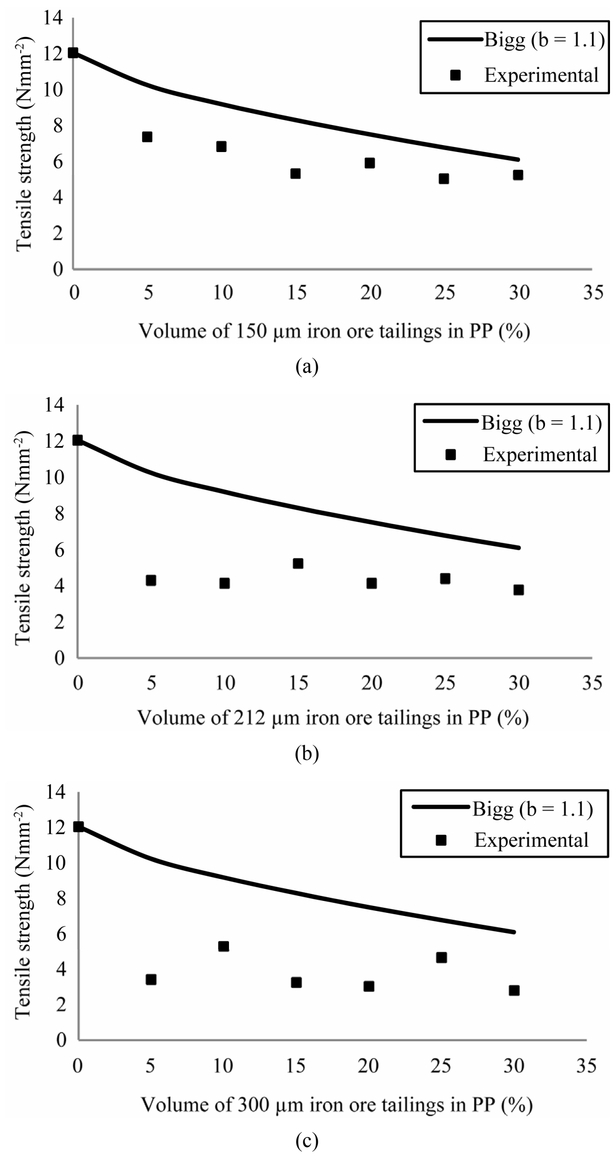


**Figure 3. Tensile strength versus volume of (a) 150 μm; (b) 212 μm; (c) 300 μm iron ore tailings in PP.**

 

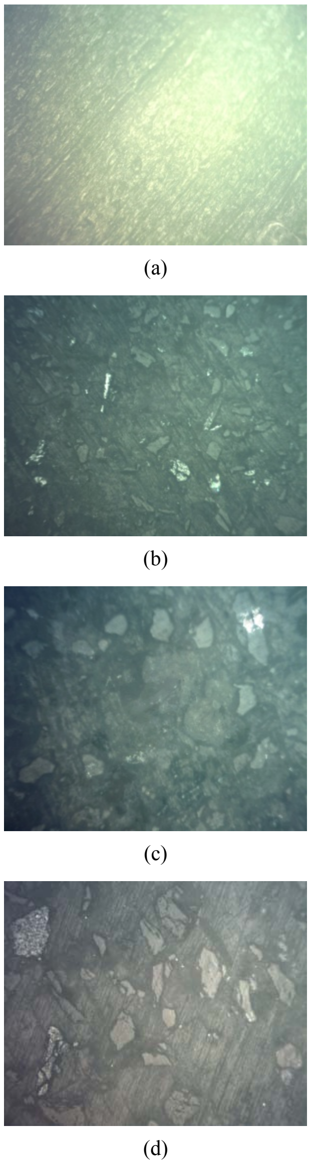


Figure 4. (a) Pure polypropylene; 25% volume content of (b) 150 μm; (c) 212 μm and (c) 300 μm iron ore tailings in polypropylene (×100).

