



Influence of trichloroacetic acid-methylene chloride on structure of polyester fabrics

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In this study, polyethylene terephthalate (PET) fabric has been treated with trichloroacetic acid–methylene chloride (TCA-MC) to modify its functional properties by altering the structure. TCA-MC interacts with both amorphous and crystalline regions of PET and alters the structure (solvent-induced-polymer modification), which directly affects the functional properties of PET fabrics. Five different concentrations (0.5, 1, 3, 5 and 10%) of TCA-MC at 1, 3 and 5 min are used to treat two fabric structures, viz 100% micro polyester fabric and 100 % texturized polyester fabric. The physical properties and wettability of the treated samples are investigated, and the results are statistically analyzed. The results reveal that TCA-MC alters the structure of polyester, with the degree of modification determined by TCA-MC concentration and treatment time.

Keywords: Micro polyester, Solvent-induced polymer, Trichloroacetic acid- methylene chloride, Texturized polyester, Wettability properties

1 Introduction

Polyester is commonly used in textile manufacturing as flat, texturized filaments and staple yarns in a range of mixed configurations^{1,2}. It is uncomfortable to wear due to its low moisture absorption and high crystalline content. The most common type of polyester is polyethylene terephthalate (PET). It is hydrophobic due to the lack of polar groups and tends to generate static, making it difficult to finish, wash, and dye³⁻⁶. These properties have an impact on wear comfort and, therefore, necessitate structural adjustments to allow for hydrophilicity or combining with another hydrophilic fibres⁷⁻⁹. Several studies have confirmed the use of different procedures to change the polyester structure, such as alkaline hydrolysis, UV radiation, plasma treatment, microwave radiation, gamma rays, surface grafting, and radical polymerization atomic transfers. Several studies focus on the change of internal structure of PET via heat, solvent, and enzymatic treatment. External molecular compounds are not present in heat treatment, whereas they are present in chemical treatment. Some studies revealed the weight decrease of polyester, while improving its functionality. Weight loss may arise from polymer structural deterioration and distortion. This

can result in polar groups formation, which can help to improve the hydrophilic properties of the polyester^{5,7,8}. Furthermore, thermal or solvent treatments may produce a decrease in fibre strength due to changes in internal structure, whereas enzymatic hydrolysis occurs only on the surface and hence has no effect on fibre strength¹⁰⁻¹⁵.

Few studies have proposed solvent-induced modification of the polyester (PET) by employing the trichloroacetic acid-methylene chloride (TCA-MC) solvent system. TCA-MC enters the polymer structure, weakening the polymer–polymer connection, causing widespread segmental motion, and decreasing the glass transition temperature (T_g). The polymer chains rearrange themselves into a lower free energy state, causing crystallisation to occur even while the polymer is swollen. PET's compact structure opens up, and as a result, structural modification (solvent-induced polymer crystallization) occurs, allowing external polymeric molecules to easily enter the PET structure¹³⁻²⁰.

Previous research, mostly focused on TCA-MC-treated yarns, has reported that the amount of structural change in the polymer varies with the yarn type, reagent concentration, and treatment period²¹⁻²⁴. However, no detailed research on the effect of TCA-MC on fabric has been conducted so far. The effect of varied TCA-MC concentrations on structural

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rearrangement of polymer chains in polyester fabric is explored in this research. An attempt has made to optimize the TCA-MC concentration across various polyester fabric structures in order to facilitate the simple entry of exogenous substances into the polymer system.

2 Materials and Methods

The characteristics of micro polyester fabric and texturized polyester fabric used in this study are shown in Table 1.

Laboratory grade Trichloro acetic acid (TCA) (CCl_3COOH) weighing 163.38 g/mol, methylene chloride (MC) (CH_2Cl_2) weighing 84.93 g/mol, and acetone (CH_3COCH_3) were used. The solution of TCA-MC was prepared according to the required concentration having a pH range of 1.2-1.8. Polyester fabric samples (micro polyester and textured polyester) were treated for 1, 3 and 5 min at room temperature ($\sim 30^\circ\text{C}$) with 0.5%, 1%, 3%, 5% and 10% TCA-MC concentration. The material-to-liquor ratio was maintained at 1:100 and the solution was continuously stirred during treatment. The treated samples were then rinsed with methylene chloride to eliminate any remaining TCA residue on the fabric surface, followed by acetone rinsing. Then the fabric was squeezed and atmospherically air dried²⁰. The polymer chain of the polyester (polyethylene terephthalate) interacts with TCA, resulting in solvent-induced polymerization. Terephthalic acid is formed as a result of the ester bond breaking. When the ester linkage is broken, hydroxyethyl trichloro acetate is produced. The changes are further confirmed with FTIR spectra. The chemical reaction is depicted in Fig. 1.

