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STUDIES ON MECHANICAL AND MORPHOLOGICAL PROPERTIES OF POLY (PHENYLENE OXIDE) FILLED WOLLASTONITE MINERAL COMPOSITES

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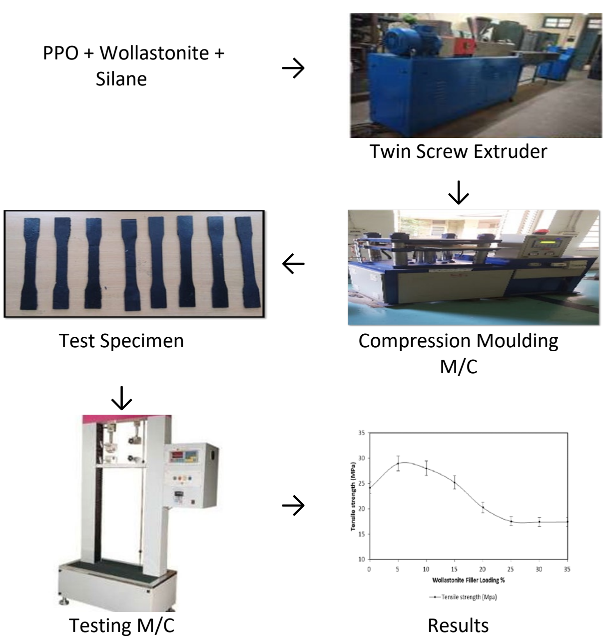
Graphical abstract



Abstract

Poly (phenylene oxide) (PPO) polymer has outstanding strength, chemical resistance, and heat distortion temperature by virtue of which it has been very useful for industrial and automobile applications. In this work PPO composite of Wollastonite, with and without coupling agent has been devised. The study enunciates that Young's modulus, tensile strength and extension at break of this composite [PPO + Wollastonite (25µm) 10% by wt] has increased and also cost saving of 10% has been reported. However, impact strength and MFI have been found to decrease marginally with the increase in % loading of Wollastonite in the polymer. The samples were analyzed for microstructure using scanning electron microscope (SEM). The values of 2-4% Silane modified Wollastonite (10 wt%) composite showed marginal higher values of Young's Modulus, impact strength, tensile strength, and extension at break in comparison with that of untreated 10 wt% Wollastonite–filled PPO composites. At 2-4% Silane loading for 10% Wollastonite–filled PPO composites showed higher mechanical properties than PPO and untreated PPO composites. Mechanical properties of Wollastonite–filled PPO composites are decided by the filler content. Morphology of the tensile fracture surfaces of the composites establishes the improvement in interfacial interaction between filler and matrix.

Keywords: Poly (phenylene oxide), Wollastonite, Coupling Agent, mechanical properties, Scanning electron microscope (SEM).

1.0 INTRODUCTION

Particulate filled thermoplastic composites have grabbed attention of researchers and industries as it exhibits enhanced mechanical and thermal properties than that of the virgin polymer matrix. PPO (Poly-phenylene Oxide) is also known as PPE (Polyphenylene ether), a thermoplastic having good heat distortion temperature, strength, and chemical resistance.

