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### An Investigation on Thermo-Mechanical Behaviour of Injection Moulded PP-Compounds

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**Abstract**: Plastic Injection moulding (PIM), produces a wide variety of plastic parts which are useful for various industrial applications. In few areas of Rajasthan, waste white marble-dust is abundant and creating the land pollution. In this paper, to make workable materials, for automobile interior, two compounds viz. PP-composite filled with  $TiO_2$  as filler and the other PP-composite with waste white marble micro-dust as filler were fabricated and their thermo-mechanical behaviour analysed with dynamic mechanical analysis. PP-TiO<sub>2</sub> with 10 weight % shows maximum elastic modulus in all the PP-  $TiO_2$  compounds which was owing to the restriction of the molecular chain motion which creates more bonding effect. Similarly, added storage-modulus (SM) was found between the PP-matrix and white marble micro-dust.

Keywords: Plastic injection moulding, Polypropylene compounds, DMA,  $\rm TiO_2$  , white marble micro-dust

### 1. Introduction

Plastic Injection moulding (PIM) process is the maximum used processing method in the domain of plastic production processes for rapid production of the useful plastic products. It was investigated by many researchers, that with the help of this PIM process, various compounds having fillers, ceramic-powders and natural fibres were prepared successfully with better thermal and mechanical properties. According to researcher Suplicz et al.<sup>1)</sup> which used filler as Boron-nitride, micro-talc powder and TiO<sub>2</sub> with Polymer PP-matrix and obtained better properties of the compounds. In other investigation of Altan et al.<sup>2)</sup> who prepared PP-  $TiO_2$  compounds by melt compounding along with a heavy extruder machine for the compounds. With the silane treatment, fine nanoparticles were adapted and then compounds were injection moulded in polished finished moulds and the mechanical tests were applied to get better Elastic modulus, bending and impact-strength.

It can be observed that the Polymer-Matrix Composite are bonded material with polymer. matrix with a fibrous reinforcing dispersed phase<sup>3),4)</sup>. They have advantage of ease of fabrication with economical manner. It was investigated that the tenacity of the main matrix is to tie various fibres together and so that the load can be transfer uniformly<sup>5) 6)</sup>. According to various investigations, the exclusive arrangement of the micro and nano-materials features, viz. size of particle,

thermal and strength-properties and very less value of concentrations essential to affect the change in a composite of PP, having the progressive features and simulation methods, have created attention in the domain of the micro and nano-compounds. It can be seen that many polymer micro and nano-compounds can be produced and treated in altered methods with creating them principally not only smart but also useful from a desired product's point of view.

It can be exhibited by many researchers that the Dynamic mechanical analysis (DMA) is an vital and effectual procedure for examining the polymer morphology along with the viscoelastic behaviour of the fabricated compounds materials which can be combined to cross-linking density, dynamic fragility, and composite - viscosity<sup>7)</sup>. The viscoelastic property of Polypropylene composite would be affected by various parameters that has an great impact on crystalline-zones with the material crystallinity, lamellar thickness along with its new interface<sup>8),9)</sup>. It can be observed that the SM (storage modulus), LM (loss modulus (E") and the material's damping factor (i.e. Tan  $\delta$ ) are sensitive to change in temperature and deliver evidence about interfacial bonding among the fabricated reinforced fibre and the main matrix of the compound material.

In this study, PP- TiO<sub>2</sub> composite and PP- white marble micro-dust composite were fabricated by PIM process and then analysed with dynamic mechanical analysis (DMA).

Fabricated compounds specimen was under gone the

analysis of DMA by using the Perkin Elmer DMA - 8000 model as shown in Figure 1. Control parameter were the fixed low frequency of 1Hz, rate of heating in range of  $3^{\circ}$ C/min, along with the temperature range with 0-160°C in the tension-mode in the environment of Nitrogen gas. For this analysis, specimens having good surface finish were prepared carefully having the measurements of  $32 \times 5 \times 2$  mm<sup>3</sup> for each composite of filler weightpercentage in the range of 0-20 weight % in the step size of 5 weight %. In Figure 2, PP-White marble micro-dust powder compounds are shown clearly and their SM, LM and the tan delta were investigated for the fabricated compounds.

