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Change in physico-mechanical and thermal properties of polyamide / silica nanocomposite film

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Abstract:- In this work, polyamide (PA)/silica nanocomposite films prepared by dissolution method of composite mixing were investigated. The morphology of nanocomposite films were characterized using scanning electron microscope (SEM), reveals that nano silica powder were uniformly dispersed on PA silica nanocomposite film. Chemical composition of film was confirmed by Fourier transform infrared spectroscopy (FTIR). The nanocomposite films obtained from dissolution mixing exhibited an increasing tensile strength with an increase in silica content. Thermal behavior of the film were evaluated by DSC, revels that incorporation of silica nano particles in PA film increased the enthalpy (ΔH) value of the film compared to the pure polyamide film.

Keywords:- Dissolution technique, Enthalpy ΔH, PA/silica nanocomposite film, Physical property, Silica nano

I. INTRODUCTION

Polyamide (PA) is a versatile polymer widely used in apparel and technical textile. In practice, commercial PA products available in various forms like surgical hosiery material in medical textiles where nano particle functions as a property enhancer.[1-2] Recently, polymer nanocomposites have received great interest due to their superior properties when compared with conventional composite materials.[3-5] Improvements in mechanical properties, such as stiffness and toughness, dimensional and thermal properties could be achieved with nanofillers.[6-8] The dispersion degree of the filler greatly influences the enhancement efficiency. Therefore, in order to meet those excellent properties, it is very important that the average size of fillers must be present in the nanometer-sized ranges with fine particle distribution in the polymer matrix. Silicon dioxide nano particles have shown a great potential as the nano-filler for plastics as well as for fibers.[9-10] Another interesting choice is nano-sized silica which has been explored as the filler for polymeric materials and held a great potential for developing high performance polymer. In this work, polyamide silica nanocomposite films were prepared by solvent dissolution method. The properties of nanocomposite film were evaluated and were compared in terms of tensile strength and thermal behavior with the film prepared from pure polyamide.

II. MATERIALS AND EXPERIMENTAL METHODS

2.1 Materials

Material used in the experiment were polyamide chips from Fairdeal filaments ltd., India, Silica (SiO_2) nano particles with average size less than 100 nm from Sigma Aldrich Corporation USA and formic acid (LR grade) supplied by Durga Chemicals, India.

2.2 Methods

Accurately weighed silica nano particles were suspended in formic acid under continuous stirring. The polyamide chips were added and mixture was heated to 60°C for 1 hr. The nanocomposite mixture was poured on a cleaned smooth glass surface, compressed manually with another glass surface and solvent was allowed to evaporate up to dryness for 12 hrs at room temperature. Different formulations used to prepare PA/silica nanocomposite film is given in table 1.

Table 1: The composition of nanocomposites preparation by formic acid mixing

Recipe	F1	F2	F3	F4	F5
Silica (wt %)	0.1	0.3	0.5	0.7	1.0
Silica (g)	0.01	0.03	0.05	0.07	0.1
Polyamide (g)	10	10	10	10	10
Formic acid (ml)	100	100	100	100	100

2.3 Characterization of film

The morphology of film sample was observed by scanning electron microscopy (Model: SEM: JSM – 5610 LV, Version 1.0, Jeol, Japan,). The chemical composition of film was recorded using FTIR spectral analysis (Model: Nicolet iS10 FT-IR Spectrometer, Thermo Scientific, Japan).

2.4 Determination of tensile strength

Measurements of the physical properties such as tensile strength and elongation at break were performed on tensile tester (Model: LRX, Lloyd, UK.) in accordance with ASTM D882-02 using a crosshead speed of 50 mm/min.

2.5 Evaluation of thermal property

Thermal behavior of the nanocomposite film was evaluated by differential scanning calorimetry (DSC, Model: 6000 from PerkinElmer, Singapore.), selecting temperature range between 50°C to 300°C.

III. RESULTS AND DISCUSSION

Silica nanoparticles at 0.1,0.3,0.5,0.7 &1.0 % concentration level were mixed with formic acid and incorporated in polyamide chips (10 gm) with constant stirring (Table 1). The mixture was heated at 60 °C for 1 hr. finally the polymer/nanocomposite mixture were poured in flat glass dish and allowed the solvent to evaporate. Results in terms of distribution of sio$_2$ nano particles throughout the film, their effect on the thermal and mechanical properties have been presented in this section.