Borodina et al., have investigated the effect of wollastonite in reinforcing the unsaturated polyester resin, on physical-mechanical and performance characteristics of the hardened composite material. It has been proved to be economical to reinforce polyester resins with inorganic mineral additives like wollastonite as it is absolutely harmless [1]. Chan et al., have done a review of mechanical properties of thermoplastic composites reinforced with wollastonite. It has been inferred that the characteristics of polymer composites with wollastonite as a filler are the function of adhesion, percentage of filler and relationship of wollastonite with the polymer. They are also function of size and shape of wollastonite particles [2]. Chan et al., have done a review of thermal and flammability behavior of wollastonite-filled thermoplastics. Wollastonite as a filler provides enhancement of mechanical strength, thermal stability, and flame retardancy. Due to these properties, it has a great potential of substituting the fillers such as glass fiber and talc. Wollastonite has good thermal stability and reinforcing ability. Polymer composites of Wollastonite (as a filler) have been very popular now a days. The review enunciates that the properties of the composites with wollastonite as a filler material are affected by the size, dispersion, shape, interfacial adhesion and loading of wollastonite with the polymer [3]. Fino et al., have studied the PPO composites with respect to their pre-screening as probable candidates in innovative ablative shields [4]. Amin and Oza have devised Nylon-6/untreated Wollastonite composites. The test results have shown that some mechanical properties and abrasion resistance of the composites have increased than that of virgin Nylon6 [5]. Wang et al., have devised composites of polyamide 6 (PA6), inorganic modified wollastonite and wollastonite particles. The test results have exhibited that the PA6 composites with WIMS as filler have better bending strength, tensile strength, bending modulus than that of plain wollastonite and virgin PA6. [6]. Singh and Ray have devised composites of polypropylene and silicon rubber with wollastonite (fibrous filler). Thermal analysis and SEM for morphology analysis reveal that the wollastonite particles (fibrous filler) were dispersed randomly in the PP matrix [7]. Svab et al., have investigated modification of polypropylene (PP) with wollastonite fillers. Binary PP/wollastonite composites were made with silane pretreated wollastonite mineral filler. Adhesion parameters at the interface were in relatively good agreement with the mechanical properties [8]. Hamzah et al., have studied epoxy filled with wollastonite particles. Tensile, flexural strength, impact and the surface properties were analyzed. The flexural modulus of the graded composites was less as in comparison with the unreinforced epoxy. Decrease in tensile strength was observed in graded composite, whereas increase in hardness was observed which was proportional to fillers contents [9]. Unal H. has studied the influence of flake-like kaolin and needle shaped wollastonite on micro-structural changes of polyamide-6 and behavioral pattern of its mechanical properties. With the increase in filler content tensile strength and modulus, flexural strength and modulus were found to be on the rise, while impact strength and elongation at break on the decline. 20 to 30 wt.% of filler content exhibited maximum enhancement in mechanical properties [10]. Tarade et al., have made a composite of recycled Polycarbonate (RPC) and Virgin polycarbonate (PC) with wollastonite. Use of compatibilizer (Styrene Butadiene block copolymer (SBC) elastomer), showed that wollastonite as a filler had very insignificant effect on the tensile strength of the composite, however 30% decrement in the impact strength was recorded at 15 % loading of wollastonite. A blend without use of compatibilizer made the composite ductile whereas the composite without compatibilizer exhibited brittle behavior [11]. Soncu and Akkoyun have made PP composites of wollastonite (40 wt%) with varying sizes and coatings to get rid of pre-treatments of parts of polypropylene polymer with wollastonite. Flexural properties and surface free energy were found to be increased for lesser particle size and wollastonite added PP composite with amino silane coat [12]. Svab et al., have studied the adhesion phenomena and mechanical properties of isotactic polypropylene/wollastonite/metallocene propylene-ethylene composites. Resulted mechanical properties asserted the efficacy of metallocene EPR elastomers as impact modifiers [13]. Singh et al., have devised a composite of lapinus and wollastonite as brake friction material. It was tested for physical, chemical, mechanical and tribological properties. With increasing content of wollastonite, fiber, density and hardness were observed to be on the rise. With rise in proportion of lapinus fiber, void content, heat swelling, water absorption and compressibility were found to be increased [14]. Sun et al., did deposit Nano-tin oxide on wollastonite using stannic chloride pent hydrate precursor and wollastonite [15]. Tiggemann et al., have studied Morphological, thermal and mechanical properties of the composite of thermoplastic elastomer of wollastonite. The results showed that the filler was in the form of nano particles in the PS domains, increasing the mechanical resistance of the materials at low concentration [16]. Ma et al., modified wollastonite with silane coupling agents to improve interface adhesion and subsequently devised composites of Wollastonite and modified wollastonite-reinforced poly (ether ether ketone) (PEEK) [17]. Ya'acob et al., have investigated polypropylene-glass fiber composite for its mechanical properties. Tensile strength of the composite was found to be decreased while tensile modulus increased with the increasing percentage of glass fibre [18]. Sahai et al., have devised PPO composite with filler Calcium carbonate with and without silane. It has shown increase in tensile strength, flexural strength and modulus and decrease in melt flow index, elongation at break and impact strength [19]. Rane et al., have used optimum quantity of untreated and surface treated talc in PPO to improve thermal stability and mechanical properties of the devised composite [20].

