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An Investigation on the Effect of Azeotropic Solvent Mixture Pretreatment of 67:33 PET/CO Blended Fabric and Yarn: Part 1

[1,2]B. Muralidharan, [1,2]S. Laya and [3]S. Vigneswari

[1]Birla Institute of Technology and Science-Pilani, Dubai Campus, International Academic City, Dubai, United Arab Emirates

[2]Department of Industrial Chemistry, Alagappa University, Karaikudi, Tamilnadu, India

[3]Department of Chemistry, Raja Duraisingam Government Arts College, Sivaganga, Tamilnadu, India

Corresponding Author: B. Muralidharan, Department of Chemistry, Birla Institute of Technology and Science-Pilani, Dubai Campus, International Academic City, P.B. No. 345055, Dubai, United Arab Emirates

ABSTRACT

Polyester fibre shows a definitive hydrophobic character and a high degree of crystallanity, making it difficult to penetration of dyes. Dye penetration can be improved by pre-treating the fibre using solvents or by annealing. Solvents and solvent mixtures can bring out modification in the fibre structure. Polyester fibre shows a definitive hydrophobic character and a high degree of crystallanity, making it difficult to penetration of dyes. The present study was made with an aim to modify physico-chemical behaviour of the fabric and yarn samples under study. This was made with analytical techniques such as scanning electron microscopy, Fourier Transform Infra Red Spectroscopy, Differential Scanning Calorimetric and X-Ray Diffractometry. The pre-treatments have resulted in solvent induced crystallization of the fabric matrix causing phyico-chemical changes. There was no loss in strength of the treated materials.

Key words: Plasticization effect, segmental mobility, solvent induced crystallization, surface modification, disperses dyes, reactive dyes

INTRODUCTION

Polyester textiles are known for their strength and crease resistance but the drawbacks like low moisture absorbency and static electricity generation lowers their demand in textile market. Polyester/cotton blend taken over the market due to their advantages of both polyester and cellulose and can be wearable (Burkinshaw, 1995). Though polyester/cotton blends have lots of advantages from consumer point of view, it is difficult to dye from the point of view of dyers as the dyeability characteristics and conditions of polyester and cotton are contradictory (Choundhury, 2006).

Annealing and solvent pre-treatments are the important processes that can induce modification in the fibre so that its dyeability can be improved. Most of the solvents and solvent mixtures are able to cause sufficient structural modification in Poly Ethylene Terephthalate (PET) fibres through pretreatments. Reports are available on the use of solvent pretreatments for improving the dyeability of 100% polyester and its blended fabrics and yarns (Shukla and Mathur, 1997; Muralidharan *et al*., 2011a). Mi *et al*. (1991) investigated the interactions of organic solvents with PET and explained structural and morphological changes that occur during solvent pretreatments. Many scientists (Chidambaram *et al*., 2003a; Muralidharan and Laya, 2011) have shown that the modification of the fiber takes place through solvent induced crystallization which is attributed to

the disruption of secondary bond in the fibre enhancing segmental mobility resulting in the formation of new crystallites. Jameel *et al.* (1981) have shown that during the interaction of the polymer with the solvent, the solvent enters in to the amorphous region of polymer structure. It weakens polymer-polymer interaction and replaces with polymer-solvent interaction. This induces an extensive segmental motion and lowers the effective glass transition temperature of material. It is a common practice that many properties of a polymer can be modified by coating or treating with interacting or non-interacting materials (Issaoui *et al.*, 2011; Aan *et al.*, 2011; Abid *et al.*, 2010; Elshafei and El-Zanfaly, 2011). Efforts were taken by many researchers to dye polyester/cotton blends in single step single bath method (Najafi *et al.*, 2008; Blus *et al.*, 2005; Maeda *et al.*, 2004; Lee *et al.*, 2003). This is achieved either by the method of pretreatment and modifying the fibre or by using special class of dye which can simultaneously dye both the component in the blend without interfering with each other. Lee *et al.* (2002) have synthesized a temporarily soluble azo reactive disperse dye with **β**-sulphatoethylsulphonyl group and applied to polyester/cotton blend in one step one bath without dispersing agent.

Reports are available on the use of several physico-chemical methods to improve multi various properties of different textile fabrics in accordance to their end use (Islam *et al.*, 2006a, b; Kusuktham, 2011; Shahbaz *et al.*, 2002). Same way this study aimed at modifying the physico-chemical behaviour of 67:33 polyester/cotton blended samples by pre-treating them with non-aqueous azeotropic ternary solvent mixtures. This was carried out so as to enable them to dye at a lower temperature in a single bath which led to saving of power and cost in dyeing industry.

