High Performance PET Nanocomposite Fibers

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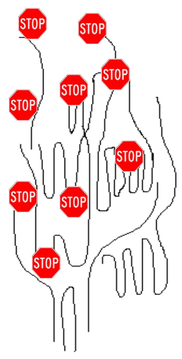
Abstract

- Preparation of PET nanocomposite fibers containing either POSS or organoclays through melt blending followed by spinning and drawing
- **POSS:** with epoxy, amine or trisilanol functionalities capable to interact with the end groups of PET chains
- **Organoclays:** alkyl phosphonium surfactants for modification:
  1. Ethyltriphenyl phosphonium bromide
  2. 4-carboxybutyltirphenyl phosphonium bromide
  3. Tributylhexadecylphosphonium bromide
- Analysis of fiber properties in terms of the particle type and loading level wrt thermal tensile, and morphologic behavior

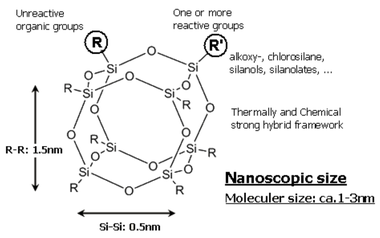
Motivation

Idea: to incorporate nanoparticles into polymer matrix to restrict the movement of the amorphous segments and hence improve the thermal and mechanical properties of the fiber.



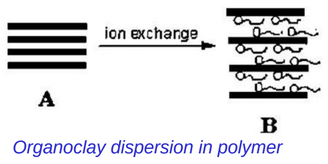
Restricted chain movement (particularly in the amorphous segments) as a result of established chemical and physical bonds between the polymer chain and the nanoparticles.

In this research, we have employed the direct method; melt compounding.



Structure of POSS

R: isobutyl
R': epoxy, amine or trisilanol functionalities capable to interact with the end groups of PET chains



Organoclay dispersion in polymer

Alkyl phosphonium surfactants for modification:
- Ethyltriphenyl phosphonium
- 4-carboxybutyltirphenyl phosphonium
- Tributylhexadecyl phosphonium

Experimental

1. Preparation of organoclays with phosphonium salts:

Conventional ion exchange procedure where 1:1 meq was used.

Clay (CloisiteNa$^+$ from Southern Clay-USA) + Organo-compound

↓ 1:1 (wt/wt) ethanol: H_2O, Rxn @70 °C , 4 h, under continuous stirring

↓ Washing with1:1 (wt/wt) ethanol:H_2O, Purification until no Br / Cl residue

↓ Drying @ 85 °C overnight

↓ Grinding in mortar

Organoclay particles

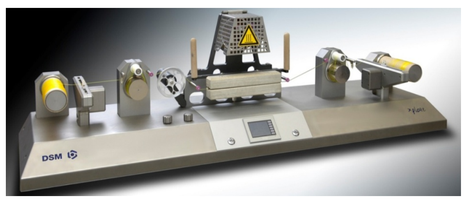
Organoclay particles were mixed with the polymer either in a conventional melt mixing procedure or in a slurry form made with a terpene solvent (Electron^{TM}). POSS's were conventioanlly mixed with PET.

2. Compounding and spinning conditions:

a. Melt mixing of each nanoparticle at concentrations of 0.1, 0.5 and 1wt% for a residence time of 3 min at 280 °C with a screw speed of 50 rpm (15 ml Xplore Microcompounder with co-rotating vertical screws

b. The monofilament transferred onto the winder section of the unit operated at 175 m/min.

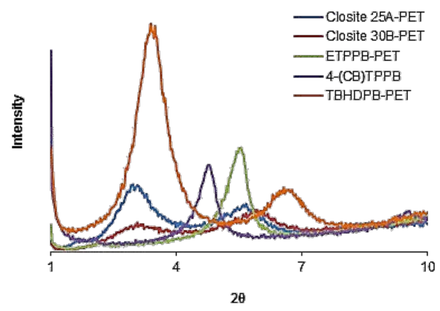
c. After spinning the monofilaments, the bobbin was transferred onto the let-off roll in the drawing unit where it was drawn to 10 times of its original length.



Results and Discussion

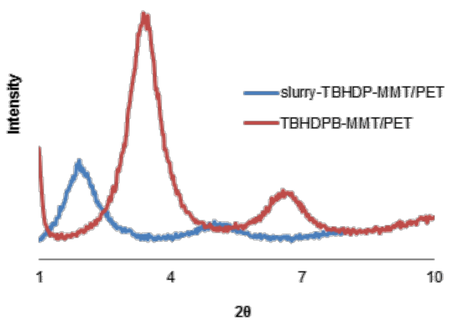
1. Effect of Slurry Process on the Intergallery Spacing

A. WAXS diffraction patterns of the compounds



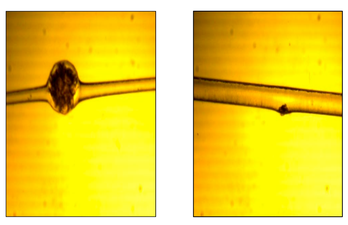
In case of the slurry compounded nanocomposites, higher d-spacing in the TBHDPB-PET composites.