## 2. Thermo-Mechanical characterization of PP-TiO<sub>2</sub> compounds

The SM (Storage modulus) of TiO<sub>2</sub> filled PP compounds shown in Figure 3. It was observed that there are 3 distinct regions as the stage - I, II and III as the glassy zone (5°C - 25°C), the transition-zone (25 °C -120°C) and rubbery or viscous zone (above the 120°C) respectively. It was also found that the significant development in St. mod. with increasing TiO2 was observed during glassy region which was for the constraint of the chain of molecules motion which creates a strengthening effect. Compound of TiO<sub>2</sub> with 10 weight % have extreme value of SM in MPa. Peak of Tan  $\delta$  shows the glass transition temp denoted by symbol  $T_g$ . It can be seen that among the PP-TiO<sub>2</sub> compounds that values of SM declines with rise in the temperature. It can be observed that to determine the viscoelastic behaviour of compounds the concept of LM (loss mod.) can be used. The dissipation of the heat energy can be determining by the bulk of a material. Figure 4 shows the loss modulus of TiO<sub>2</sub> filled PP compounds. Value of E" also increases along the rise in temperature value, and then it decreases regardless of the weight % of TiO<sub>2</sub>. It can be seen that the decreases of ductility occur duet to the enhancement of stiffness. But here in this case PP-TiO<sub>2</sub> compounds show the better stiffness without losing the desirable ductility property. Figure 4 exhibited that the value of loss-modulus (LM) rises along the increase content of TiO<sub>2</sub> during the observed range of temp. The rise in value of LM with the rise in filler content can be recognized as intensification in energy-absorption with collective filler element.

It can be observed that existence of small length fibers and fine fillers in main matrix shaped a countless quantity of filler-matrix bonding zone in compounds where it can be investigated that the energy can be easily dissipated<sup>10</sup>. By lowering the heat and energy dissipation a better zone of interface can be observed.

It can be observed that among elastic & viscous-phase of a compounds, the amount of Tan-delta ( $\delta$ ) is revealing of the polymer used. Various standards of the obtained Tan delta of fabricated PP-TiO<sub>2</sub> compounds with given temperature and controlled frequency in tune of 1 Hz are

showed in the Figure 5. Observation shows that rise in Tan delta ( $\delta$ ) values with the adding of filler TiO<sub>2</sub> was found as gathering of fine filler TiO<sub>2</sub> in the compounds<sup>11</sup>). This behaviour can be reflected as a rise in the damping stuff of the compounds. It can be detected that Tan  $\delta$  increases with the all values of controlled temperature; and obtained the utmost value in the zone of transition and further drops in the viscous zone.

In the study, it was investigated that compound's specific glass transition temperature somewhat shifts towards the upper values, showing drop of the fine particles along with the molecular-chain mobility conveyed by the fine cross linked and the  $TiO_2$  filler<sup>12</sup>. Similarly, with greater weight percentage of fine filler TiO<sub>2</sub>, it was observed that T<sub>g</sub> of the concern compound Polymer -TiO2 reduce as the higher inter-fine particle molecule layout in compounds prone to enhance the flexibility of polymer molecules-chains and therefore reduce the glass transition temp. Tg of Polymer in the compound. T<sub>g</sub> was observed in the tune of near  $9 \,^{\circ}\text{C}$  -14°C for titanium oxide compounds. It was also looked that good support with the dispersing energy under viscoelastic distortion can be added with the help of size of the cluster and uniform spreading of fine TiO<sub>2</sub> filler in polymer<sup>13).</sup>

For assessment of the damping behaviour of composite materials, it was investigated by researchers that Tan delta > 0.1 can be measured as a standard value. Similarly, the temperature duration and range of the Tan delta > 0.1 for Polypropylene was tuned from 116°C to near 157°C and corresponding Tan  $\delta$  peak was near the value of 0.11 and for various composite 5, 10, 15 and 20 weight percentages the range was observed as 116° to 160 °C, 120 to 163°C, 117 to 161 °C and 117 to 162 °C with conforming crests with the values 0.11, 0.15, 0.13 and 0.12 respectively.



Fig. 1 Perkin Elmer DMA Analyser



Fig. 2: PP-white marble micro dust compounds

### 3. Thermo-Mechanical characterization of PP-White marble micro-dust composite

DMA is an well-organized tool for examining the composite material's viscoelastic properties and morphology which is correlated to principal relaxations and vital parameters, viz brittleness, molecule-link compactness along with the dynamic viscosity<sup>14)15)</sup>. Figure 6 shows the curvature for the disparity of storage modulus (SM) with the change in temperature for matrix -white marble micro-dust compounds. It displays 3 distinct areas I, II & III viz, glassy-zone in range of 5 to 30°C, glassy transition zone in range of 30 to 140°C and further viscous zone near the 140°C respectively.

