Mechanical, Thermal and Morphological Properties of Recycled and Virgin PC/ wollastonite Composite and its Compatibilization by SBC

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Abstract
Composite of recycled Polycarbonate (RPC) and Virgin (PC) with various amount of (5,10 & 15 wt.%) wollastonite were prepared using co-rotating twin-screw extruder. And there mechanical, thermal and morphological properties were investigated. Styrene Butadiene block copolymer (SBC) elastomer (0,1,3,5 &7%) were used as a compatibilizer to study the effect on the properties of the composites. The results showed that wollastonite had no significant effect on the tensile strength of the composite, but drastic 30% deterioration in the impact strength was observed at 15 % loading of wollastonite. Notched izod impact strength tests indicate that SBC compatibilized blend increased the impact strength of blends to some extent. Maximum izod impact strength was noted with 7% SBC compatibilizer. Scanning electron microscopy micrographs showed the particle size of the dispersed phase was decreased in the continuous phase of PC by using the compatibilizer. A blend without compatibilizer showed brittle behavior while the blends containing compatibilizer showed ductile fracture.

1. Introduction
Polycarbonate has been a promising material for various applications such as electronic appliances, automobile industries, home appliances due to its excellent balance of properties toughness, transparency, high heat deflection temperature, dimensional stability and electrical properties.[1]–[3]

Blending of polymers with diverse types of fillers is rather a strength forward way to produce new polymer material with desired properties. Some of the materials used for these purposes with greater or smaller success are diene rubbers, ethylene copolymer elastomers, polyolefins, mixtures of rubbers with polyolefins and others.[4]–[6] Use of the particulate filled composite have increased because of the cost to performance ratio [7], [8]. The cost reduction as a primary interest of adding mineral fillers to polymers this increasing demand leads to polymer composites[9], [10]Since presence of fillers and elastomers affect the structure of the polymer matrix and change the overall properties of composites, the control of dispersion and phase morphology of composites is very important for tailoring the mechanical properties[11], [12]

Wollastonite is naturally occurring inorganic filler incorporated in plastics which leads to improvement in some mechanical properties such as strength, stiffness but reduces the impact strength.[13], [14] This drawback can be extricated with the addition of proper elastomer as an impact modifier or can act as a compatibilizer. Wollastonite had been applied as a reinforcement for PA6 [14] , Epoxy resin [15], PMMA[16] , Polystyrene [17], PVC[18]. Describe improvement in properties.

Virgin PC toughening has been premeditated using acrylonitrile butadiene styrene (ABS)[19], polymethylmethacrylate (PMMA)[20]. Kumar et al. have reported for PC/polydimethylsiloxane (PDMS)[21] and observed improvement in impact properties of virgin PC. Ramteke et.al. investigated PC/ modified acrylonitrile-styrene-acrylate (MASA)[22] terpolymer where the impact properties were increased, however, tensile strength is found to be reduced. There are also some findings available on blending of virgin PC with ABS-g-maleic anhydride (ABS-g-MA) and (ethylene–vinyl acetate)-g-(maleic anhydride) (EVA-g-MA)[23],poly(methyl methacrylate-co-butadiene-co-styrene) (MBS)[28], and poly(styrene-b-(ethylene-co-butylene)-b-styrene) (SEBS)[26]. Bagotia et al. studied mechanical properties of Virgin PC/ Ethylene Methyl
Acrylate (EMA) blends and observed the improvement in impact strength of virgin PC/EMA blend and there is a slight decrease in tensile strength without affecting other mechanical properties.[27] Also, some investigations on the modification of recycled PC have been presented. X.wang et al. investigated the recycled PC/Core-shell structural poly(methyl methacrylate-co-butadiene-co-styrene, MBS) and polymethyl methacrylate-co-methyl phenyl siloxane-co-styrene (MSiS) blends and observed improvement in impact after addition of MSiS [28].

The aim of the investigation is to evaluate simultaneously the effectiveness of the wollastonite and the influence of the SBC elastomer as a compatibilizer on the mechanical, thermal and morphological properties of PC. In the present work, test materials have been prepared a sequence of filled PC with wollastonite filler (5, 10 and 15 wt. per cent) to study the consequence of the filler content and SBC elastomer and differences found in the properties are correlated with the blend morphology.