It is found from the literature review in polymer composites that Wollastonite has not been used as filler material with Poly (phenylene oxide) (PPO) polymer. In general, loading, surface treatment, matrix, morphology of wollastonite, hybridization with other fillers improve the mechanical properties of composites with wollastonite as a filler. To optimize mechanical properties, investigation of the effect of shape, size, and quality of wollastonite needs to be done. In this work we have devised PPO composite of Wollastonite with and without coupling agent and test the composite to find out its mechanical properties. Wollastonite filled PPO composites were prepared by adding Wollastonite in wt % of 5, 10, 15, 20, 25, 30 to the polymer without coupling agent. The samples were tested for mechanical properties and optimum results were obtained for loading of 10% by wt. For this polymer composite coupling agent, 'Silane' was added in concentration of 2%, 4%, 6%, 8% and 10% of the filler weight and the composites were tested for mechanical properties.

2.0 METHODOLOGY

2.1 Experimental Design Method

Melt compounding of wollastonite and PPO was carried out on twin screw extruder. All the processing parameters of the extruder were optimized and various batches with varying % of wollastonite were extruded under the same optimized parameters.

Compression moulding process variable such as temperature was optimized to make compression molded sheets for all extruded batches.

Mechanical tests were performed on all the samples and optimum % loading of wollastonite was decided on the basis of better mechanical properties.

To improve the bond between the filler and polymer, coupling agent was added in different concentration to the batch having optimum % of wollastonite with better mechanical properties.

2.2 Materials

Poly (phenylene oxide) grade Noryl N 110-701 (Figure 1) was procured from Nitin Plastics, Ashwini Apartment, Powerhouse Road, Pimpri, Pune-411017. This grade renders excellent performance in variety of applications. Wollastonite white powder of 25 µm size and aspect ratio 3:1 (Figure 2) was procured from Chemax Ahmadabad. Its molecular formula is CaSiO3, and its theoretical composition is 48.28% CaO and 51.72% SiO2. Vinyl Trimethoxy silane (VTMO) coupling agent was purchased from Krishna Enterprise, Borivali, Mumbai.

Figure1 Polyphenylene oxide

Figure 2 Wollastonite Powder

2.3 Surface Modification Of Wollastonite

To improve bonding between the polymer and wollastonite filler, the as-received wollastonite powder was treated with Silane, coupling agent. The concentration of coupling agent was varied from 2 to 10% weight of filler. Ethanol was used to prepare solution of silane. For silane content of 1 wt %, 100 ml of ethanol was mixed with 1 gm of silane and the stirring was done for 30 minutes. Another 15 minutes stirring was done with addition of 100 gm of wollastonite, to ensure uniform dispersion of coupling agent on the wollastonite surface. The treated wollastonite was then dried at 105^0C for 8 hours in an air circulated oven till a constant weight was attained. Similarly other compositions were prepared using silane coupling agent.

2.4 Methods Of Preparation Of Test Specimens

2.4.1 Dry Blending

An air circulated oven at temperature of 100°C was used to preheat the PPO resin and Wollastonite for 1 hour which helps to desiccate PPO as well as Wollastonite. Untreated Wollastonite (25 µ) was added in different concentration of 5%, 10%, 15%, 20%, 25% and 30% by wt to PPO resin. Tumbler mixer was used to mix different proportions of Wollastonite with PPO resin for 5 minutes. Wollastonite was treated with VTMO coupling agent with 2%, 4%, 6%, 8%, 10% of weight of filler. This treated Wollastonite (10% by wt) was then added to PPO. Weight % of PPO resin and untreated Wollastonite is given in Table 1. Weight % of PPO resin, Wollastonite and Silane is given in Table 2.