3.1 Characterization of polymer/nano silica composite film

3.1.1 SEM Study

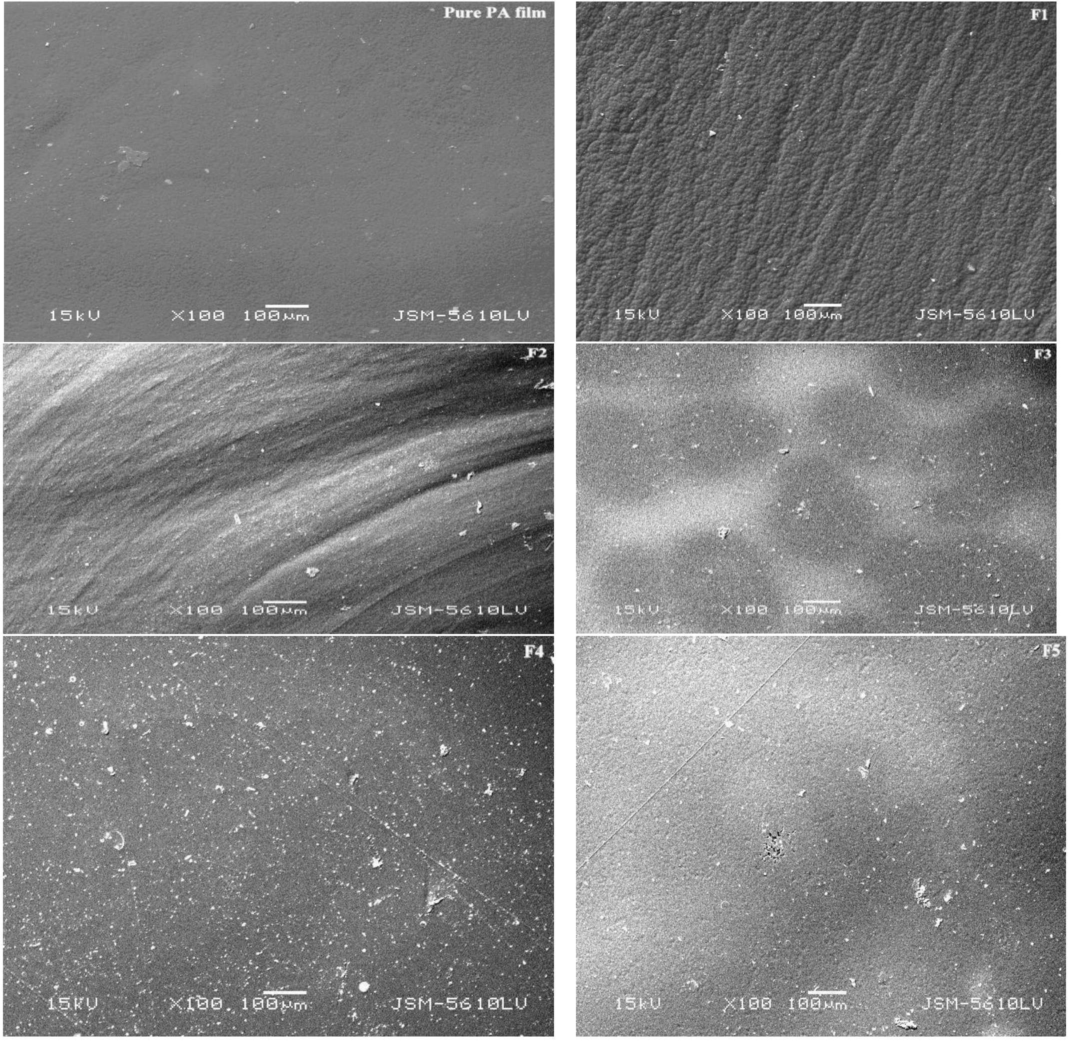

Figure 1 SEM micrographs of pure PA and PA/SiO$_2$ nanocomposites by solution mixing.