![Figure 1: Optical Microscopy of wollastonite particles](image)

2. Material and Methods
2.1. Material
PC Makrolon ET3113 (MFI 6 gm/10min, the density of 1.2 gm/cc) was supplied by Covestro, Mumbai. Wollastonite (AF1 Fillex 7 – KFB) with d (50%) =8.2 µm procured from Wolkem India Pvt., Mumbai. Optical microscopy images of the wollastonite particles are shown in Figure 1. and Styrene Butadiene block copolymer (TPE TS6005) from Chi hung Plastics Taiwan,

2.2. Preparation of Composites:
The components for the RPC7/VPC3 blends, RPC/VPC/Wollastonite composite, RPC/VPC/Wollastonite/SBC composites formulations of the composite showed in Table 1 were premixed by tumble mixing and subsequently melt-blended in twin screw co-rotating extruder and temperature profile from the hopper to the die as 180°C, 190°C, 220°C, 230°C, and 250°C respectively with a speed of 80 rpm. Extruded strands were water cooled at 30°C and pelletized. Pellets obtained were carried for injection molding after pre-drying at 120°C for 4-5 hrs.

<table>
<thead>
<tr>
<th>Sr.no.</th>
<th>Blend code</th>
<th>Recycled PC / Virgin PC</th>
<th>Wollastonite (% wt.)</th>
<th>SBC (% wt.)</th>
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<tbody>
<tr>
<td>1</td>
<td>RPC7/VPC3</td>
<td>70/30</td>
<td>-</td>
<td>-</td>
</tr>
<tr>
<td>2</td>
<td>RPC7/VPC3W5</td>
<td>70/30</td>
<td>5</td>
<td>-</td>
</tr>
<tr>
<td>3</td>
<td>RPC7/VPC3 W10</td>
<td>70/30</td>
<td>10</td>
<td>-</td>
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<td>70/30</td>
<td>15</td>
<td>-</td>
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<td>10</td>
<td>3</td>
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<td>5</td>
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<tr>
<td>8</td>
<td>RPC7/VPC3 W10C7</td>
<td>70/30</td>
<td>10</td>
<td>7</td>
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</table>
Injection molding (Toshiba Machinery India Ltd, Mumbai, India) was done maintaining temperature profile of 190°C, 210°C, 230°C and 260°C from the hopper to the injection nozzle respectively. Standard ASTM based specimens for tensile, flexural and impact testings were obtained from injection molding. The amounts of Wollastonite in the 70 virgin PC/ 30 Recycled PC blend and composites samples were 5, 10, and 15 wt. %, whereas the amounts of SBC in composites were 1, 3, 5, and 7 %.

3. Characterization Techniques

Mechanical tests

Dumbbell-shaped specimens were imprimed to a tensile test using LLOYD UTM with 50 kN load to determine tensile strength, % elongation as well as the Young modulus. The crosshead speed was 50 mm/min. Specimens dimension (Type 1) as per ASTM D638 was 165 mm × 13 mm × 3.2 mm for flexural strength specimen dimension as per ASTM D790 was 125 mm × 12.7mm×3.2mm. 3-point flexure test fixture was used. The grips were displaced at a rate of 10 mm/min to cause the flexure. For Izod Impact Test Sample dimension was 50 mm × 13 mm × 3.2 mm. Numbers were consequential from three replicas of the same samples producing close results.

Differential scanning calorimetry (DSC)

To examine the melting behaviour of neat polymers and their composites, differential scanning calorimetry (TA Q100, USA) was used. The samples were heated from room temperature to 200°C at 10°C min⁻¹ and kept for 2 min at this temperature to obliterate thermal history. The melting tests were run at a heating rate of 10°C min⁻¹ from 0°C to 200°C. The second melting curve was recorded. The weight of each sample was 5 mg.

Rheological Properties

Rheological properties of the prepared composite were determined by using a parallel plate rheometer (MCR101; Anton Paar, Austria). Round specimens of a diameter of 25 mm equivalent to the diameter of parallel plates (PP35-SN20785) of the rheometer were used. The distance between the parallel plates was maintained as 1 mm. Data analysis was performed using the software Rheoplus/32 V3.40 supplied by the manufacturer.

Scanning electron microscopy (SEM)

The cross-section of Tensile fractured specimens was characterized by SEM (scanning electron microscopy) on a FEI Quanta 200 ESEM model.