The treated samples and the untreated samples have been tested for loss in tensile strength, tear strength, weight loss, shrinkage and change in cover factor to evaluate the effect of the TCA-MC treatment on PET fabric structure. Tensile strength of fabric was tested according to ASTM standard D5035 using

Table 1 — Fabric specifications

Specifications	Micro polyester	Texturized polyester
Mass per unit area, g/m^2	160	120
Weave	2/1 twill	1/1 plain
Ends/cm \times picks/cm	72 \times 60	46 \times 28
Yarn linear density (warp \times weft), denier	270 \times 202.5	342 \times 360
Tensile strength (warp \times weft), N	1600 \times 1400	1400 \times 1200
Elongation-at-break, %	25 \times 20	40 \times 35
Thickness, mm	1.2	1.3

Tinus Olsen universal tensile tester²⁵. Tear strength was evaluated by using tongue tear tester in accordance with D 2261 test standard. Shrinkage percentage was calculated according to standard IS 9:1982. The mean of 10 readings was taken for interpretation of results. Bending length was used to determine structural stiffness using an ASTM D1388 cantilever test (by Testex stiffness tester). The specimen was 20cm \times 2.5 cm in size, and in each case average of five readings was taken. All the treated samples were tested for wetting and wicking properties. Wetting was measured in terms of total water absorbency test; it was determined by using 0.2% soap solution. A conditioned sample of the size 20 cm^2 was dipped in the prepared solution for 5 min, and then hung vertically to allow any extra water to drop down. It was then weighed and the percent gain in weight of the fabric sample was taken as a measure of its total absorbency. The vertical wicking test according to TAPCC standard measured the wicking ability of the test materials. In this case, the fabric samples of 20 cm \times 2.5 cm were cut along the weft direction and then hung vertically on the fabric holder. The specimens were then dipped into a 2 g/L cold brand reactive dye water solution until a mark created at a distance of 3 cm from the edge of the material. After 15 min, the vertical wicking ability, represented as the wicking height in cm, was then observed. The presence of functional groups and the change in surface structure due to TCA-MC treatment were analyzed by using FTIR and SEM respectively.

3 Results and Discussion

3.1 Effect of Treatment on Surface Morphology and its Characterization

The surface morphology investigation of the fabric samples shows that the TCA-MC interacted with PET polyester as demonstrated in Figs 2(a-f). Figure 2 depicts the surface morphology of untreated and TCA-MC-

