Tensile Behavior of Polyester Yarns Modified by Trichloroacetic Acid–Methylene Chloride Treatment

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SYNOPSIS

The effect of trichloroacetic acid–methylene chloride (TCAMC) solvent system on the mechanical properties of poly(ethylene terephthalate)-based flat, high twist, and spun yarns were studied. Pretreatments were carried out in an unrestrained state at different concentrations for various times and without employing any heat treatment. The stress–strain behavior of yarns was analyzed. The stress–strain curves showed that the treated yarns exhibit different morphologies. A significant plastic flow region could be detected in treated yarns. The higher interaction between the reagent and the polyester revealed the closeness of their solubility parameter values. Structural modification of yarns due to TCAMC treatment led to an increase in elongation without significant reduction in strength. The reagent caused reduction in initial modulus, yield stress, and an increase in work of rupture. The increase in work of rupture was higher in flat yarn but not appreciable in the other two yarns. Statistical analysis of the test data was done. © 1996 John Wiley & Sons, Inc.

INTRODUCTION

Poly(ethylene terephthalate) (PET) fibers generally exhibit low surface energy and limited chemical reactivity, resulting in poor moisture absorption.[1] Because of this, dyeing of PET is somewhat difficult at temperatures below 130°C. Improvement in dyeability by reducing the glass transition temperature (T_g) is possible by appropriate modification of PET polymer matrix. When PET fibers are pretreated with highly interacting solvents, under suitable conditions the internal structure, especially the amorphous region, is changed, and thereby more voids, crackes, etc., are produced, which facilitate the entry of dye molecules at lower temperatures. With such polymer modification treatment, although aimed to improve the dyeability, care must be taken to ensure the required performance characteristics of the modified polymer.

Our earlier studies[2-4] have shown that the interacting power of trichloroacetic acid–methylene chloride (TCAMC) solvent system with the PET is very high and the reagent attacks the polymer matrix, disintegrates, and finally dissolves out the PET at about 25% (w/v) concentration in 5 min duration at room temperature condition (about 30°C). This indicates that the solubility parameter of TCAMC reagent is very close to the solubility parameter of PET.

With this background in mind, an attempt was made to develop a carrier-free low-temperature dyeable PET. It is expected that at certain lower concentrations of TCAMC treatment the compact structure of polyester opens up. In this investigation we have reported the tensile behavior of three different types of PET yarn that have been pretreated with TCAMC reagent at various concentration levels for 5 min time and at room temperature condition.

EXPERIMENTAL

Materials

The following three commercially available PET-based polyester yarn samples were used for this investigation. (1) flat filament yarn (FFY); (50D/36); (2) high twist filament yarn (HTFY); (40D/

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27); (2200 ± 5% TPM); (3) spun polyester yarn (SPY); (Ne-20/1).

The FFY sample was obtained from Orissa Synthetics (a group of J. K. Corp. Ltd.), Dhenkanal, India and the HTFY and SPY were procured from Reliance Industries Ltd., Bombay, India.

Chemicals

The laboratory grade (LR) trichloroacetic acid ($CCl_3 \cdot COOH$), methylene chloride ($CH_2 \cdot Cl_2$), and acetone ($CH_3 \cdot CO \cdot CH_3$) were used.

Pretreatment

Sample skeins, weighing approximately 2 g, were prepared by utilizing the shirley yarn winding device. Pretreatment of these skeins with TCAMC reagent was carried out in a specially made closed trough at room temperature (30–33°C). The yarn samples in an unrestrained state were immersed in the reagent of desired concentration for 5 min duration. The material-to-liquor ratio was maintained at 1 : 100, and the contents were shaken manually at regular intervals to ensure uniform treatment. After the treatment, the samples were rinsed with pure methylene chloride followed by acetone to remove any adhering reagent on the yarn. The treated samples were squeezed and air dried at atmospheric condition, taking advantage of the quick evaporation of acetone at the lower temperature condition. After drying, the samples were conditioned at a standard atmospheric condition of 65% RH ± 2% and at 27 ± 2°C before being subjected to various testing procedures.

TEST METHODS

Yarn Denier

A direct method of weighing a definite length of yarn as per the Bureau of Indian Standards (BIS)[5] was used. A Mettler ultramicrobalance (0.0001 mg precision) was utilized to determine the weight of the samples accurately. The mass in grams of 9000 meters length of yarn was calculated for determining the denier of filament yarn samples.

Tensile Strength and Extension at Break

Tenacity of the yarn samples was measured on an Instron Tensile Tester (Model No. 6021). A specimen length of 50 mm was used at an extension rate of 50 mm/min. More than 30 specimens were tested

in each case. From the load–extension curves, the average breaking strength and percent extension at break were determined.

The stress–strain curves were drawn by superimposing five selected load–extension curves, where the breaking load was close to the average value, normalizing the characteristic curves and reconstructing the same in terms of stress in g/den and strain in percent. The area under the stress–strain curve was measured and the work of rupture was, thus, calculated.