Table 1 Weight % of PPO resin and Wollastonite (25µ).

S.N.	PPO+Wollastonite	Polymer wt %	Filler wt %
1	Batch 1	100	0
2	Batch 2	95	5
3	Batch 3	90	10
4	Batch 4	85	15
5	Batch 5	80	20
6	Batch 6	75	25
7	Batch 7	70	30
8	Batch 8	65	35

Table 2. Weight % of PPO resin, Wollastonite (25µ) and Silane.

S.N.	PPO+ Wollastonite + Silane	Polymer wt %	Filler wt %	Silane wt % of filler weight.
1	Batch 1	90	10	0
2	Batch 2	90	10	2
3	Batch 3	90	10	4
4	Batch 4	90	10	6
5	Batch 5	90	10	8
6	Batch 6	90	10	10

2.4.2 Melt Compounding

The Melt compounding of Wollastonite and PPO was carried out on co-rotating twin screw extruder (Figure 3) having 16 mm screw diameter and L/D ratio of 38:1. The extruder zones temperature for melt compounding is given in Table 3. The extruded plastic material which was Wollastonite filled PPO resin composite was pelletized on Mayuresh pelletizer. Pelletizer machine was run in the range of 350-400 rpm.

Table 3 Extruder zones temperature for melt compounding

	Zone1	Zone 2	Zone 3	Zone 4	Die
Temp (°C)	200	220	240	260	280

Figure 3 Twin screw extruder

2.4.3 Compression Moulding.

Samples were made by Compression moulding (Figure 4). Teflon Sheets were used to avoid contact between the mould equipment and resin. The known quantity of material was kept in between two Teflon sheets inside mould and then kept the mould inside the two halves of compression molding. Top plate and bottom plate of the compression moulding were set at 270°C and 275°C respectively. Pressure was 150 Kg/sq.cm Breathing was done, the pressure was applied to close the mould. After breathing, once the temperature reached the desired temperature, the mould was closed, and the curing was done for 8 min. After removing the mould from the compression machine, it was allowed to cool for 10-15 minutes.

Figure 4. Compression moulding machine

3.0 RESULTS AND DISCUSSION

The tensile strength of specimen was evaluated according to ASTM D638 on universal testing machine. The crosshead jaw speed of 50 mm/min was used for testing and load cell of 0-1 ton was used. The result reported for an average of 5 test specimens. Rectangular test bar of dimension 165 mm x 13 mm x 3.2 mm (Length x width x thickness) moulded from injection moulding. Izod impact strength was measured as per *ASTM D256* by using pendulum type impact strength tester, with 2.7J striker. Five specimens' average value was taken.

3.1 Tensile Testing (ASTM D638)

The test specimens of Wollastonite filled PPO composite are shown in Figure 5. The tensile strength test report (Figure 6) shows that tensile strength increases (as compared to virgin PPO) with increasing wollastonite filler loading percentage from 5 to 15%. Due to good dispersion of filler in matrix, rise in tensile strength was observed which evident from scanning electron micrograph. It slightly decreases at 20% wollastonite filler content. The change of tensile strength is constant at 25%, 30%, and 35% wollastonite filler content. At higher loadings, brittle tendency of Wollastonite filled PPO composite increases may be due to agglomeration tendency and poor wettability. The test results (Figure 7) show that the composite polymer of PPO and treated Wollastonite (10 % by wt) with Silane percentage up to 4 wt % of filler weight has exhibited marginal improvement in the tensile strength properties than that of the composite polymer of PPO and untreated Wollastonite (10 % by wt). This may be attributed to improved bonding at filler -matrix interface

Figure 5. Test specimens of Wollastonite filled PPO composite.