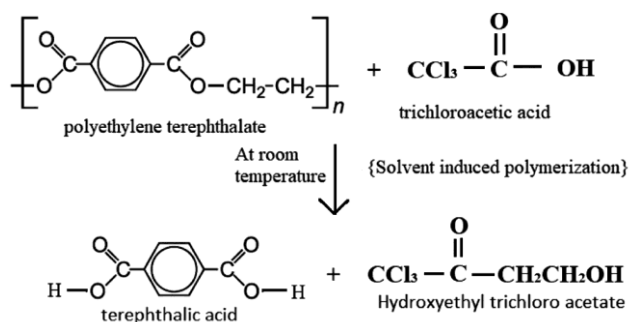


Fig. 1 — Schematic representation of chemical changes after treatment with TCAMC

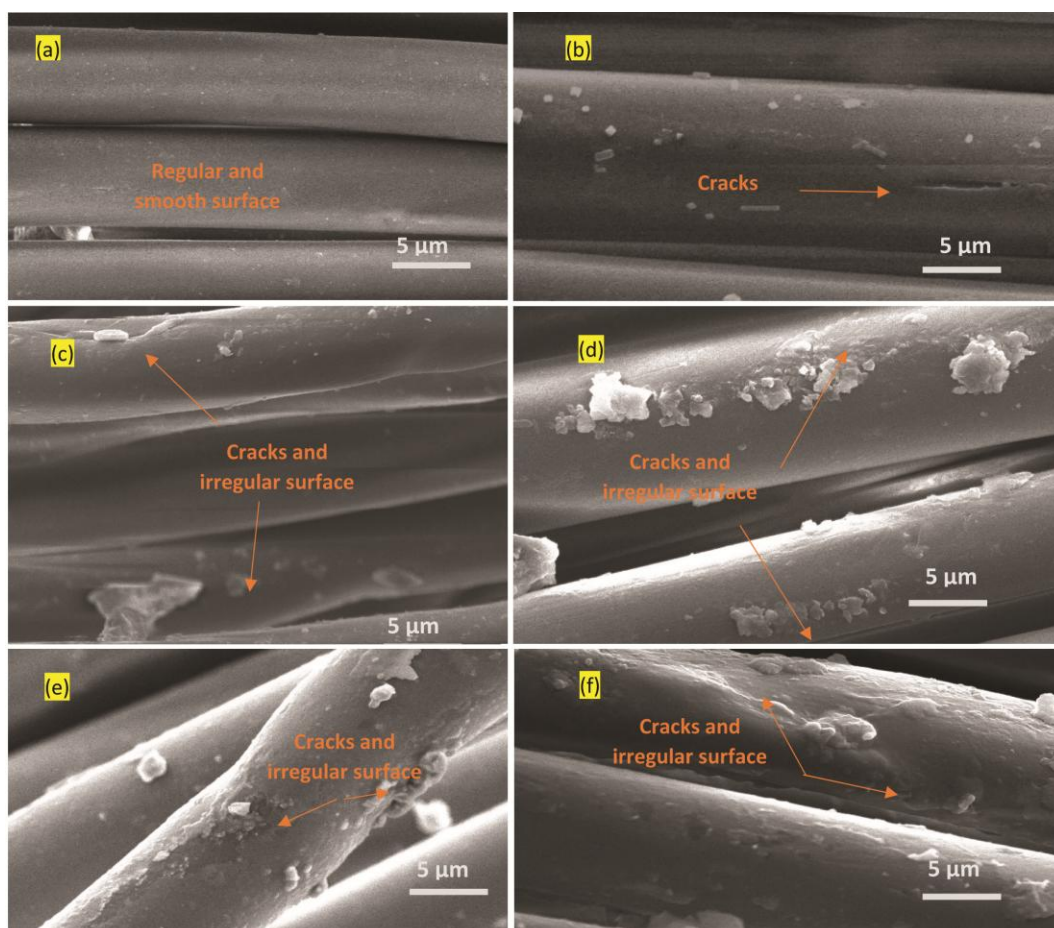


Fig. 2 — SEM analysis of micro polyester fabric samples (a) untreated, (b) 0.5% TCA-MC treated, (c) 1% TCA-MC treated, (d) 3% TCA-MC treated, (e) 5% TCA-MC treated and (f) 10% TCA-MC treated.

treated micro-polyester textiles at different levels (0.5, 1, 3, 5 and 10%). Figure 3 shows the untreated and 1% TCA-MC treated textured polyester fabric SEM images.

The FTIR analysis has been carried out to assess the possibility of a change in the spectra of the TCA-MC treated samples. The fingerprints of micro polyester and texturized polyester fabric samples treated with a 10% concentration of TCA-MC solvent are shown in Fig. 4. Figures 2 and 3 show that TCA-MC interacts with the polymer structures of treated samples. With TCA-MC treatment, bond order of the ester group($C=O$) decreases from 1709 cm^{-1} to 1706 cm^{-1} due to the attachment of H^+ ion of TCA. The formation of terephthalic acid results in polymer chains shrinking. Similarly, the increase in C-H stretching (band from 2933 cm^{-1} to 2975 cm^{-1}) of treated samples due to the attachment of O^- ions increases the C-H frequency of the polymer chain. The C-O bond order also increases to 1246 cm^{-1} with the H^+ ion attachment. Therefore, the C-O bond leads in increased material strength. It might assist in enhancing