MATERIALS AND METHODS

Materials

67:33 Polyester/cotton blended fabric (67:33 PCF): Partially texturized commercial fabric of following specifications from Universal Textile Mills, Mumbai, India was used for the study:

- Wt/sq. meter: 79.8 g cm^{-2}
  - Type of end: Filament
  - Type of pick: Filament
  - Ends/inch:121
  - Picks/inch: 74
  - Crimp of weft yarn: 9.2 cm
  - Crimp of warp yarn: 8.7 cm

67:33 Polyester/cotton blended yarn (67:33 PCY): Fine filament yarn of denier 50's supplied by Karpaka Vinayaka Mills, Karaikudi, Tamilnadu, India was used in this study.

Solvents used for pre-treatment: The fabric materials were pretreated before dyeing using ternary azeotropic organic liquids of Fischer-LR grade which were prepared as per the composition stated in Table 1. The composition of the above ternary mixtures was fixed by referring to azeotropic data published by Ryland (1899) and Lecat (1918).

Solvent pre-treatment of the yarn: The fabric was cut into pieces of 0.5 g and yarns were made into 0.5 g hanks (Sartorius-GD 503-Germany of accuracy ±0.0001 g) and were treated with

Table 1: Details of azeotropic solvent mixtures

Solvent system	Weight (%)	Volume (%)	Solubility parameter of the mixture	Solubility parameter	Polarity index	B.P. (°C)
Acetone	24.3	30.6	10.0			
Ethyl alcohol	10.4	13.2	12.7	11.73	14.5	63.2
Chloroform (Ac-EA-Cf)	65.3	44.0	9.3			
Acetone	51.1	64.3	10.0			
Methyl acetate	5.6	6.0	9.8	13.22	9.5	49.7
n-Hexane	43.3	66.1	7.3			
(Ac-MAc-nH)						

azeotropic mixture of solvents prepared based on the composition stated in Table 1 at room temperature for various time intervals, viz. 2, 4, 6, 8, 10, 20 and 30 min. Pretreatments were carried out in a closed trough without allowing the solvents to vaporize out. The amount of solvent mixture set free into the environment was highly negligible as the solvents were reused and consequently the air pollution was minimized. The pre-treated samples were then squeezed using a padding mangle with a wet pick up of 4-5% and then air dried at 50°C ensuring no residual solvent to be present in the solvent treated fabric.

Studies on physical and structural properties: Weight loss measurements of the treated samples were carried out using an electronic balance of accuracy ±0.0001 g (Sartorius-GD 503-Germany). Abrasion resistance of the fabric was found out before and after solvent pre-treatment by using martindale abrasion tester as per ASTM D4966 test method (ASTM D3885, 2007).

The tearing strength of the untreated and solvent pretreated fabric samples were measured using Tenso meter (W 10241-MONSANTO-England) and the tensile strength of the untreated and solvent pretreated yarns were measured using ASTM D638 standard test procedures (Strauch *et al*., 2008).

Scanning Electron Microscopy (SEM) observations were carried out for solvent pre-treated and untreated samples using S-3000H-Hitachi, Japan to understand the surface modification of the fabric if any caused by solvent pretreatments.

Both treated and untreated samples were subjected to fourier transform infrared (FTIR) analysis using spectrophotometer with built in spectral matching computerized software (PERKIN-ELMER, Spectrum BX, USA) to understand any structural modification that took place during solvent pretreatments.

Differential Scanning Calorimetric (DSC) analysis of both untreated and solvent pre-treated samples was carried out using Perkin Elmer, Pyris 6 model-USA. Approximately 10 mg of each sample was fed into the instrument for each run. Pure Nitrogen gas was used to provide inert atmosphere at a rate of 20 mL min^{-1}. All the observations were done at a heating rate of 50°C min^{-1}.

X-ray diffraction (XRD) studies using PANalytical-Model X'pert PRO, Netherland were conducted for the fabrics and yarns before and after solvent treatment to analyze the change in crystalline and amorphous nature of the pretreated samples.

RESULTS AND DISCUSSION

Physical properties: Table 2 and 3 show the observations recorded for the untreated and solvent pre-treated fabric and yarn samples revealing the trend in weight loss, abrasion resistance and

Table 2: Weight loss, abrasion resistance, yarn denier and tearing strength of azeotropic solvent mixture pre-treated and untreated 67:33 PCF

Solvent system	Pretreatment time (min)	Weight loss (%)	Abrasion resistance No. of cycles	Yarn denier	Change in tearing strength (%)
Ac- EA- Cf	2	0.070	7610	1.66	+1.5625
	4	0.185	7725	1.67	+4.687
	6	0.625	8060	1.70	+8.750
	8	0.750	8145	1.72	+3.750
	10	0.850	7905	1.74	0
	20	1.010	7815	1.75	-4.0625
	30	1.145	7715	1.79	-8.125
Ac-MAc-nH	2	0.035	7580	1.64	+0.625
	4	0.100	7700	1.65	+3.437
	6	0.425	8020	1.66	+8.125
	8	0.565	8105	1.69	+3.125
	10	0.715	7865	1.70	-0.625
	20	0.920	7785	1.71	-3.125
	30	0.970	7680	1.74	-6.875
Untreated	-	-	7540	1.64	-