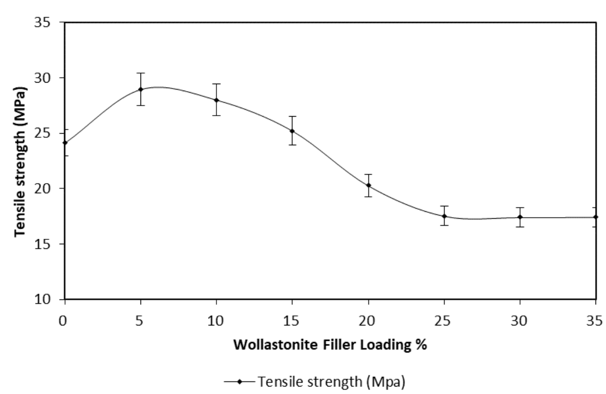


Figure 6. Tensile strength of the composite of PPO and Wollastonite for varying % of Wollastonite.

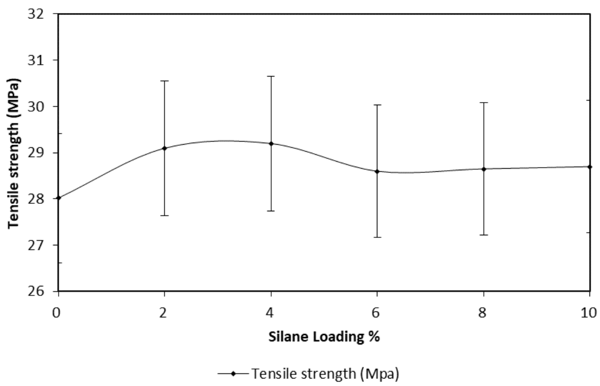


Figure 7 Tensile strength of the composite of PPO and treated Wollastonite (10 % by wt) for varying % of 'Silane'.

3.2 Extension At Break

The result shows (Figure 8) that extension at the break is increased at 5% and 10% wollastonite filler loading as compared to pure PPO because of uniform good dispersion of wollastonite in PPO Matrix. The decrement in elongation is observed at 15% than that of pure PPO resin and thereafter remains constant from 20 to 35% wollastonite filler loading. At 15% rate of elongation is slightly decreased, may be because the presence of mechanical restraints causes interference resulted through the immobilization and physical interaction of the polymer matrix. It exhibits that the filler concentration increases, the elongation declines. The test result (Figure 9) shows that the composite polymer of PPO and treated Wollastonite (10 % by wt) with Silane has exhibited marginal improvement in extension at break than that of the composite polymer of PPO and untreated Wollastonite (10 % by wt), which also supports better wetting characteristics due to silane modification for Wollastonite filled PPO composite.

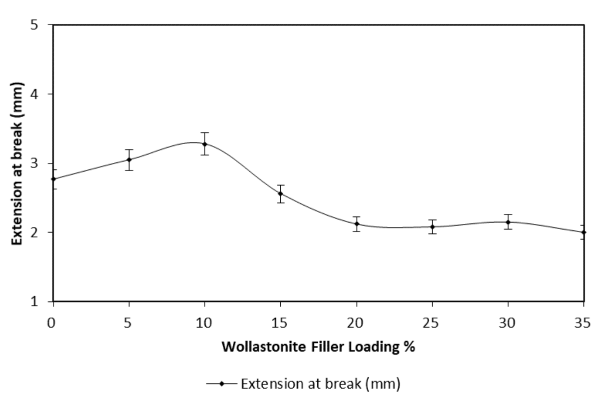


Figure 8 Extension at break of the composite of PPO and Wollastonite for varying % of Wollastonite.