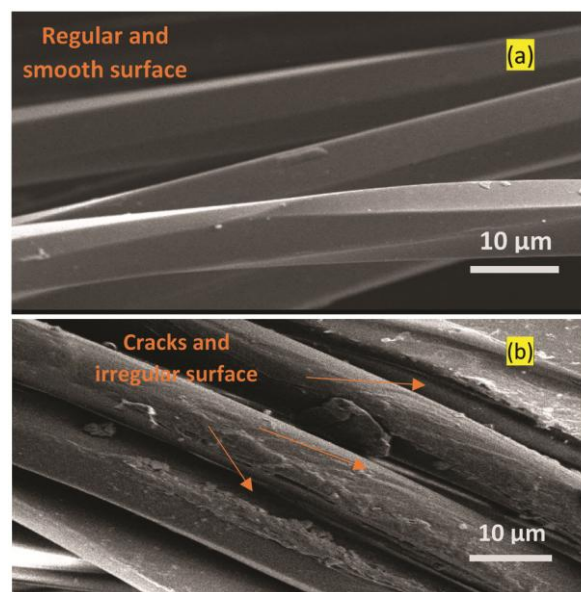


Fig. 3 — SEM analysis of texturized polyester fabrics (a) untreated, and (b) 1% TCA-MC treated

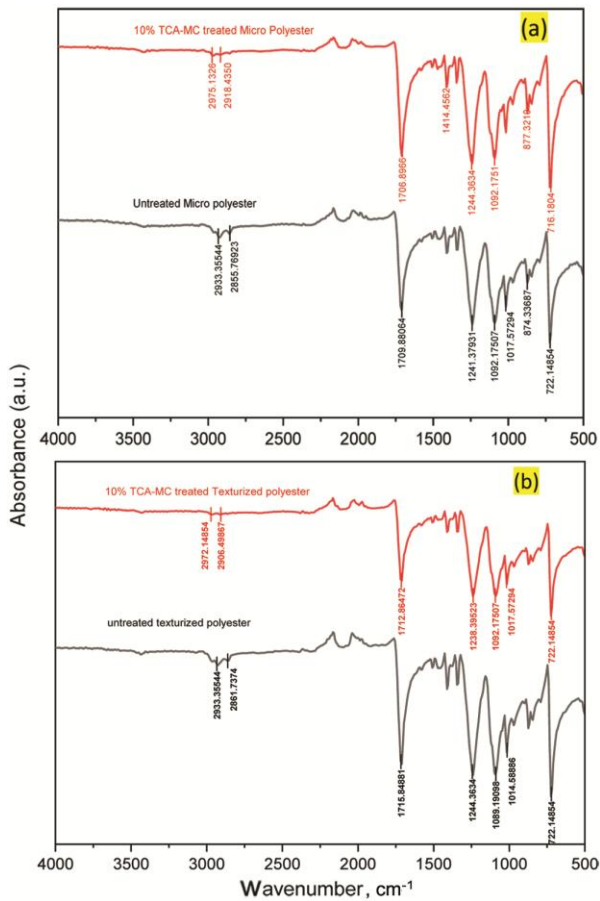


Fig. 4 — FTIR results for untreated and 10% TCA-MC treated (a) micro polyester fabric and (b) texturized polyester fabric

strength by shrinking 30-59.5% of micro-polyester and 33-66.5% for textured polyester. A similar pattern is observed for textured polyester samples. The ester linkage peaks are observed at 1715 cm^{-1} which after treatment with TCA-MC shifted to 1712 cm^{-1} . Table 2 reveals that TCA-MC treatment can result in a weight loss in both micro polyester (2.66-8.33%) and textured polyester (3.16-12.3%) with an increase in the concentration of the TCA-MC solvent. This loss in weight may happen due to the action of strong acid on the polymer chain, which results in the acid hydrolysis of ester linkage groups on the polymer chain. Acid-hydrolysis is also responsible for the formation of polar groups on the fabric surface, which allows innovative functional finishes to be attached to the fabric surface and improves the moisture absorption properties of the structure.