RESULTS AND DISCUSSION

Strength–Elongation Properties

Table I shows the tensile strength and elongation of both the untreated control and the pretreated FFY, HTPY, and SPY yarns with TCAMC reagent at various concentration levels for 5 min duration at room temperature in the unrestrained state. It is known that textile polymers are normally partially crystalline in nature and their mechanical properties are mainly dependent on its percentage.

The PET fibers are partially crystalline in nature and are comprised of repeated ethylene segment units as well as aromatic rings, which influence the intrinsic stiffness of the chain. The tensile strength of PET is a function of the degree of orientation in polymer, which has a direct impact on application requirement and service performance in a finished textiles. The various processing parameters such as spinning, winding speed, stretching, thermal treatment, application of speciality finishes, and the action of different chemicals and solvents influence the strength–elongation properties of the substrates. Besides, the various secondary valence forces present in the polymer also influence the mechanical properties.[6]

It can be seen from Table I that the tenacity of the treated yarns decreases with an increase in concentration of the reagent, irrespective of the sample type. The loss in strength of FFY due to the treatment is higher than that of HTFY and SPY samples. This trend is depicted in Figure 1. A strength drop of about 24% is observed in FFY at extreme treatment (5% concentration), and in the case of SPY and HTFY, a loss of about 10% and 4%, respectively, are noted. In FFY, it is seen that there is a sudden decrease in strength at low concentration of 1% treatment, and thereafter the magnitude of loss in strength is not appreciably high. Contrary to the strength loss, it is interesting to note (Table I and Fig. 2) that the elongation of all the treated samples

Table I Strength and Elongation of Untreated and Treated Polyester Yarns

| Percent Concentration w/v	Flat Filament Yarn				High Twist Filament Yarn				Spun Yarn			
	Tenacity g/den	Percent Elongation	Percent Decrease in Strength	Percent Increase in Elongation	Tenacity g/den	Percent Elongation	Percent Decrease in Strength	Percent Increase in Elongation	Tenacity g/den	Percent Elongation	Percent Decrease in Strength	Percent Increase in Elongation
0	4.12	35.2	—	—	2.85	38.3	—	—	2.87	13.5	—	—
1	3.57	61.0	13.35	73.3	2.83	43.5	0.70	13.6	2.84	31.2	1.05	131.1
2	3.49	63.8	15.29	81.3	2.81	46.6	1.40	21.7	2.83	36.6	1.39	171.1
3	3.40	66.7	17.48	89.5	2.80	47.0	1.75	22.7	2.80	42.0	2.44	211.1
4	3.35	75.2	18.69	113.6	2.78	47.6	2.46	24.3	2.65	49.1	7.67	263.7
5	3.15	84.2	23.54	139.2	2.73	47.9	4.21	25.1	2.59	56.0	9.76	314.8
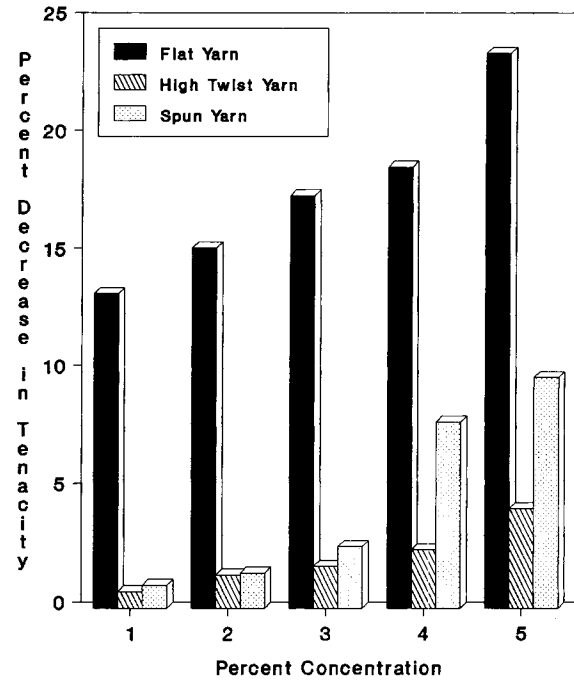
Figure 1 Effect of concentration on tenacity.

are increasing progressively from high to very high percentage levels. The trend in increase in elongation of SPY is very high, which is in the order of 131% to 315%, whereas, FFY registered 73% to 139% and a lower value of 14% to 25% in the case of HTFY at lower to higher concentration treatments (1% to 5%) of TCAMC, respectively. A close look at the
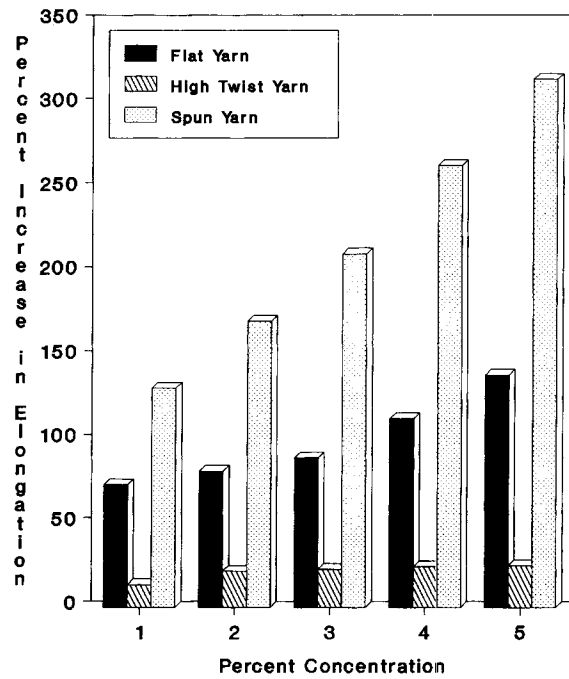