The dispersion of silica particles in PA matrix was observed by SEM. Figures 1 show the composite with 0.1-1 wt% silica loading prepared from dissolution mixing method. The uniform dispersion of nano-sized silica was achieved by solution mixing as shown in Figure 1. An increase in percent silica loading results in agglomeration problem and poorer particle distribution.

3.1.2 FTIR Spectroscopic analysis

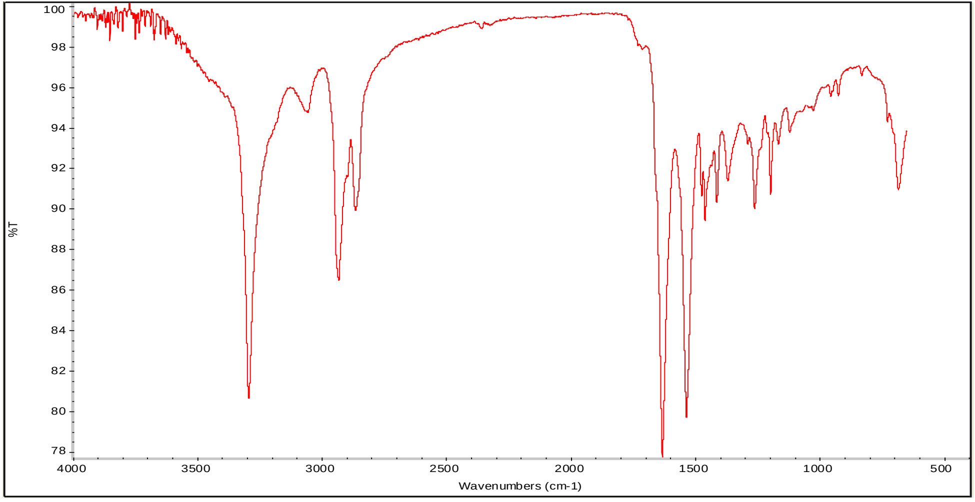

Figure 2 IR Characterization absorption peak of PA/Silica nanocomposite film

The chemical compositions of the virgin and PA/silica nanocomposite film were evaluated using FTIR Spectroscopy. Figure 2 represents the IR Characterization absorption peak of PA/silica nanocomposite film. From the figure it can be seen that the major peaks associated were hydrogen bonded N-H stretching at 3294 cm^{-1} and ~ 3700 cm^{-1}, anti symmetric CH_2 stretching at 2935 cm^{-1}, symmetric CH_2 stretching at 2867 cm^{-1}, C=0 stretching at 1634 cm^{-1}, in plane N-H bending at 1538 cm^{-1}, CH_2 symmetric bending at 1463 cm^{-1}, at 1477cm^{-1} C=C stretching, at 1416 cm^{-1} O-H bending of carboxylic acid, at 1200 cm^{-1} C-N stretching of amine, at 1029 cm^{-1}. The intramolecutar changes in the virgin PA are also illustrated in IR spectra, the main characteristic peaks of Si-O-Si bonds a vibration mode was detected around 1086 cm^{-1}, which are attributed Si-O-Si antisymmetric stretching vibration band in PA/Silica nanocomposite film, so its indicated that the silica nano particle present in film.

3.1.3 Tensile strength

Table 2: Effect of SiO_2 nano filler on **t**ensile properties of nanocomposite films

Sample Name	**Thickness in mm**	**Maximum Load in gf**	**Elongation %**	**Area = Width x thickness in mm^2**	**Specific strength in gm/mm^2**
Pure PA film	0.0427	1418.4	12.02	1.0694	1326.35
0.10%	0.03	1098.8	9.52	0.75	1465.07
0.30%	0.0819	3484.3	13.41	2.0486	1700.82
0.50%	0.0444	2102.8	14.37	1.115	1885.92
0.70%	0.0491	2463.6	16.39	1.2291	2004.39
1.00%	0.0472	1244.2	14.48	1.1805	1053.96

Note:Width of film = 25.04 mm

The results given in Table 2 revels that the obtained nanocomposite film exhibits an increase in tensile strength with an increase in percent silica loading (silica loading of 0.1 wt% to 0.7 wt%, respectively (Figure 3), it may be due to the phenomenon of reinforcement effect at nanoscopic level. However, the composite film at higher concentration of nano silica exhibits the erratic trend of tensile strength value due to the phase separation problem arising from particles' agglomeration.