Table 3: Weight loss, abrasion resistance, yarn denier and tensile strength of azeotropic solvent mixture pre-treated and untreated 67:33 PCY

Solvent system	Pretreatment time (min)	Weight loss (%)	Abrasion resistance No. of cycles	Yarn denier	Change in tensile strength (%)
Ac- EA- Cf	2	0.085	7540	1.30	+3.750
	4	0.210	7660	1.32	+6.325
	6	0.685	7995	1.34	+12.750
	8	0.790	7980	1.37	+10.909
	10	0.890	7760	1.40	-1.272
	20	1.050	7690	1.41	-4.650
	30	1.175	7585	1.42	-8.636
Ac-MAc-nH	2	0.04	7295	1.29	+2.000
	4	0.110	7410	1.31	+5.1000
	6	0.450	7800	1.32	+10.750
	8	0.600	7830	1.34	+9.800
	10	0.730	7545	1.3`6	0
	20	0.940	7470	1.38	-3.600
	30	0.995	7265	1.39	-6.525
untreated	-	-	7280	1.28	-

yarn denier. The weight loss was found to increase with increase in pretreatment time in the case of both fabric and yarn samples. A maximum weight loss of 1.145% was obtained for fabric sample treated 30 min duration using Ac-EA-Cf and 0.975% using Ac-MAc-nH. The corresponding weight loss % for yarn samples was 1.175 and 0.995, respectively. This indicates that interaction of Ac-EA-Cf with fabric and yarn samples are more as compared to Ac-MAc-nH. This is in conformity with the polarity index and solubility parameter of the solvent systems. Since, the solubility parameter of Ac-EA-Cf (11.73) is closer to that of polyester (10.3), it dissolves more the polyester component of the blend resulting a higher weight loss. The higher weight loss observed for yarn samples compared to fabric can be attributed to the fact that the yarn samples are free from strain and less tenacious than fabric samples. The values of abrasion resistance indicate that, it increases with increase in the pre-treatment duration up to 8 min and then decreases for higher durations in the case of treated fabric samples. In the case of yarn samples a maximum increase was observed

for 6 min pre-treatment duration for Ac-EA-nH and 8 min for AC-MAc-nH solvent systems. From the yarn denier results it has been observed that denier is getting increased with increase in the pre-treatment duration in the case of both the solvent systems and both of the samples. Among 67:33 PCY and 67:33 PCF, the penetration of solvent being much easier in the case of yarns, greater increase in denier was observed in the former than later. From the results it can be concluded that yarn samples are more influenced by the solvent treatment and solvent with high polarity index and solubility parameter close to polyester can cause more impact on the fibre matrix. It is clear from the above mentioned results that there was no adverse effect on these properties after solvent pre-treatment and the best treatment durations range is between 6-8 min. As the solvents penetrate into the interior of the fiber structure, the intermolecular force of attraction in the fiber matrix reduces leading to swelling and plasticization effect of the fiber substrate. Reports are available on the concept of solvent induced crystallization of the fibre as a result of solvent treatment (Rajendran and Mishra, 2007; Muralidharan *et al*., 2004; Muralidharan and Laya, 2011).

Tearing strength measurements: Results of tearing strength measurement of untreated and azeotropic solvent mixture pretreated fabric samples are presented in Table 2. There was an increase in the percentage of tearing strength till 6 min treatment duration up to maximum of 8.75 and 8.125% for the solvent systems Ac-EA-Cf and Ac-MAc-Cf, respectively. This is again is in conformity with observed results of other physical properties. For the pre-treatment duration above 10 min the tearing strength found to decrease. The solvents penetrate into inter-crystalline region i.e., amorphous region of the polymer material and reduces the lateral forces operating in the chain molecules of the fibre leading to increased flexibility to the chain molecule and higher breaking extension. The same kind of observations has been reported earlier literature (Muralidharan *et al*., 2011a). In the case of samples subjected to prolonged pretreatment the flexibility of the chain molecules was found to decrease due to induced crystallization.

Tensile strength measurements: Results of tensile strength measurements of untreated and azeotropic solvent mixture pretreated yarns are presented in Table 3. There was an increase in the tensile strength till 6 min treatment duration up to maximum of 12.750 and 10.725%, respectively for the solvent systems Ac-EA-Cf and Ac-MAc-Cf. For the pre-treatment duration above 8 min the tearing strength was found to decrease as compared to untreated samples. The improvement in the strength of treated yarns can be due to improvement in the structural order of the polymer matrix and generation of more number of crystallites, leading to improvement in the resistance power to deform the material with higher inter chain bond. These observations are further supported by the XRD and DSC results also. The present observations are in conformity with the reports available on the effect of solvent pretreatment on polymers wherein the solvents do not penetrate the compact crystalline region in the polymer and therefore do not affect the strength of the polymer material rather improve the strength of the treated material (Weigmann and Ribnick, 1974; Muralidharan and Laya, 2011).

