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Study on Effect of Wollastonite on the Thermal Properties of Nylon-6 and Morphological Analysis

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Abstract— Nylon-6/Wollastonite composites were prepared with different concentration of 15, 25 and 35 % w/w of Wollastonite (acicular ratio- 15-20%) by co-rotating twin screw extruder. Heat Deflection Temperature (HDT) of injection molded specimens of Nylon-6/ Wollastonite composites was evaluated; Differential Scanning Calorimetric (DSC) and Thermo Gravimetric Analysis (TGA) were studied for these composites. Scanning Electron Microscope (SEM) of tensile fractured sample was revealed uniformity of filler distribution throughout the matrix of Nylon-6/Wollastonite. Wollastonite improves Heat Deflection Temperature (HDT) of the Nylon-6/Wollastonite composites with reduction of product cost. The results suggest application of Nylon-6/Wollastonite in automotive parts, electric motors, gears, power tool housings etc. Due to fibrous nature, Wollastonite replacing short milled glass fibers for both thermoplastics & thermo-sets and short-fiber Asbestos which are hazardous to human health as well as difficult to handle.

Keywords- *Wollastonite; Nylon-6; Thermal Properties; Heat Deflection Temperature (HDT); Differential Scanning Calorimetric (DSC); Thermo Gravimetric Analysis (TGA); Scanning Electron Microscope (SEM)*

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I. INTRODUCTION

NYLON-6 is widely used in engineering fields due to its excellent properties such as easy process ability and good mechanical characteristics. However, its low heat distortion temperature and water absorption restrict its applications [1-6]. Wollastonite is used as reinforcing filler in Nylon-6 because of its low oil and moisture absorption, high brightness, acicularity, and availability-which reduces the ultimate cost of the product.

Commercial grades of Wollastonite (Calcium Meta Silicate - CaSiO3) are typically high in purity because most ores must be beneficiated by wet processing, high-intensity magnetic separation, and/or heavy media separation to remove accessory minerals. The minerals most commonly found associated with Wollastonite are calcium carbonate, calcium magnesium silicate and calcium aluminum silicate.

The reaction has been as follows:

$$SiO_2 \quad + \quad CaCO_3 \quad = \quad CaSiO_3 \quad + \quad CO_2$$

Silica + Limestone = Wollastonite + Carbon dioxide

Wollastonite is hard, white, and alkaline (pH 9.8). It is the only naturally occurring, nonmetallic, white, needle-like natural mineral with a specific gravity of 2.9, Mohs hardness of 4.5 and refractive index between 1.63 and 1.67. It is widely used with Polyamide 6 & 66, Polypropylene (PP), Polycorbonate (PC), Polyurethane (PU), PEEK, Polystyrene (PS), Thermoplastic elastomers (TPE) etc.

Addition of Wollastonite to Nylon-6 eliminates moisture from hygroscopic material, improves stiffness and HDT due to its higher mohs scale of 4.5; surface appearance because of its white color; surface reflectivity as it has refractive index in the range of 1.63-1.67; excellent fire and smoke suppressant properties as Wollastonite is a natural mineral filler

II. MATERIALS AND METHODOLOGY

A. MATERIALS

Polymeric matrix material Nylon-6 (Grade: M28RC, Manufacturer: GSFC) for this work was procured from GSFC, Vadodara. The MFI of Nylon-6 is 28gm/cc. Untreated Wollastonite (Grade: KEMOLIT-A60) & treated Wollastonite (Grade: Fillex "A") were procured from Amgeen Minerals, Ahmedabad. L: D ratio of acicular Wollastonite is 15 (fiber length-75 μm & fiber diameter-5μm). Coupling agent for treated Wollastonite is 3-AminoPropylTriEthoxySilane

The general properties of Wollastonite are:

Table I. Physical Data Of Wollastonite

1.	Color	Brilliant White
2.	Luster	Pearly
3.	Brightness	96-98%
4.	Hardness	4.5%
5.	Oil Absorption	25-30lbs/100
6.	Ph of 10% Slurry (fresh prepared)	9.0
7.	Solubility in Water	0.00095 (gm/100cc)
8.	Melting Point	1540° C
9.	Co-Efficient of Expansion (mm/°C)	6.5x106
10.	Thermal Conductivity (btu/hrft2/of/in)	0.87
11.	Refractive Index	1.627
12	Specific Gravity	2.9
13.	Bulk Density	855 kg/m^3
14.	Bulking Value (Gal/Ib)	0.0413

Table II. Chemical Composition Of Wollastonite

1	Calcium Oxide	Cao	46.5
2	Silica	Sio2	49.7
3	Iron	Fe2o3	0.4
4	Alumina	Al2o3	0.6
5	Loss On Ignition	Loi	2.66

B. COMPOSITE AND SPECIMEN PREPARATION

Nylon-6/Wollastonite composites were prepared by co-rotating twin screw extruder (Make: SPECIFIC ENGINEERING & AUTOMATES) in processing laboratory, HLC, CIPET, Ahmedabad. L: D ratio of screw is 40:1 and screw diameter is 21 mm. The temperature range used was 180-220°C. As Nylon-6 is hygroscopic material, it was pre-dried at 85 °C for approximately 3 hours to remove moisture in an oven before compounding. Wollastonite was also pre-dried at same conditions to remove moisture, which is necessary to have voided free samples. Three batches, each of 3kgs, of untreated as well as treated Wollastonite/Nylon-6 composites were prepared as shown in Table III.

Table III. Batch Composition

BATCH	COMPOSITION
PA6N	Nylon-6 + Untreated Wollastonite 0 Wt%
PA6W15	Nylon-6 + Untreated Wollastonite 15 Wt%
PA6W25	Nylon-6 + Untreated Wollastonite 25 Wt%
PA6W35	Nylon-6 + Untreated Wollastonite 35 Wt%
PA6TW15	Nylon-6 + Treated Wollastonite 15 Wt%
PA6TW25	Nylon-6 + Treated Wollastonite 25 Wt%
PA6TW35	Nylon-6 + Treated Wollastonite 35 Wt%

The specimens for HDT test were prepared with the temperature range of 230 - 275°C by using automatic injection molding machine (Make: ELECTRONICA, Model: ENDURA 90) in processing laboratory, CIPET, Ahmedabad. Before loading the material in the hopper, the material was pre-dried for about 3 hours at 85°C to remove moisture which eliminates voids in the samples.

III. CHARACTERIZATION TECHNIQUES

HDT was measured by using HDT testing machine (P.S.I.SALES (P) LTD,) as per ASTM D 1525. The test was carried out using 66 psi fiber stress and test span 100mm with heating rate of 120°C/hr. DSC characterization (PERKIN ELMER, Diamond DSC) was carried out to investigate the effect of filler concentration on T_g value of composites using heating rate 2°C/min and nitrogen purging rate at 50 ml/min. Virgin Nylon-6 as well as Nylon-6 with fiber composite samples were subjected to thermo gravimetric analysis using TGA (PERKIN ELMER, Pyris 1 TGA) equipment. Samples of approximately 10 mg were heated from 50 to 800°C at a heating rate of 10°C/min in nitrogen atmosphere. The TGA data were reported by corresponding weight loss. All above mentioned tests were carried out at testing laboratory, CIPET, Ahmedabad. SEM analysis of tensile fractured specimen was performed with JEOL (JSM-5610LV) at Metallurgy Department, Faculty of Technology and Engineering, M.S. University, Vadodara.

IV. RESULTS AND DISCUSSION

As shown in fig. 1, the HDT was increased with increasing load of Wollastonite, which results due to increased in stiffness of composite. The Wollastonite can withstand the service temperature up to 1000^{O}C as it has decomposition temperature in the range of 1540^{O}C. The changes in heat deflection temperature of the treated composite were not significant as compare to untreated filler; this may be due to instability of coupling agent. The HDT was increased in the range of 9.75% as compare to Nylon-6 HDT value.