3.2 Effect of Treatment on Tensile Strength and Tear Strength Properties

The effect of different concentrations of TCA-MC on structural rearrangement of polymer chains in the

polyester fabric is investigated. From the literature, it is observed that the elongation of yarns increases by $>780\%$ after TCA-MC treatment²¹⁻²⁴. So, practically it becomes impossible to weave the fabric using TCA-MC treated yarns. Therefore, for using TCA-MC solvent induced modification for various applications, it is necessary that the effect of TCA-MC concentration and treatment time is studied on different fabric structures, and the optimum value of these parameters is selected without significantly affecting the tensile properties of the fabrics. The tensile strength and tear strength of untreated and treated fabric samples with TCA-MC at different concentrations and treatment duration is shown in Table 3.

It is observed that the tensile strength of both the fabrics decreases as the TCA-MC concentration or treatment time increases. The strength loss% lies in the range 1.8- 54.5% is observed in case of all TCA-MC treated fabric samples. The strength loss of 10-15% for polyester fabrics is normally accepted from practical point of view. A loss of more than 15% may be treated as degradation in the fibre and may lead to significant change in the properties of the fibre and the fabrics made there from. The warp tensile strength of micro polyester control sample is found 1600 N and that of texturized polyester control sample is 1400 N. It is obvious from Table 3 that the strength loss registered for micro polyester and texturized polyester at 10% TCA-MC concentration for 5min is 47.8% and 54.5% respectively. However, at lower concentration treatment (0.5%), the loss in tensile strength is not appreciably high irrespective of treatment time and the fabric structure. It is reported elsewhere²⁰ that TCA-MC attacks both the amorphous and crystalline regions of PET polymer and completely dissolves out the polyester fibre at 25% concentration (w/v) in 5 min. At lower concentrations, it initially enters the amorphous region and disrupts the polymer chains and creates more voids.

Figure 5 demonstrate that at higher concentration, the TCA-MC enters, after attacking the amorphous region, into the crystalline region and increases the segmental mobility, and ultimately weakens the polymer structure. This results in significant loss in tensile strength at 10% TCA-MC concentration. The extent of polymer interaction, modification and degradation depend on the concentration of TCA-MC used and the treatment time. It is also observed that texturization process, when compared to micro polyester, resists the penetration of TCA-MC molecules into the PET structure which affects the

Table 2 — Effect of TCA-MC concentration on shrinkage, weight change, bending length and wettability properties

Property	Duration min	Micro polyester						Texturized polyester					
		Control	0.5%	1%	3%	5%	10%	Control	0.5%	1%	3%	5%	10%
Shrinkage, %	1	0	30	34	35.2	36.5	43.3	0	32	35	42	52	55.5
	3	0	33.5	37	40.6	43.7	52	0	34.5	38.5	48	54	61
	5	0	35.5	40	44	46.5	59.5	0	37.5	49	55	58	66.5
Weight loss, %	1	0	2.66	3.08	3.46	4.11	4.98	0	3.16	3.88	4.76	4.85	6.5
	3	0	3.43	3.41	4.27	5.38	6.42	0	4.8	5.66	5.86	7.45	9.92
	5	0	3.52	3.74	5.14	6.34	8.30	0	5.10	6.11	7.82	9.15	12.3
Cover factor	1	21.1	23.1	25.2	25.3	25.3	25.7	15.9	17.9	18.5	18.8	19.3	20.6
	3	21.1	23.2	25.3	25.4	25.6	25.7	15.9	18.0	18.7	19.2	19.7	21.4
	5	21.1	23.4	25.5	25.6	25.7	25.8	15.9	18.1	18.8	19.6	19.8	22.7
Bending length, cm	1	6	6	5.8	5.7	5.6	4.9	5	5	4.9	4.8	4.7	3.5
	3	6	5.9	5.7	5.6	5.4	4.7	5	5	4.7	4.7	4.5	3.3
	5	6	5.8	5.6	5.5	5.2	4.5	5	4.8	4.8	4.6	4.1	3.2
Total water absorbency, g	1	4.6	5.5	7.4	7.9	8.5	9.4	2.6	3.4	4.2	5.1	6.3	7.4
	3	4.6	5.7	7.7	8.4	8.9	9.8	2.6	3.7	4.5	5.7	6.6	7.6
	5	4.6	5.8	7.8	8.7	9.3	10.0	2.6	3.8	4.8	5.9	6.8	7.8
Wicking, mm	1	40	41	44	55	61	69	65	68	71	77	82	93
	3	40	43	48	58	66	75	65	70	76	79	84	97
	5	40	46	52	62	71	78	65	73	78	83	86	102

0.5, 1,3,5 &10% are TCA-MC concentration.