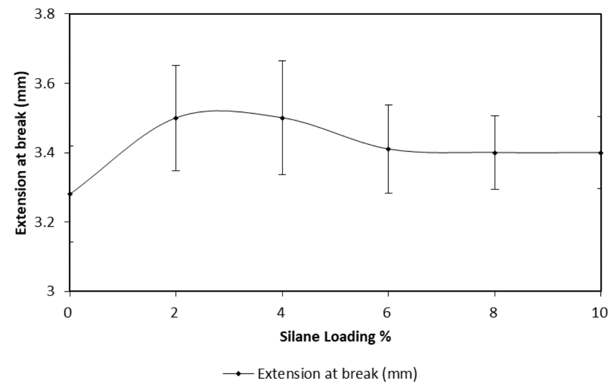


Figure 9 Extension at break of the composite of PPO and treated Wollastonite (10 % by wt) for varying % of 'Silane'.

3.3 Young's Modulus

The result shows (Figure 10) that the young's modulus of Wollastonite filled PPO composite is increased from 5% to 15% wollastonite filler loading as compared to pure PPO. The presence of dispersed rigid particles supports chain stiffening of the matrix leads to increase in modulus. At lower loading up to 15% shows continuous increment in modulus and at higher loading deterioration in the values of modulus which also confirm poor adhesion. The test result (Figure 11) shows that the composite polymer of PPO and treated Wollastonite (10 % by wt) with Silane modification has exhibited good improvement in the Young's modulus than PPO and untreated Wollastonite filled composites which also supports chain stiffening and better adhesion and dispersion of treated Wollastonite in PPO matrix.

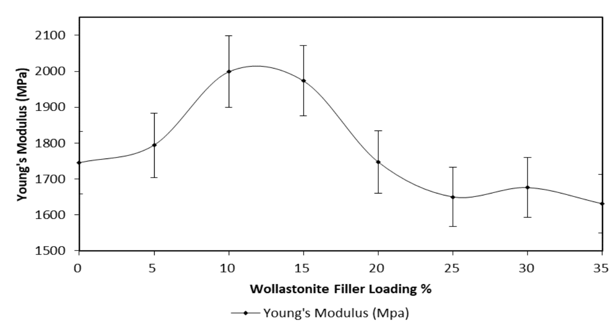


Figure 10 Young's modulus of the composite of PPO and Wollastonite for varying % of Wollastonite.

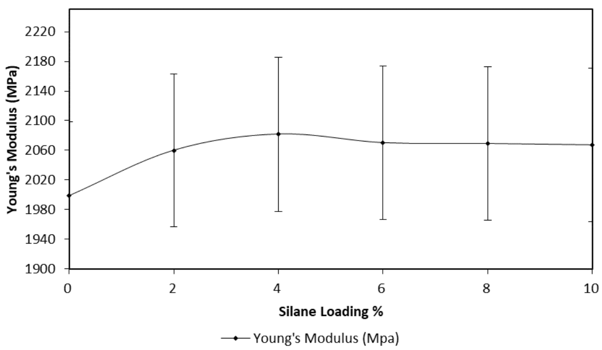


Figure 11 Young's modulus of the composite of PPO and treated Wollastonite (10 % by wt) for varying % of 'Silane'.

3.4 Izod Impact Test (ASTM D256)

The result shows (Figure 12) that impact strength is decreased. The stress transfer rate goes down, which results in lowest impact strength. With the increment in filler loading, the tendency of agglomeration in the composites increases. Therefore, decline in impact strength with increasing filler content is higher leads to brittle characteristics of Wollastonite filled PPO composite. The test result (Figure 13) shows that the composite polymer of PPO and treated Wollastonite (10 % by wt) with Silane has exhibited marginal improvement in the impact strength than that of the composite polymer of PPO and untreated Wollastonite (10 % by wt).

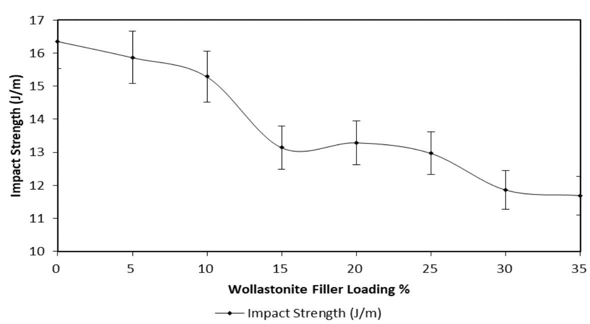


Figure 12 Impact strength of the composite of PPO and Wollastonite for varying % of Wollastonite.