4. Results and Discussion

Tensile properties

Tensile properties deliver information about the behaviour of the material when it is exposed to stretching or pulling force before it miscarries. In this work, the mechanical properties such as tensile, flexure and impact testings were used to evaluate the strength and toughness. virgin PC/ recycled PC and virgin PC/ recycled PC wollastonite filled and their hybrid composites with SBC. The characterization of the composites discloses the insertion of fillers has a fervent influence on the mechanical properties of composites. By incorporating a filler into the virgin PC/ recycled PC and composites, synergistic effects, as expected were achieved in tensile and flexural properties. Tensile properties of virgin PC/ recycled PC composites are shown in Figure 2. The inclusion of wollastonite into virgin PC/ recycled PC has no such deterioration the tensile properties at 10 % loading of wollastonite, and the addition of SBC into composites also show the deterioration of tensile properties.[29] The addition of SBC in the composites deteriorated their tensile properties by 12 % in 7% loading of SBC with 10 % wollastonite shown in Figure 3. cause maximum reduction in the composite’s tensile strength which may have occur due to the poor interface bonding between the matrix, filler resulting in poor adhesion of particles and which are not good to transfer the tensile stress.[14][30].

Flexural properties

Flexural properties provide information about the behaviour of the material in bending. The tested result of flexural strength and flexural modulus values of virgin PC/ recycled PC and virgin PC/ recycled PC wollastonite filled shown in Figure 4 and their hybrid composites with SBC shown in Figure 2 & 3. The wollastonite filled composite showed improved flexural properties due to the fibrous structure of filler.[16][31] The result in the investigation reveals that wollastonite filled PC exhibits better flexural properties than SBC compatibilized system as the SBC increasing the flexibility. This may be due to the even dispersion of filler.
Figure 2. Effect of various concentration of wollastonite on Tensile Strength, Flexural Strength and Elongation of RPC/VPC.

Figure 3. Effect of various concentrations of wollastonite and SBC on Tensile Strength, Flexural Strength and Elongation of RPC/VPC.

Figure 4. Effect of various concentration of wollastonite and SBC on Modulus of RPC/VPC.

The functional load penetrates the matrix material and shifted around the filler instead of penetrating it.[32] This composition could act as very good reinforcing filler in the composites. Also, the stresses are established by these fillers, hence, which lead to reasonable deflection.

**Impact strength**

It is the ability of the material to resist fracture under stress applied at high speed. The specimens are deformed within a short time and therefore exposed to high strain rates. Impact strength of virgin PC/ recycled PC and virgin PC/ recycled PC wollastonite filled and their hybrid composites with SBC are shown in Figure 5. The notch causes reduction in impact strength of PC. It is observed that in wollastonite filled PC there is a drastic decrease in impact strength as the amount of wollastonite increasing the impact strength is decreasing.[7][33] In the case of wollastonite with SBC, there is an improvement in impact strength, but the improvement is very low. The addition of 15% of wollastonite to RPC7-VPC3 reduces the impact strength to 150 J/m. The gathering of elastomers to the PC/wollastonite composites steadily increases the notched impact strength. Thereby, the composites with 7% of elastomer exhibit an increase of impact strength range from 162 J/m to 182 J/m. Although both the wollastonite leads to a reduction in impact strength and SBC elastomers are effective as an impact modifier, the composites with wollastonite with SBC shows better impact strength than the composites without SBC.[34] SBC is additional effectual impact modifier in the present case due to its soft elastomeric interlayers around filler particles, which can act as bumpers that absorb the impact energy and, also play as crack originator stopovers.[35]–[37]

![Izod Impact Strength](image)

**Figure 5.** Effect of various concentration of wollastonite and SBC on Izod Impact Strength of RPC/VPC.

**Thermal Properties**

The Glass transition temperature data of virgin PC/ recycled PC wollastonite filled and their hybrid composites with SBC are shown in Table 2. As shown in DSC curve Figure 6 (a) and (b), shows the behaviours of Tg with respective loading of wollastonite filler and SBC copolymer loading in PC. The VPC shows Tg at 144°C and RPC shows at 141°C while the 30/70 blend of VPC and RPC shows no change in Tg. DSC data reported in the table shows that, as the filler loading increases into the blend of VPC and RPC, the Tg value tends to be reduced from 144°C to 131°C. Similarly, loading of SBC in wollastonite filled PC shows the lowering in Tg value due to soft segments of SBC by elastomeric domains.[13][38] The flexible and Long chain SBC chain in a PC increases the free volume and acts as a compatibilizer for filler.[13][39] The mechanical properties also supported the DSC data with increases in % elongation with increases in elastomer loading and lowering in Tg.
Table 2. DSC results of PC wollastonite and Compatible blends.