TBHDPB: best clay modifier among the candidates used



The effect of slurry process can be clearly seen in the d-spacing values as an increase from 2.61 nm to 4.6 nm in the basal spacing.

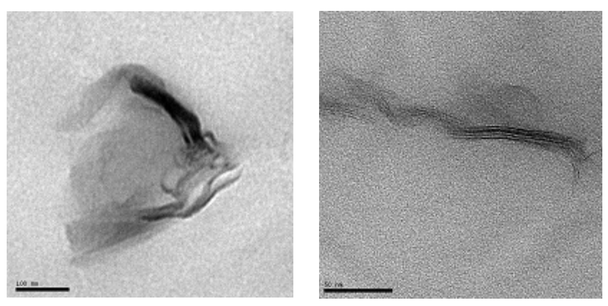
B. Optic microscope images of PET-TBHDPB composites



w/o sonication with sonication

Decreased particle size after sonication in Electron

C. Transmission electron micrographs of 1% TBHDP-PET



w/o sonication with sonication

Better dispersion with slurry process for the clay particles

Since the POSS particles are already dispersed in molecular level, no need for such pretreatment.

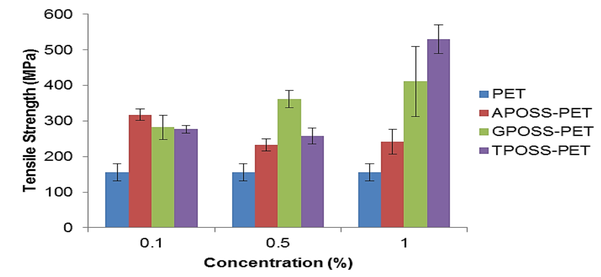
2. Thermal Stability of the Composites:

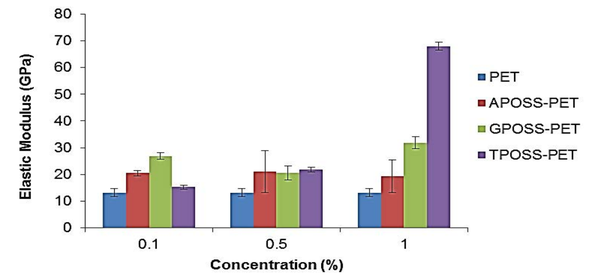
Nanocomposite composition	Weight loss at 280 °C (%)
PET	<0.5
1% APOSS-PET	0.5
1% GPOSS-PET	<0.5
1% TPOSS-PET	0.5
1% ETPP-PET	2
1% THPB-PET	1
1% 4CBTPB-PET	<4

Thermally stable compounds at the processing temperature of the matrix polymer, i.e. PET.

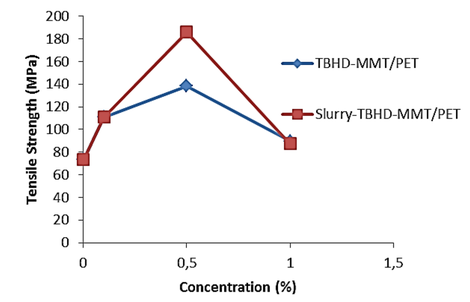
3. Tensile Properties of Fibers

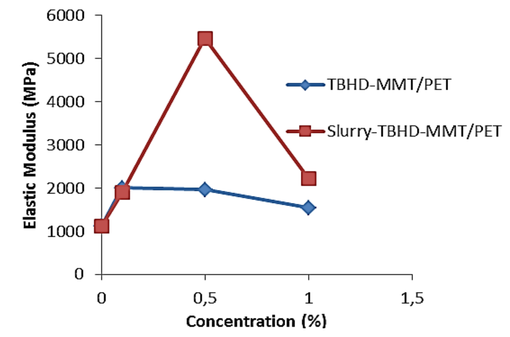
A. POSS-PET nanocomposite fibers



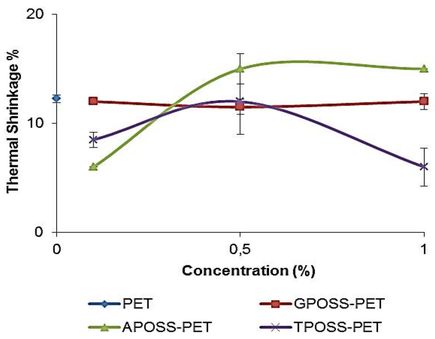


B. TBHDPB-PET nanocomposite fibers





4. Thermal Shrinkage of the Fibers:



Conclusions

1. Even though in the spun fibers, there is practically no change in elastic modulus with concentration of the nanoparticle, once it is drawn to its highest extreme possible, the increase was significant.
2. It was seen that there was a five-fold increase in the elastic modulus in comparison to the neat PET fiber in case of drawn nanocomposite fiber reinforced with TPOSS at 1wt% concentration.
3. Similarly, organoclay-modified PET fiber demonstrated improved tensile properties such that three-fold improvement was recorded on TBHD-MMT-PET fiber at 0.5wt% nanoparticle concentration.
4. Such noticeable differences in the mechanical properties of the nanocomposite fibers were attributed to the improvement in the % crystallinity values obtained for the respective fibers compared to the neat PET fiber.

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