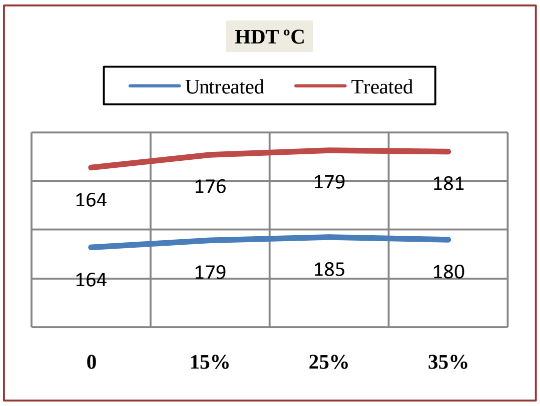

Fig. 1: Effect of Wollastonite concentration on heat deflection temperature of NYLON-6/Wollastonite composites

Fig. 2 shows DSC of various compositions i.e. PA6N, PA6W15, PA6W25 and PA6W35. All samples exhibited melting temperature around 223^{0}C which is an appropriate melting temperature for Nylon-6 material. The change in melting temperature values might be due to hindered chain movement by Wollastonite concentration. Wollastonite melts at around 1450 ^{O}C.

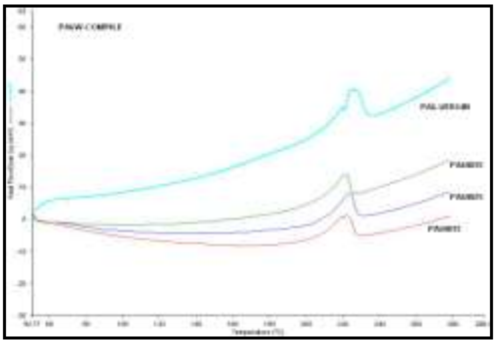

Fig. 2: DSC overlay profiles of virgin Nylon-6 and NYLON-6/untreated Wollastonite composites

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Wollastonite used in this work is inorganic in nature and thus, TGA is an ideal technique to evaluate content in the final composite samples prepared during this work and analysis of decomposition temperature of Nylon-6 natural and Nylon-6 filled with different Wollastonite concentration. In TGA, a sample was heated from room-temperature to 800°C at a constant heating-rate with Nitrogen purge to prevent oxidation. The data is plotted as % Weight V/S Temperature (°C). Fig. 3 shows individual TGA profile of each composition i.e. 15%, 25% and 35% of Wollastonite concentration. Nylon-6 starts to decomposed around 450^{O}C and pyrolysed completely before 550^{O}C and left inorganic material only as a residue. Thermo Gravimetric Analysis conformed, the filler content in the various composition of composite when added during compounding. There was no significant change in content of filler in specimen but it was reduced due to improper mixing of density different materials.

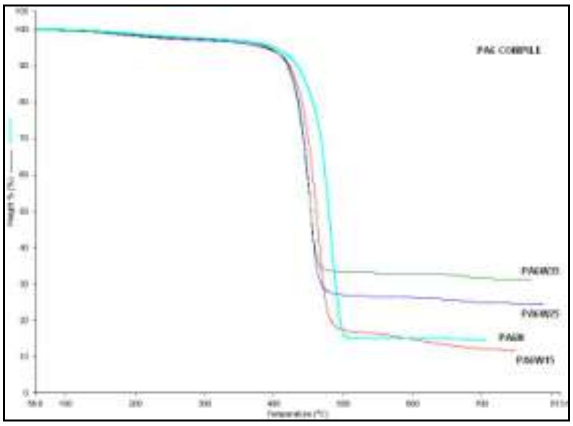
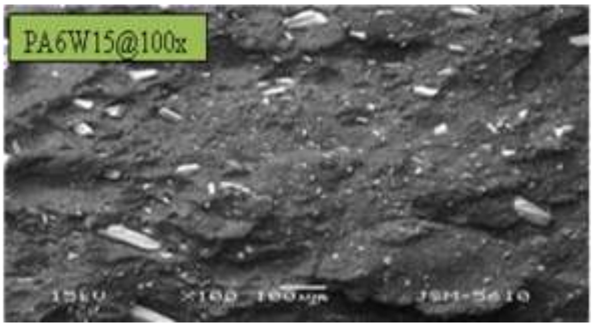