SEM morphology: The Scanning electron micrographs of untreated and solvent pre-treated 67:33 PCF and 67:33 PCY are presented in Fig. 1-6. It is evident from the photographs that the solvent mixtures attacked almost the entire surface of the fabric which is treated with solvent mixtures while the surface of untreated fabrics was found to be smooth. The solvent pre-treatment caused elongated pits or cavities on the surface. In fabrics treated with both solvent systems, the fabric

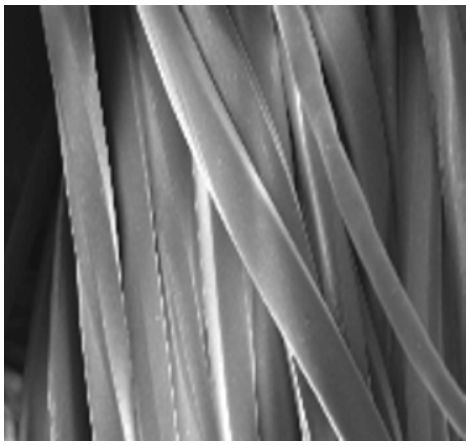

Fig. 1: Untreated 67:33 PCF

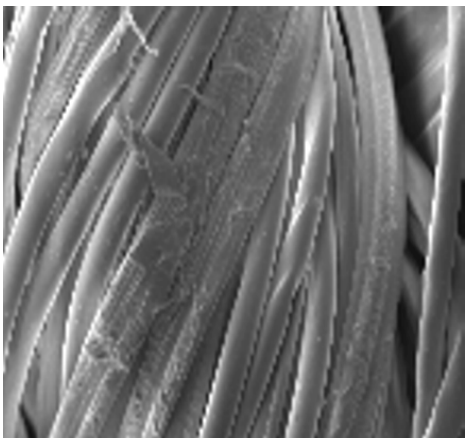

Fig. 2: 67:33 PCF treated with Ac-EA-Cf

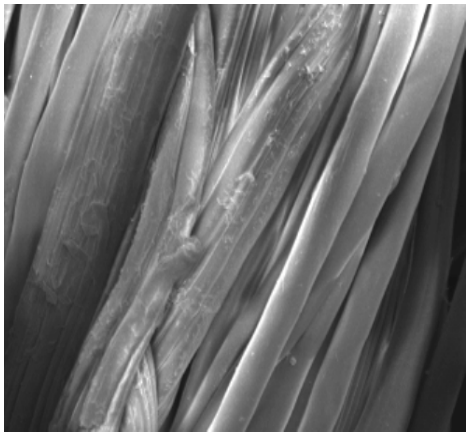

Fig. 3: 67:33 PCF treated with Ac-MAc-nH

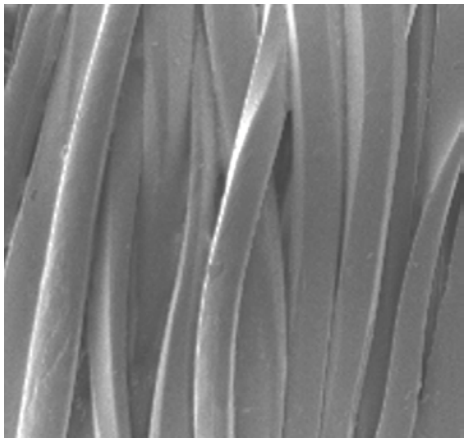

Fig. 4: Untreated67:33 PCY

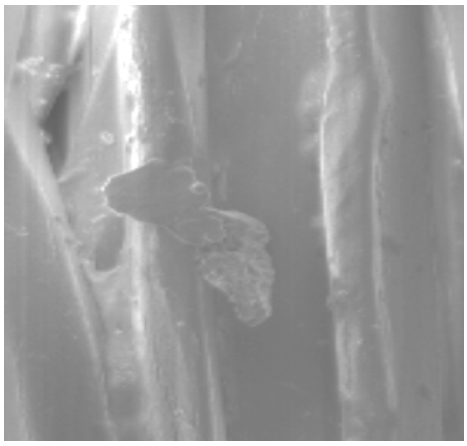

Fig. 5: 67:33 PCY treated with Ac-EA-Cf

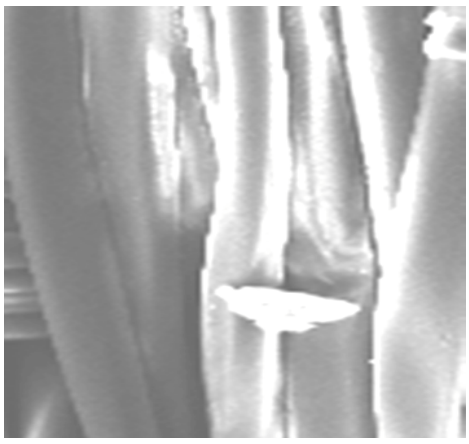

Fig. 6: 67:33 PCY treated with Ac-MAc-nH

surface got affected by the attack of the solvent. The observed changes in the fabrics were probably caused by the release of stresses in the polymer during solvent induced swelling. The formation of pits has been reported to be due to the attack of the solvent in the amorphous region which is more susceptible (Muralidharan and Laya, 2011). It is clear from the above study that the solvent pre-treatments have modified the surface of the fabric which was reflected in weight loss study also. Among the two different solvent systems used, the extent of attack of Ac-EA-Cf solvent system on the yarn materials was found to be maximum and least in the case of samples treated with Ac-MAc-nH with fabric materials. The observed results were further supported by FTIR Spectral studies, XRD, DSC and dyeing results which are discussed in detail under appropriate sections in this research paper.