<table>
<thead>
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<th>Sr. No</th>
<th>Batches</th>
<th>Tg (°C)</th>
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<tbody>
<tr>
<td>1</td>
<td>VPC</td>
<td>144</td>
</tr>
<tr>
<td>2</td>
<td>RPC</td>
<td>141</td>
</tr>
<tr>
<td>3</td>
<td>RPC7/VPC3</td>
<td>144</td>
</tr>
<tr>
<td>4</td>
<td>RPC7/VPC3 W5</td>
<td>141</td>
</tr>
<tr>
<td>5</td>
<td>RPC7/VPC3 W10</td>
<td>137</td>
</tr>
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<td>6</td>
<td>RPC7/VPC3 W15</td>
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</tr>
<tr>
<td>10</td>
<td>RPC7/VPC3 W10 C7</td>
<td>139</td>
</tr>
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</table>

Figure 6. DCS Curves of Wollastonite filled PC (a) and SBC compatibilized PC wollastonite Composite (b)

Rheology
The shear viscosity as a function of shear rate for PC with different loading of wollastonite filler and SBC compatibilizer is shown in Figure 7. It is seen that there is a gradual increase in viscosity with the addition of elastomer. In all cases, the shear thinning behaviour is observed just as in PC. It is evident that there is a sudden rise in viscosity even at low concentration of SBC (1%) and it continues to remain high for other concentration.

Figure 7. Viscosity Vs shear rate of RPC 70/VPC 30-Wollastonite 10% / SBC (1,3,5& 7)
However, for SBC the increase of viscosity is more gradual up-to concentration of 5%, but it decreases beyond this for higher concentration. The addition of fillers and elastomer to polymer melt is known to increase the viscosity and there is several the thermoticalmodels suggested explaining the same [40][41]. The storage and loss modulus (G’ and G”) were determined from the rheometer for all composition Figure 8 and 9 shows the plot of G’ and G” with radial frequency for SBC compatibilized PC filled with wollastonite it is seen that the curves for G’, which are much below the G” curves at lower frequency crossover and go to the higher values above the G”. the crossover points for G’ and G” is well reported for polymer containing fillers it indicates the change of viscoelastic behaviour of dispersion going from flowing melt to semisolid like behaviour and this shows the good compatibilization of PC with 5 % SBC loading.

Figure 8. Storage Modulus Vs Angular Frequency of RPC 70/VPC 30-Wollastonite 10% / SBC (1,3,5&7)

Figure 9. Loss Modulus Vs Angular Frequency of RPC70/VPC30-Wollastonite 10% / SBC (1,3,5& 7)
Morphology

Scanning Electron Microscopy (SEM) examination of tensile fracture surface of Polycarbonate composite with wollastonite showed that the wollastonite inclusion and the Figure 10. (b) & (a) showed micrographs of PC only in which smooth surfaced was observed.[42] While the SBC compatibilized system the wollastonite inclusion is better with the soft SBC boundary and the dispersion (marked by arrow) of the wollastonite seems good as compare to without SBC system shown in figure (c).[39][43][16] The dispersion of SBC leads to softening of the system and increase the flexibility which evident to the increase in impact properties and marginal decrease in tensile properties.

Figure 10. SEM photographs of the tensile fracture surface morphologies of Polycarbonate (a) blend with wollastonite filler 10 wt. % (b) SBC compatibilized (c)

Conclusions

This paper studies tensile, elongation, flexural, izod impact properties, DSC and surface morphology data for VPC/RPC (70/30) and VPC/RPC (70/30) with wollastonite and SBC rubber as a compatibilizer. Virgin and recycled PC with only wollastonite showed an increase in Flexural properties but observed a drastic decrease in impact properties. Higher wollastonite concentration resulted in higher brittleness and lower the elongation. There is no variation found in Tensile properties. PC with wollastonite and rubber showed a slight improvement in impact properties. 1% SBC rubber showed highest tensile strength there is no such change in flexural
properties. As the concentration of SBC rubber increasing, there is an obvious increase in % elongation due to the soft segment of SBC. Scanning electron microscopy studies showed the filler inclusion and the distribution with and without compatibilizer that is SBC rubber. Soft distribution of SBC surrounds the filler which improves the adhesion of filler with PC and support to increase the flexibility of the system 7% SBC with 10 % wollastonite showed the promising results.

References

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