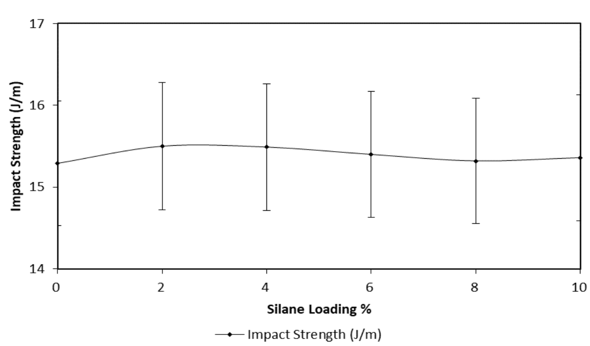


Figure 13 Impact strength of the composite of PPO and treated Wollastonite (10 % by wt) for varying % of 'Silane'

3.5 Melt Flow Index

The result shows (Figure 14) that melt flow index goes on decline with increase in concentration of wollastonite. The reduction in melt flow index is may be because of agglomeration of particles, thereby reducing the total surface area for the interaction with the matrix. The test result (Figure 15) shows that the composite polymer of PPO and treated Wollastonite (10 % by wt) with Silane has exhibited marginal improvement in the Melt Flow Index than that of the composite polymer of PPO and untreated Wollastonite (10 % by wt).

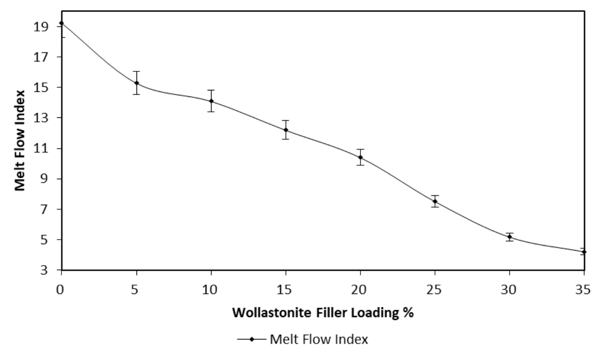


Figure 14 Melt flow index of the composite of PPO and Wollastonite for varying % of Wollastonite.

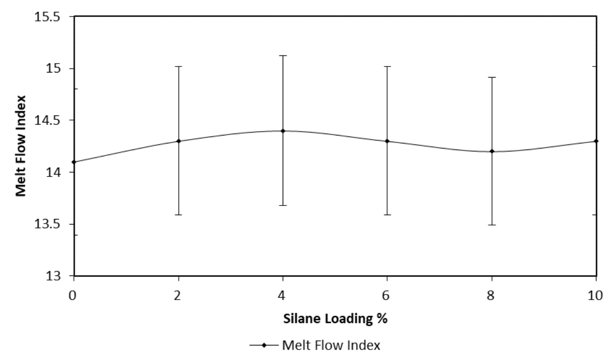


Figure 15 Melt flow index of the composite of PPO and treated Wollastonite (10 % by wt) for varying % of 'Silane'

3.6 Morphological Properties

Field Emission Scanning Electron Microscope (ZEISS Gemini SEM 300) was used to analyse the fractured samples from tensile test. The micrographs of the samples were recorded and studied. SEM images of PPO + untreated Wollastonite (5% and 10% by wt) and PPO + 2% Silane treated Wollastonite (10 % by wt) are shown in Figure 16 and Figure 17 respectively and confirms uniform distribution of Wollastonite in the polymer and better adhesion in the matrix due to treatment.