Table 3 — Effect of TCA-MC concentration on tensile strength and tear strength of treated fabric samples

Property	Duration min	Micro polyester fabric						Texturized polyester fabric						
		Control	0.5%	1%	3%	5%	10%	Control	0.5%	1%	3%	5%	10%	
Tensile strength N	1	Mean	1600	1535	1427	1416	1286	988	1400	1374	1322	1269	1225	872
		Loss %	-	4.1	10.8	11.5	19.6	38.2	-	1.8	5.57	7.4	12.5	37.7
	3	Mean	1600	1485	1358	1296	1175	881	1400	1358	1224	1205	1193	758
		Loss %	-	7.2	15.1	19.0	26.5	44.9	-	3.0	12.57	13.9	14.8	45.8
	5	Mean	1600	1450	1207	1195	1120	835	1400	1287	1165	1125	1095	637
		Loss %	-	9.4	24.5	25.3	29.7	47.8	-	8.1	16.78	19.7	21.8	54.5
Tear strength N	1	Mean	140	137	130	125	115	102	90	88	83	75	67	55
		Loss %	-	2.1	7.1	10.7	17.8	27.1	-	2.0	7.7	16.7	25.5	38.9
	3	Mean	140	135	127	120	112	97	90	86	79	68	60	52
		Loss %	-	3.6	9.3	14.3	20.0	30.7	-	4.4	12.2	24.4	33.3	42.2
	5	Mean	140	133	124	118	110	95	90	80	72	61	57	48
		Loss %	-	5.0	11.4	15.8	21.4	32.1	-	11.1	20.0	32.2	36.7	46.7

0.5, 1,3,5 &10% are TCA-MC concentration.

subsequent solvent-induced-polymer modification as evidenced in Table 3. The tensile strength loss for treated micro polyester fabric is higher than that of texturized polyester fabric, irrespective of treatment concentration and time. Similar trends are also observed in case of tear strength properties of TCA-MC treated different polyester structures. Figure 5 shows that the tear strength of the TCA-MC treated samples decreases significantly as the concentration of the TCA-MC solvent increases. It

could be related to the above-mentioned breakdown of polymer chains. It has been discovered that at greater concentrations of TCA-MC, such as 10%, the decline is greatest in all situations. Figure 5 also shows that the treatment duration has a significant impact on the tear strength parameters of all treated samples. The largest tear strength loss of 32.1% for treated micro polyester and 46.7 % for texturized polyester is found after 5 min of treatment with a 10% TCA-MC concentration.