FTIR observations: FTIR studies were carried out on to 67:33 PCF and 67:33 PCY samples with and without solvent pre-treatments. The spectra were analyzed (Fig. 7-10) to assess any structural

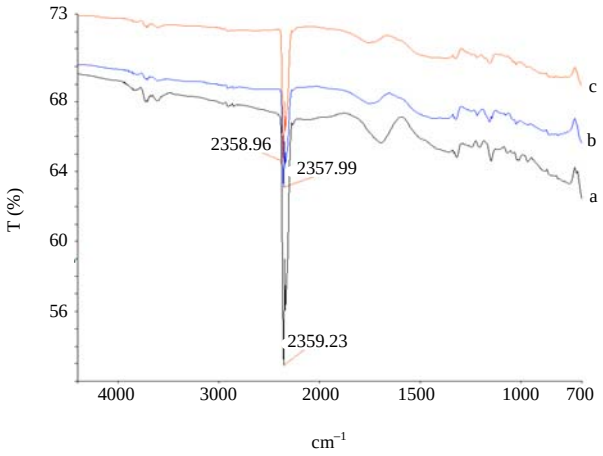

Fig. 7: FTIR spectra of 67:33 PCF treated with Ac-EA-Cf for different durations (a) Untreated (b) Treated for 4 min and (c) Treated for 30 min

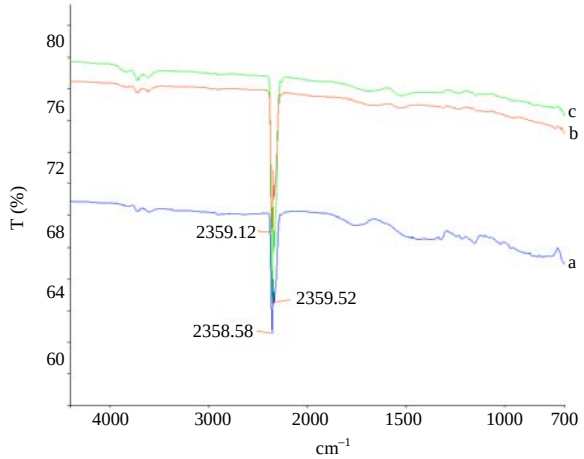

Fig. 8: FTIR spectra of 67:33 PCF treated with Ac-MAc-nH for different durations (a) Untreated (b) Treated for 4 min and (c) Treated for 30 min

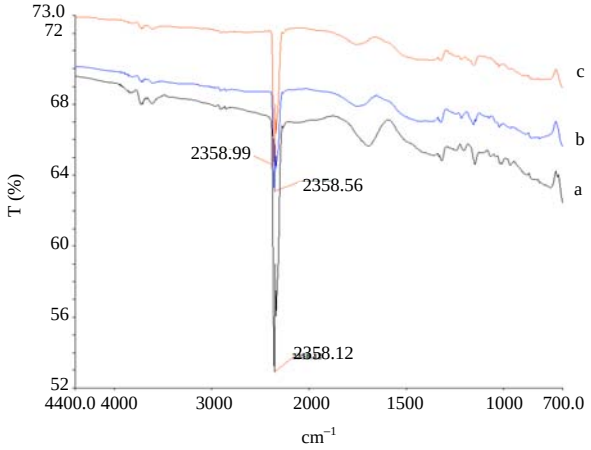

Fig. 9: FTIR spectra of 67:33 PCY treated with Ac-EA-Cf for different durations (a) Untreated (b) Treated for 4 min and (c) Treated for 30 min

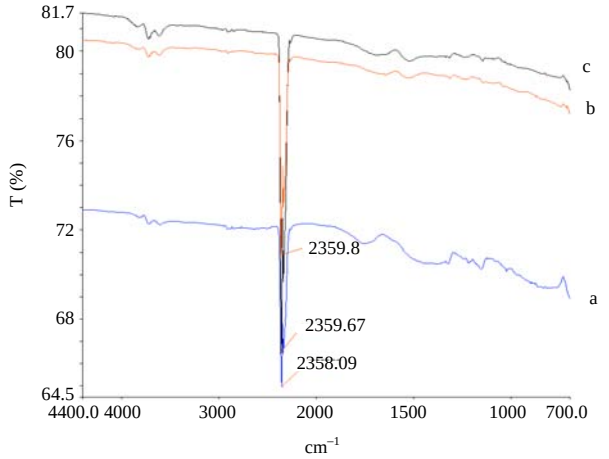

Fig. 10: FTIR spectra of 67:33 PCY treated with Ac-MAc-nH for different durations (a) Untreated (b) Treated for 4 min and (c) Treated for 30 min