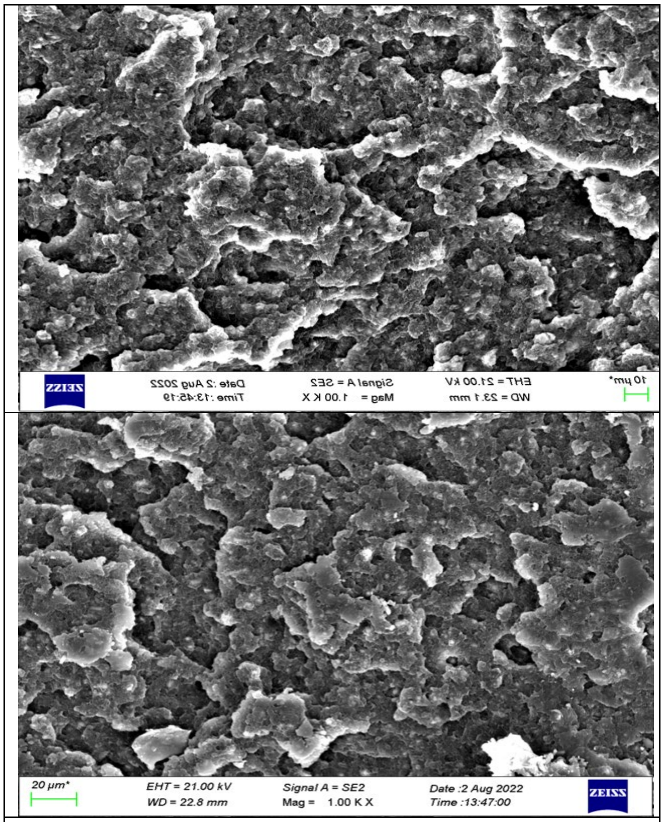


Figure 16 SEM image of the composite of PPO and Untreated Wollastonite (5 and 10 % by wt)

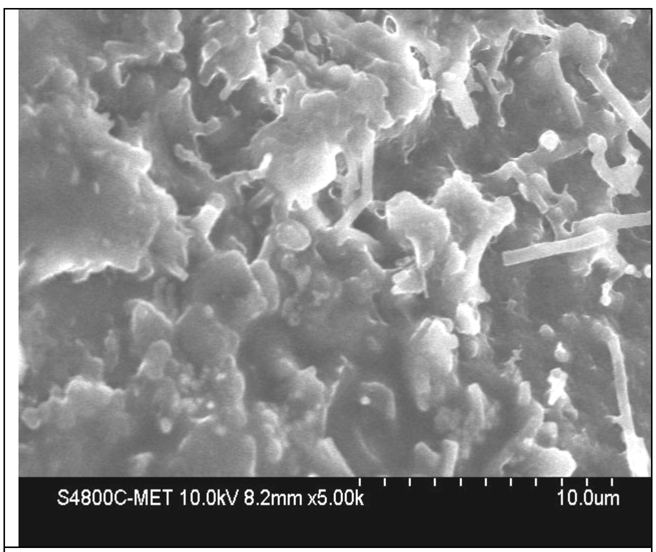


Figure 17 SEM image of the composite of PPO and treated Wollastonite (10 % by wt) with 2% 'Silane'

4.0 CONCLUSION

The untreated and Silane treated wollastonite filled PPO composites were successfully prepared with compression moulding. Young's modulus, tensile strength and impact strength were found to be optimum at 10% wt of wollastonite filler loading. The silane coupling agent was found to be compatible with both filler and polymer matrix thus showing improvement in mechanical properties. At 2-4% Silane loading for 10% Wollastonite–filled PPO composites showed higher overall mechanical properties than PPO and untreated PPO composites. Mechanical properties of Wollastonite–filled PPO composites are decided by the filler content. Morphology of the tensile fracture surfaces of the composites establishes the improvement in interfacial interaction between filler and matrix. It is concluded that the cost-effective composites can be prepared using wollastonite as a filler in PPO polymer. Currently impellers are being prepared using virgin PPO which can be replaced by 10% by wt of wollastonite in PPO offering almost 10% cost reduction.

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Conflicts of Interest

The author(s) declare(s) that there is no conflict of interest regarding the publication of this paper

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