Fig. 3: TGA overlay profiles of virgin Nylon-6 and NYLON-6/untreated Wollastonite composites

The SEM micrographs of tensile fractured surfaces of PA6W15, PA6W35 and PA6TW25 samples were shown in the fig. 4. From the images, following observations were outlined. Image of micrographs indicates that the Wollastonite was uniformly dispersed and distributed in matrix of Nylon-6. L: D ratio of fiber in the range of 15-20% can be measured. The fibers were broken in length due to shearing effect into extruder during compounding and specimen preparation by injection molding machine. From the graphs it was clear that there were not any voids in the tensile fractured sample. Coating of silane coupling agent was shown in the SEM of PA6TW25.



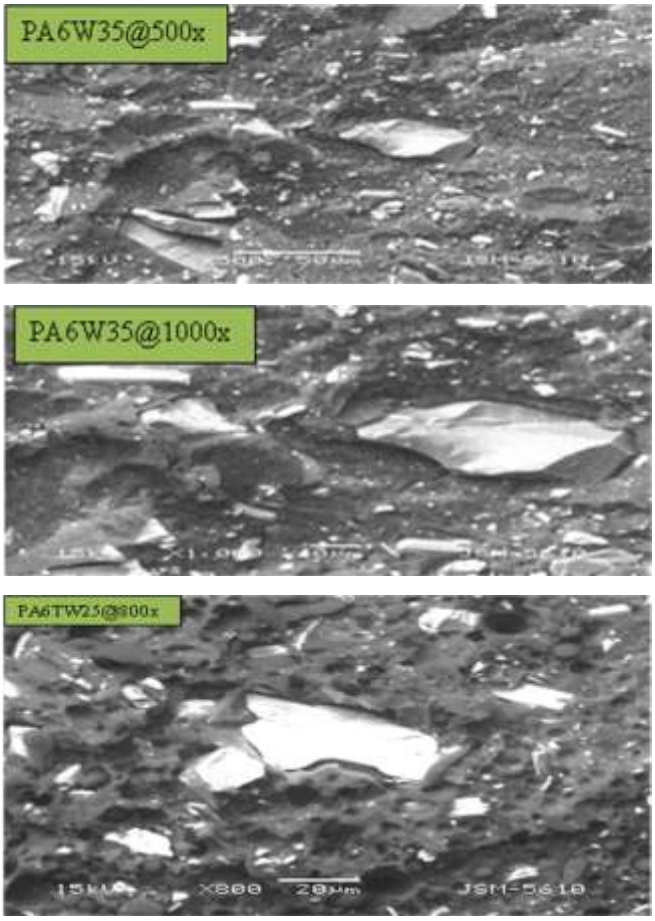


Fig. 4: SEM micrographs of tensile fractured surface

V. CONCLUSION

- Heat deflection temperature was increased with loading of filler but there was not significant role of coupling agent.
- The value of glass transition temperature (T_g) and melting temperature observed in DSC is unaffected by filler content for all compositions.
- Thermal stability of composites were unchanged for all concentration of Wollastonite which revealed from decomposition temperature in TGA & residue conformed the filler in the Nylon-6/Wollastonite composites.
- Micrographs of SEM revealed the distribution and dispersion of Wollastonite throughout in the matrix of Nylon-6 and no void formation shown.

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