change, creation of any new functional groups or the alteration of existing groups in these materials as a consequence of solvent treatments. The spectra of samples subjected to solvent pretreatments for 4 and 30 min were considered for comparison with the spectrum of untreated sample. There was a slight shift in the position of the observed peaks in the case of samples treated with solvents. An intense peak at 2350-2360 cm^{-1} can be attributed to methylene C-H stretching. The peak height for this peak has increased with solvent treatment in all the cases indicating the strong interaction of solvent mixtures with the treated material. For fabric samples treated with AC-EA-Cf, the peak values got decreased from 2359.23 to 2357.99 and 2358.96, respectively for 4 min and 30 min treatment durations. In the case of AC-MAc-nH treated fabric samples the values got increased from 2358.58 to 2359.12 and 2359.52, respectively for 4 min and 30 min durations. This observed behaviour is in conformity with the solubility parameter and polarity index of the solvent. A similar trend was observed in the case of yarn samples. Factors such as type of solvent

system used and solvent pre-treatment duration were found to have effect on the extent of shift in the position of absorption peaks as found in earlier report (Muralidharan *et al*., 2004). All these observations led to a conclusion that the solvent pretreatments have not introduced any new functional groups into the polymer matrix. It was also inferred from the results that only physical changes to the polymer matrix have happened during solvent pre-treatment.

Differential scanning calorimetry: DSC thermograms obtained for untreated and azeotropic solvent mixtures pre-treated 67:33 PCF are presented in Fig. 11-14. The thermogram patterns of treated fibres are found to show small changes in their starting, peak and melting temperatures, however melting heat has considerably got increased for solvent treated samples (Table 4, 5). The increase in the melting heat was more pronounced (increased from 36.22 to 46.65) in the case of the solvent system Ac-EA-MAc which is more interacting in nature. In the case of Ac-MAc-nH the value got increased from 36.22 to a maximum of 45.07. This observation is due to solvent

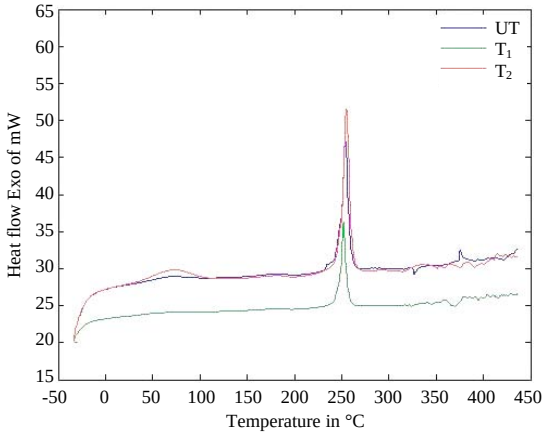

Fig. 11: DSC of 67:33 PCF treated with Ac-Ea-Cf for different durations. UT = Untreated, T_1 = 4 min and T_2 = 30 min

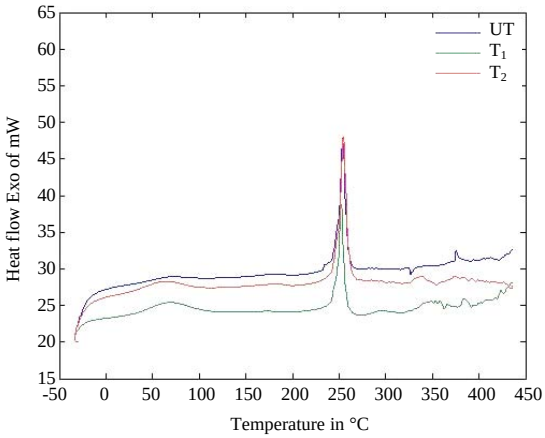

Fig. 12: DSC of 67:33 PCF treated with Ac-MAc-nH for different durations. UT = Untreated, T_1 = 4 min and T_2 = 30 min

Table 4: Thermal behaviour of azeotropic solvent mixture treated 67:33 PCF

Solvent system	Pretreatment time (min)	T_1	T_0	T_2	T_M	Max. heat flow in mW
Untreated	0	232.8	240.3	265.1	252.3	36.22
Ac-EA-Cf	4	233.7	243.6	264.9	253.1	46.09
	30	233.2	243.9	265.2	253.6	46.65
Ac-MAc-nH	4	233.5	241.9	264.8	252.9	44.89
	30	233.7	242.2	265.3	253.1	45.07

T_1, T_0, T_2 and T_m, correspond to starting, onset, final and melting temperatures, respectively