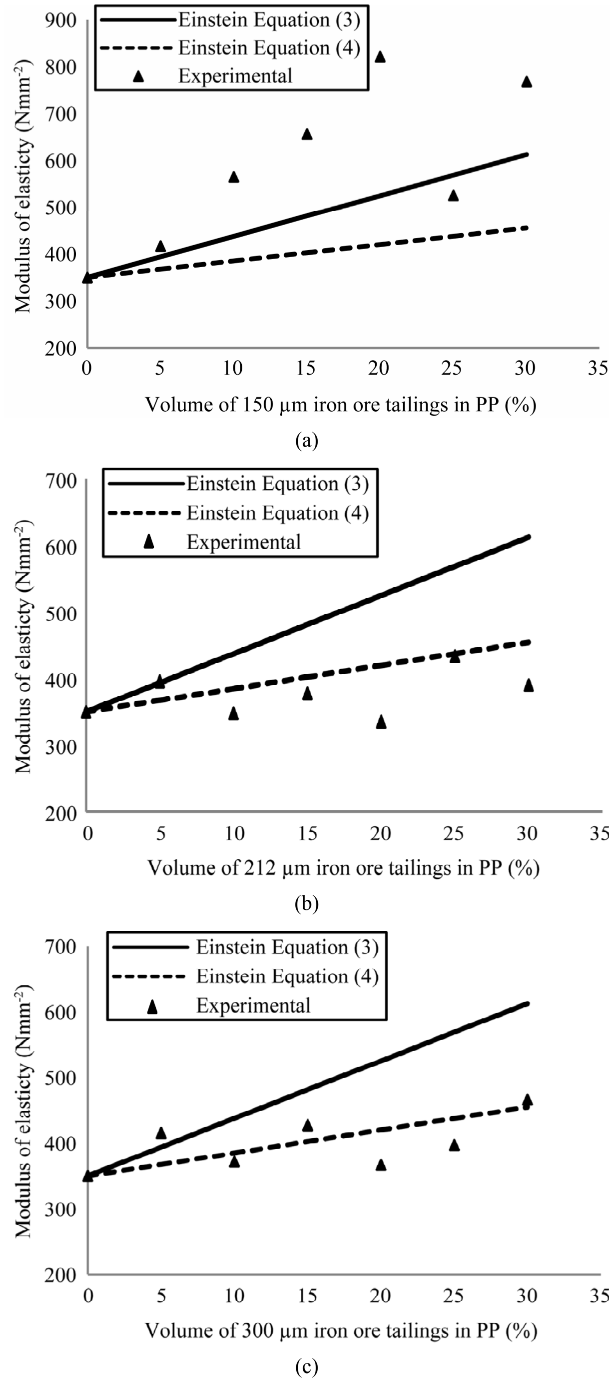


**Figure 5. Modulus of elasticity versus volume of (a) 150 μm; (b) 212 μm; (c) 300 μm iron ore tailings in PP.**

good match for volume fractions below 5% for all the particle sizes. Einstein equation which assumes poor adhesion exhibits an improved predictability with increasing particle size. This trend confirm the fact that the composite produced, as earlier stated, is expected to exhibit poor adhesion with increasing volume content of filler or particle size due to the absence of binding agent.

**Figure 6** shows the average impact energy versus volume content of iron ore tailings particle sizes 150 μm, 212 μm and 300 μm in PP. The entire specimens experienced complete break when impacted. Izod impact strength increased with increasing volume of 150 μm iron ore tailings except at 10% where average impact energy of 4.393 J was recorded compared with 4.501 J for polypropylene. Addition of 212 μm iron ore tailings causes improved impact strength for volume ratio considered; with the highest value of 4.867 J recorded at 5%. After the initial drop at 5%, addition of 300 μm iron ore tailings causes increase in impact strength with increasing filler content contrary to the trend highlighted in Maiti and Mahapatro's work on nickel-powder-filled PP and $CaCO_3$ filled PP [5,6].

 

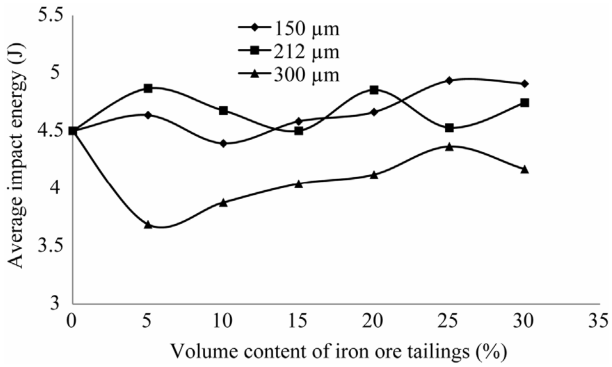


**Figure 6. Average impact energy versus volume content of iron ore tailings curves for PP-filled with 150 μm, 212 μm and 300 μm iron ore tailings particle sizes at volume content 0% to 30%.**

## 5. Conclusion

Nielson's model shows better predictive capability with the smallest particle size and decreasing volume ratio for ITR-PP. The predictability of the Nielsen's model can be enhanced by addition of binding agents to improve interfacial adhesion. The Bigg equation shows improved predictability with decreasing particle size of filler in PP while the Einstein equation which assumes poor adhesion gives the best prediction of modulus of elasticity with increasing particle size in PP. The least volume content of iron ore tailings that can be predicted by the Einstein equation which assumes perfect adhesion is 5%. Izod impact strength increased with increasing volume of 150 μm iron ore tailings except at 10% volume content of iron ore tailings.

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