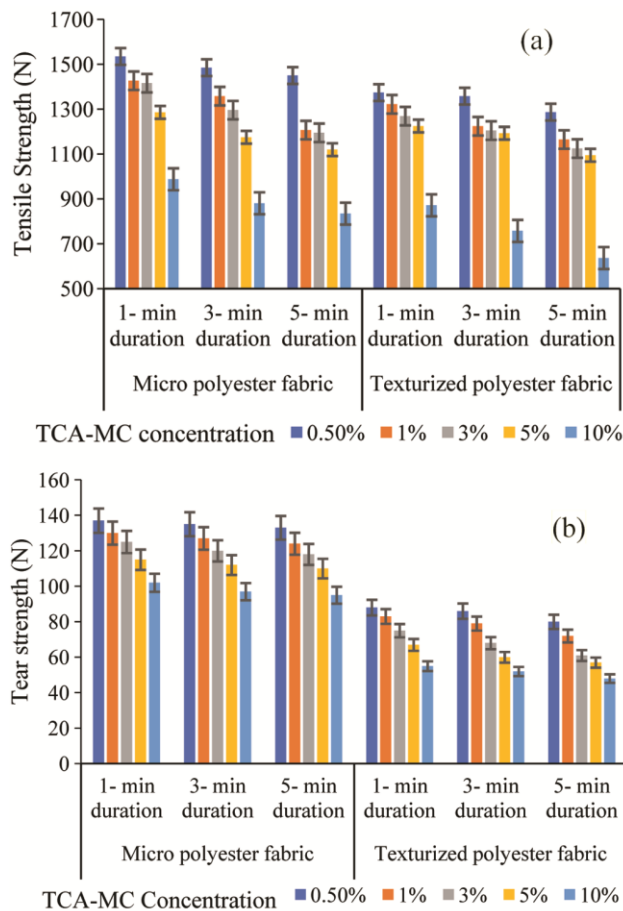


Fig 5 — Effect of TCA-MC concentration on (a) tensile strength properties and (b) tear strength properties

3.3 Effect of Treatment on Shrinkage, Weight Loss and Bending Length Properties

The degradation of polyester fabric after TCA-MC treatment is also evidenced with decrease in weight of the fabrics (Table 2). As the reagent concentration and treatment time increase, there is a significant increase in the weight loss for both fabric samples. The yarn denier and the yarn linear densities are found higher in micro polyester fabric than that of the texturized polyester fabric, which is also substantiated in the cover factor of the fabric (Table 2). Nevertheless, the total surface area available for the reagent to attack the polymer is higher in the case of micro polyester fabric than in case of texturized polymer, which leads to higher loss in strength for micro polyester fabric samples. When polyester fabric is treated with TCA-MC, solvent-induced-polymer modification is affected and in other words the solvent attacks the crystalline and amorphous regions of polyester structure.

As a result, the rearrangement of polymer chains and disintegration, which depends on the

concentration of TCA-MC, takes place and this influences the shrinkage. The shrinkage percentage of the fabrics is found to increase, as the concentration of TCA-MC increases (Table 2). The shrinkage percentage of TCA-MC treated micro polyester is lower as compared to the texturized polyester fabric. At 10% concentration and 5 minutes of treatment time, the shrinkage is 59.5% for micro polyester fabric and 66.5% for texturized polyester fabric. This shows that the interaction of TCA-MC with plain woven texturized fabric with open structures is easy than that with the twill woven micro polyester structure which is dense. Shrinkage percentage is also found to increase in all the fabric samples, as the treatment time increases. However, texturized polyester fabric shows statistically insignificant increase in shrinkage as the treatment time increases from 1 min to 3 min. Table 2 reveals that TCA-MC treatment can result in a weight loss in both micro polyester 2.66 -8.33% and textured polyester 3.16-12.3% with an increase in the concentration of the TCA-MC solvent. A maximum weight loss of 8.3% is observed in case of 10% TCA-MC treated micro polyester for 5-min duration. Similarly, treated texturized polyester samples show a weight loss of 12.3%. This loss in weight may happen due to the action of strong acid on the polymer chain, results in the acid hydrolysis of ester linkage groups on the polymer chain. The effect of TCA-MC concentration on the bending length of all fabric samples is shown in Table 2. In all situations, a decrement in bending length is observed as the concentration of the TCA-MC solvent increases. There is no noticeable difference in the bending behavior of the treated samples at low concentrations of TCA-MC solvent. However, with an increase in concentration from 5% to 10%, the decrease is considerable in both the cases. This could be attributed to an increase in the shrinkage per cent of the treated samples at higher TCA-MC concentrations, which aids in enhancing the cover factor value of the treated samples. The reduction in spacing increases the stiffness of the fabric sample.