Table 5: Thermal behaviour of azeotropic solvent mixture treated 67:33 PCY

Solvent system	Pretreatment time (min)	T_1	T_0	T_2	T_M	Max. heat flow in mW
Untreated	0	232.3	240.0	264.7	252.0	36.08
Ac-EA-Cf	4	233.2	243.1	264.5	253.0	46.01
	30	233.3	243.7	264.9	253.5	46.56
Ac-MAc-nH	4	233.3	241.7	264.5	252.7	44.83
	30	233.6	242.0	265.3	252.8	44.99

T_1, T_0, T_2 and T_m, correspond to starting, onset, final and melting temperatures, respectively

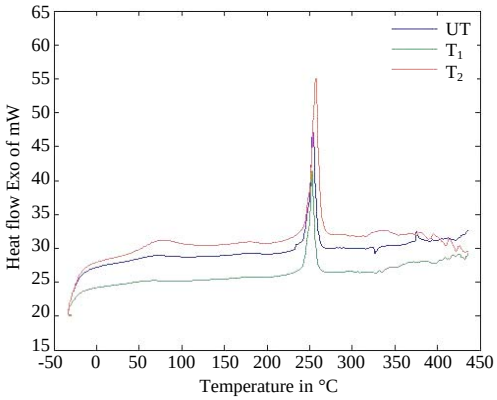

Fig. 13: DSC of 67:33 Pcy treated with Ac-EA-Cf for different durations. UT = Untreated, T_1 = 4 min and T_2 = 30 min

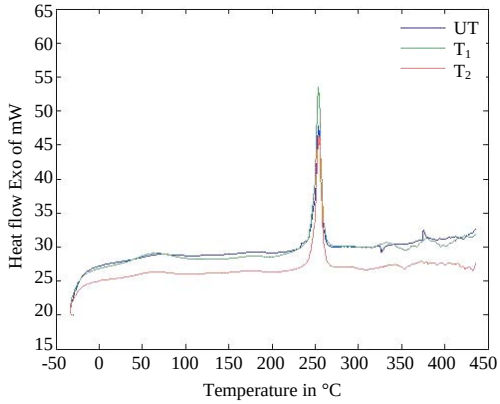

Fig. 14: DSC of 67:33 PCY treated with Ac-MAc-nH for different durations. UT = Untreated, T_1 = 4 min and T_2 = 30 min

induced crystallization of the fibre that resulted from solvent pre-treatments. The increase in crystallanity due to solvent treatment has been reflected in XRD patterns also indicating that the solvent systems have interacted with polymer chains disturbing the morphology and fine structure leading to solvent induced crystallization. The solvent molecules penetrate into the polymer structure weakening the polymer-polymer interaction, compensating with polymer-solvent interaction. This type of behaviour of polymers during solvent treatment has already been reported in literature (Jameel *et al.*, 1981; Chidambaram *et al.*, 2003b). The interaction of solvent with the polymer may be of two types viz., inter-crystalline interaction and intra-crystalline interaction. In the case of inter-crystalline interaction, the solvent penetrates inside the amorphous region only. On the other hand, in the case of intra-crystalline interaction the interacting solvent penetrates inside the crystalline region, decrystallizes the sample and affects lateral order parts of the fibre. The polymer chains within this region are under lower stress and generally results in the rearrangement of molecular chains. Rajendran and Mishra (2007) have proved that crystallization within the polymer takes place even in the swollen state and crystalline areas of the sample get increased. In the present study, the interaction of solvent with the fiber material was found to be inter-crystalline interaction. This is evident from the considerable increase in the melting heat for solvent treated samples due to solvent induced crystallization as reported in earlier literature (Muralidharan *et al.*, 2011b). The trend in the increase of melting temperature and melting heat was found to increase in the following order for the samples used in the study; 67:33 PCF>67:33 PCY.

X-ray diffraction studies: XRD studies were made on the solvent pre-treated and untreated samples used in the present study and XRD patterns obtained are presented in Fig. 15-18. It is very distinct from the observations that the broad and diffused peaks found in the untreated samples were sharpened after solvent pretreatments with both the solvent systems. As the duration of pre-treatment increased, the peaks became sharper indicating the increase in crystallanity of the fibre. The peak heights got increased with increase in the treatment duration as well as increase in the polarity index of the solvent systems. The sharp peaks correspond to crystalline regions, the diffused and the broad ones refer to amorphous regions (Gowarikar *et al.*, 1996). The interaction

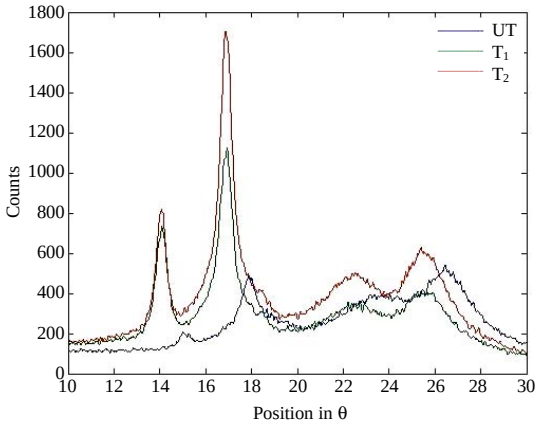

Fig. 15: XRD of 67:33 PCF treated with Ac-EA-Cf for different durations. UT = Untreated, T_1 = 4 min and T_2 = 30 min