As a general trend, it can be seen that with surge in temperature, there was drop in the SM. It was observed because rise in temp. led to increase the molecules motion of compounds, which results into soften the compounds. Similarly, it was detected from temp. range between 5°C -30°C, molecules were found toughly arranged and SM values were high. It was observed that region of glassy - stage to viscous region, this values of SM decreased extremely. SM became almost constant in rubbery region.

It was detected in compound of 5 weight percentage, when White marble micro-dust filler were combined in matrix, the stately SM was initiate improved. Additional increase of white marble dust-filler resulted with big improvement in the value of SM for compounds. Due to high SM values, it was observed that ceramic & brittle inorganic-oxide filler, increase in SM values along with increase in white marble-filler<sup>16)17</sup>.

The loss modulus was determined to control the viscous response of compounds<sup>14)18)</sup>. It shows the capability of a compound to dissipate the energy. It can be seen that loss modulus of white marble micro-dust filled composite shown in Figure 7. It displays that in the glassy zone near the temp. 5 to 30°C, the values of loss-modulus (LM) increases with rise in temp., but further observed that it attains extreme temp. near the glass-transition, where it takes place to reduce in zone of the glass transition<sup>19)20)</sup>.



Fig. 3: Variation of SM (E') with temperature for PP-TiO2 compounds



Fig. 4: Variation of LM (E") with temperature for PP-TiO<sub>2</sub> compounds



Fig. 5: Variation of Tan Delta (Tan  $\delta$ ) with temperature for PP-TiO<sub>2</sub> compounds

The increase in LM with increase in white marble fine powder was considered the occurrence that increase in marble weight % contributes to increase in the inside surface molecular-high abrasion that boost up the energy dispersion<sup>21,22</sup>. During the adding of white marble micropowder up to 10 weight % in the matrix of PP, growths the LM and it attains best value. Thus, it establishing that, 20 wt.% of adding of white marble micro-dust caused in reduction in LM of compounds.



Fig. 6: Variation of SM (E') with temperature for PP-White marble micro-dust compounds



**Fig. 7:** Variation of LM (E") with temperature for PP-White marble micro-dust Compounds



Fig. 8 Tan-Delta (Tan  $\delta$ ) variations with the temp. for PP-White marble micro-dust compounds

It was also observed that the drop in LM could be for the fine clustering of white marble filler minute atoms, brittle interfacial organic bond between the matrix and marble filler along with random dispersion of white fine marble in the matrix<sup>23,24</sup>.

It was investigated in the study that the extent of Tandelta is good gauge of a polymer-compounds and it delivers symmetry between the elastic viscous stage in a PP compounds. The measurement of the Tan delta extravagant the applied information related to glass transition temperature and reflects a sign of strength of a polymer-compound to stock and scatter the dynamism. Figure 8 shows the values of Tan-delta fabricated injection-moulded compounds of the PP-white-marble micro-dust compounds with temp. at frequency in range of 1 Hz. It was detected that the values of Tan-delta increases along the increase in the temperature; it approached at extreme level in the transition-zone and then drips in viscous zone. In further study, it was also detected that below temp.  $T_g$ , the factor of damping was little, among the all compounds due to molecules cross-segments that can be observed in the frozen phase<sup>25</sup>).

The higher values of Tan- $\delta$  peak, more the degree of molecules mobility in the compounds. It can be seen in Figure 8 that Tan delta < 1, reflects fabricated PP-compounds show equivalent to solid. Moreover, the interface of white marble compounds decreases the peak values of parameter Tan-delta, along with the integration of further firm filler which control the random motion behaviour of the molecules of PP-filler, which can be useful for automobile interior.

#### Conclusion

- The glass-transition temp. of PP-compounds improved with the rise in micro-filler content in PPmatrix. At the same, the steadiness in thermal behaviour and degradation behaviour are also obtained better with rise in the filler weight percentage. Thus, better thermo-mechanical properties can be obtained with PP-matrix compounds with TiO<sub>2</sub> and white marble filler.
- It was observed that for PP-TiO<sub>2</sub> compounds the values of SM (storage modulus) are in tune of 630 to 1030 MPa at lesser temp. (near 30°C) and these remains greater for 10 weight% of TiO<sub>2</sub> material. Though, at greater temperature near 150°C, the SM exist in tune of 65 to 80 MPa.
- It was found in study that the PP-white marble microdust compounds the values of SM (storage modulus) are in the tune of 625 MPa to 855 MPa at lesser temp. near 30°C and it remains higher for PP-white marble compounds having 10 weight %. Though, at greater temperature near 150°C the SM exist in tune of 65 MPa to 135 MPa. This study suggests that the waste white-marble dust can be used for fabrication of new compounds.

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