3.4 Effect of Treatment on Wicking Behavior and Water Holding Capacity

Table 2 shows the wicking behavior and water holding capacity of the treated samples. It is evident that the treatment significantly affects the wicking and wettability properties of the treated samples. Wicking is the spontaneous flow of liquid in a porous medium driven by capillary force. Wicking is the most effective way to preserve comfort. For clothing

Table 4 — ANOVA analysis on tensile strength for TCA-MC treated micro polyester and polyester texturized fabrics

Effect of	Sum of square	Degree of freedom	Mean square	F calculated	Contribution %
Fabric type	79980	1	79980	92.25	5.70
Duration	127059	2	63529	73.27	9.06
Concentration	1162692	4	290673	335.26	82.94
Type * duration	915	2	458	0.53	0.06
Type*concentration	15583	4	3896	4.49	1.11
Duration*concentration	8662	8	1083	1.25	0.61
Error	6936	8	867		0.49

with excellent wicking capabilities, moisture from the skin is dispersed throughout the fabric, offering a dry feeling and the dispersion of the liquid makes it easy to evaporate. Vertical wicking data from Table 2 shows that treatment considerably alters the wicking behavior of treated samples. The wicking behavior of micro-polyester-treated samples improves from 2.4% to 48.7% as the TCA-MC concentration increases from 0.5% to 10%. In the case of micro-polyester fabric samples, a maximum wicking height of 78 mm is observed in 5 min treatment with 10% TCA-MC concentration, while a minimum wicking height of 41 mm is observed in the case of a 1 min treatment with 0.5% TCA-MC concentration. Similar results are observed in case of texturized polyester fabric samples. The treated samples show an improvement of 4.4-36.3% as the TCA-MC concentration increases from 0.5% to 10%. In the case of texturized-polyester fabric samples, a maximum wicking height of 102 mm is observed in the case of a 5 min treatment with 10% TCA-MC concentration, while a minimum wicking height of 68 mm is observed in 1 min treatment with 0.5% TCA-MC concentration. Water absorption has been shown to rise considerably with an increased concentration of the TCA-MC solvent. This may be attributed to fibre morphology changes related to test sample deterioration. The increase in wicking height may occur due to the structural rearrangement of the polymer chain, additional voids and cracks in the structure. Total water absorption of all treated samples has been measured to estimate the water retention capacity of treated fabric samples. Table 2 demonstrates that the treatment improves the water holding capacity of all treated samples. Test results show that, with increased concentration of TCA-MC solvents, the water retention capacity of all treated samples improves significantly.

Variance analysis (ANOVA) demonstrates that the tensile strength of polyester fabrics is controlled by

solvent concentration, treatment time, and fabric structure (Table 4). TCA-MC solvent concentration has a largest contribution of 82.94%, followed by treatment time, with a contribution of 9.06%. The fabric structure has a considerable contribution (5.70%) to fabric tensile strength decrease. Type of fabric together with concentration of TCA-MC has a substantial contribution of 1.11%.

4 Conclusion

The solvent induced polymer crystallization is taken place in the polyester structure due to pretreatment with TCA-MC reagent. The following conclusions are drawn from this investigation:

4.1 TCA-MC reagent is found to be highly interactive with polyester even at a very low concentration of up to 0.5% for 1 min duration. The treatment decreases the tensile strength of both the fabric structures. The loss in strength significantly relates to the concentration of the reagent as well as the treatment time. Significant strength loss is observed at higher concentration of 10% TCA-MC concentration for 5 min duration for both micro polyester and texturized polyester fabric. This is also reaffirmed in SEM images of the fibres, before and after treatment.

4.2 The extent of loss in strength for minimum to maximum concentration and time used in this investigation varies from 4.06% to 47.81% for micro polyester fabric from 1.85% to 54.5% for polyester texturized fabric. Texturized polyester fabric has a wider range of loss in tensile strength. Maximum desired surface irregularity is achieved with acceptable range of strength loss (~15%) at 1% TCA-MC concentration. This has also been investigated from the SEM analysis.

4.3 Bending length values show a decreasing trend as the concentration of TCA-MC increases from 0.5% to 10% with a treatment period of 1-5 min.

4.4 It is noticeable that the wicking height of the TCA-MC treated micro-polyester samples is significantly improved (up to 78 mm) than the wicking height (40mm) of the control micro-polyester sample.

4.5 ANOVA analysis shows that the interaction of all the three variables- TCA-MC concentration, treatment duration, polyester structure is found to be significantly influenced by the structural changes in both the polyester fabrics.

4.6 The investigation pays the way for future research on imbuing functional finishes, for instance antimicrobial and flame retardant finishes, into PET polymer. This study suggests that the TCA-MC treatment will facilitate the easy entry of external molecules into the compact structure of PET polyester.

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