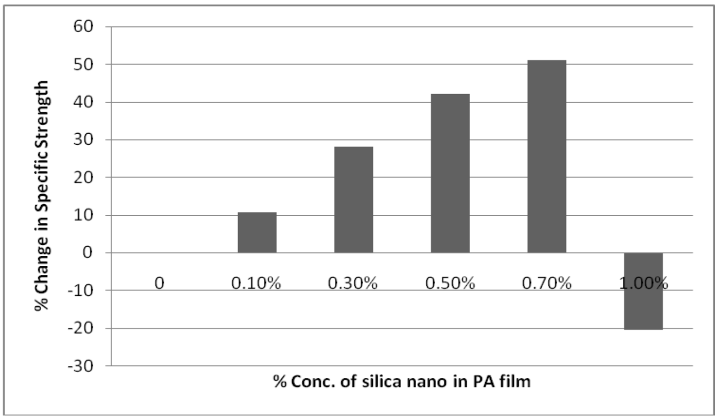

Figure 3 Comparative tensile properties of nanocomposite films

3.1.4 Enthalpy (ΔH)

Tabel 3: Enthalpy (ΔH) of pure PA and PA / silica nanocomposite film

Concentration of nano SiO_2	Enthalpy ΔH (J/g)
00	30
0.1	64.85
0.3	52.89
0.5	49.55
0.7	46.58
1.0	22.87

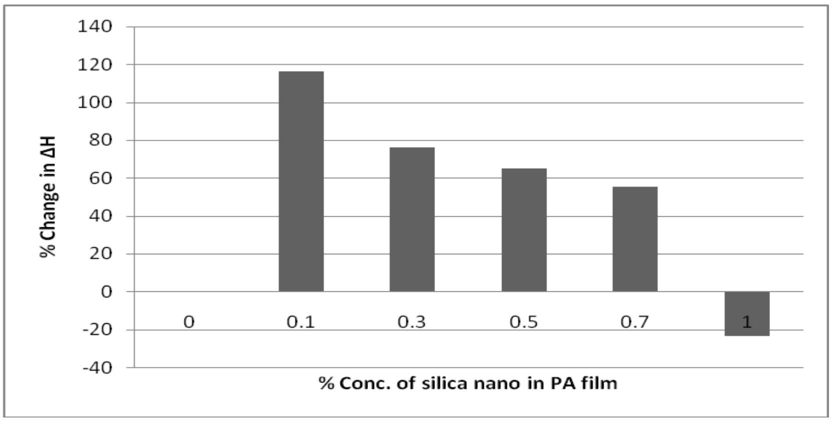

Figure 4 Changes in Enthalpy (ΔH values) of PA / silica nanocomposite film

Table 3 shows the enthalpy (ΔH) values of composite film determined by DSC. The results show that there is a significant difference in ΔH between virgin PA and PA/silica composite at higher concentration of silica nano in PA matrix, indicating that silica in agglomerate form was not able to alter the thermal behavior of PA. On the other hand, lower concentration of nano silica in dissolution mixed PA/silica nanocomposite has a higher ΔH value than virgin PA. In this case, silica size is reduced into a nanoscopic level, hence inducing better thermal stability of the film.

IV. CONCLUSIONS

Polyamide/ silica nanocomposite films by formic acid dissolution can successfully produced under atmospheric condition. Silica particles were uniformly distributed on composite film, when examined by SEM. The composite film exhibits an increased tensile strength with an increase in silica content. However, composite films contain 1.0 % by wt. silica content in mixing exhibited erroneous trend of tensile properties due to poor particle distribution. It was also found that the smaller the quantity of silica particle in dissolution mixture, the higher the enthalpy (ΔH) value of PA film. But increase in the concentration of silica nano particles beyond (0.7 wt. %) resulted in tendering of the composite film. This type of composite film can be successfully utilized for manufacturing of packing materials.

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