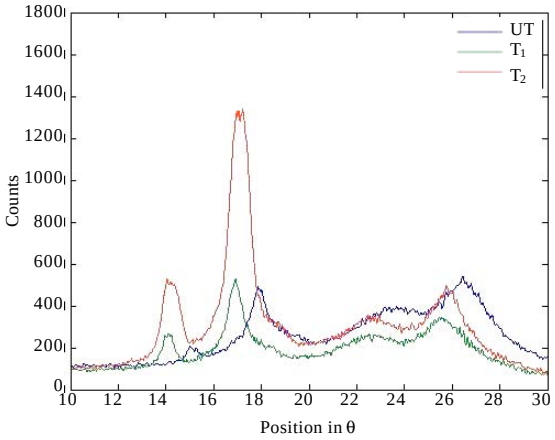

Fig. 16: XRD of 67:33 PCF treated with Ac-MAc-nH for different durations. UT = Untreated, T_1 = 4 min and T_2 = 30 min

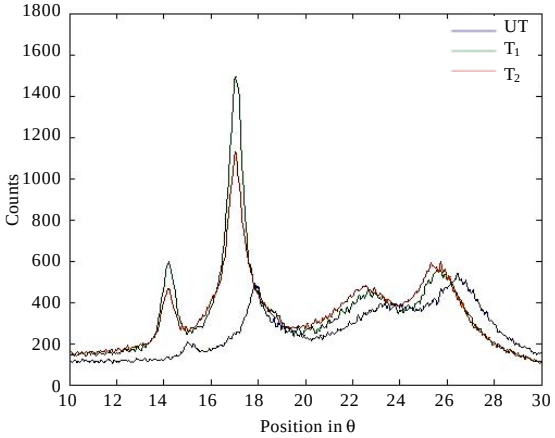

Fig. 17: XRD of 67:33 PCY treated with Ac-EA-Cf for different durations. UT = Untreated, T_1 = 4 min and T_2 = 30 min

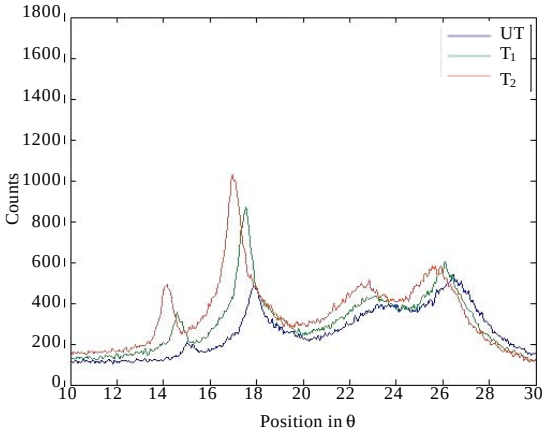

Fig. 18: XRD of 67:33 PCY treated with Ac-MAc-nH for different durations. UT = Untreated, T_1 = 4 min and T_2 = 30 min

of solvent with polymers results in re-crystallization and de-crystallization of the corresponding polymer contents. The diffraction pattern of original untreated fabric and yarn samples show low to medium sharp peaks in (010), (110) and (100) planes indicating the presence of crystalline as well as amorphous regions. The peak height was very less pronounced in (010) plane. The peak intensities of untreated samples were observed at values 15, 18 and 26 corresponding to (010), (110) and (100) planes. The first two peaks in all the figures were sharp compared to the third peak due to polycrystalline nature of the material. In the case of pre-treated samples, the peaks became very sharp and the intensity counts increased to a larger extent compared to untreated samples. These observations indicate that the solvents acted as plasticizer in the non-crystalline region breaking intermolecular bonds and enhancing segmental mobility of the polymer which induced crystallization leading to the formation of crystallites (Muralidharan *et al.*, 2004). The creation of micro-voids in the polymer structure due to solvent treatment which was clearly seen in SEM photographs is further supported by the present XRD studies.

CONCLUSIONS

From the present investigation it can be concluded that azeotropic solvent mixture pre-treatment can bring physico-chemical modification to polyester/cotton blended yarn and fabric materials without damaging the material. The modification depends on the polarity index, solubility parameter and pre-treatment time of the solvent mixture. This pretreated material may be used for dyeing at a lower temperature leading to energy saving in dyeing process. From the weight loss and abrasion resistance measurements, it is evident that there is no appreciable damage to the polymer matrix pre-treated for 6-8 min duration. Rather, the treatment has improved the overall properties of the polymer material through solvent induced crystallization which is evident from SEM, DSC and XRD studies as well. FTIR observations substantiated that there is no introduction of new functional groups in the fibre matrix.

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