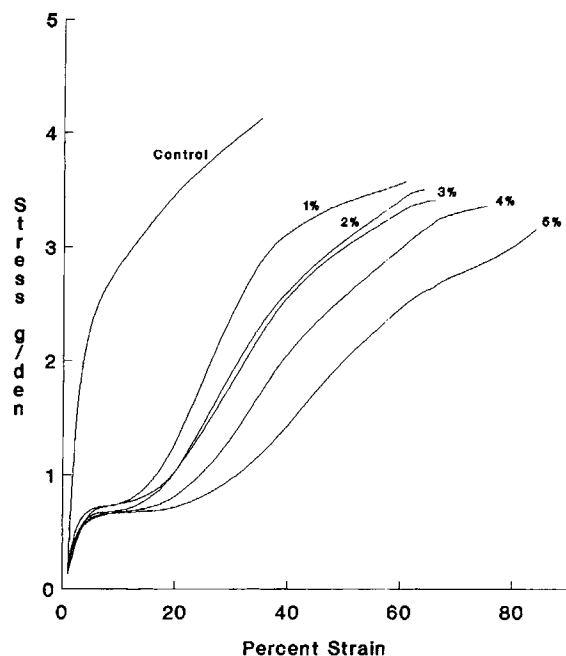**Figure 2** Effect of concentration on elongation.

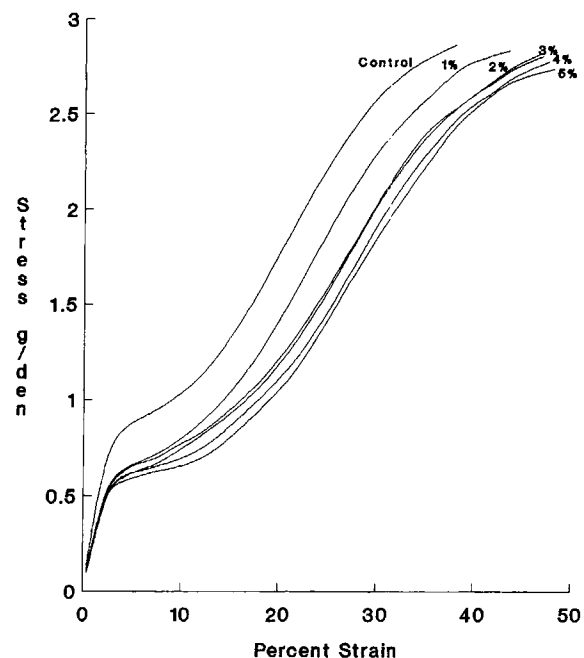**Figure 3**   Stress–strain behavior of polyester flat yarn.

Figure 4   Stress–strain behavior of polyester high twist yarn.

Table I indicates that SPY exhibits higher elongation with lower strength loss of about 10% at an extreme treatment level. On the other hand, a higher strength loss of 24% with a significant increase in elongation (139%) is observed in FFY, and an insignificant strength drop (4%) with an appreciable improvement in elongation (25%) is obtained in HTFY. Considering that in industry a loss in tenacity higher than 20% is not recommended, the losses obtained for all the three types of yarn under various pretreatment conditions, except 5% treatment level of FFY, are within the level of acceptance.

The interpretation of strength–elongation characteristics and other related properties such as initial modulus and work of rupture of all types of yarn are discussed below, making use of their respective stress–strain curves.

Stress–Strain Characteristics

The stress–strain curves of TCAMC treated FFY, HTFY, and SPY samples with their relevant control ones are depicted in Figures 3, 4, and 5, respectively. It is seen that the stress–strain curves of all the three types of yarn are quite different, indicating different morphologies. It is known that the stress–strain behavior of polymers are greatly influenced by the morphological state of their samples. Generally, load–elongation curves are used by many authors to describe the morphological changes of textile substrates. However, Mocherla et al.[7] studied the stress–

strain behavior of oriented PET by infrared (ir) technique with a view to correlate the state of molecular bonds in the polymer chain to the stress behavior. The principle of this ir method is that the ir-vibrational frequencies of a polymer molecule are dependent on the interatomic bond strengths and valence angle. While the bonds are distorted by
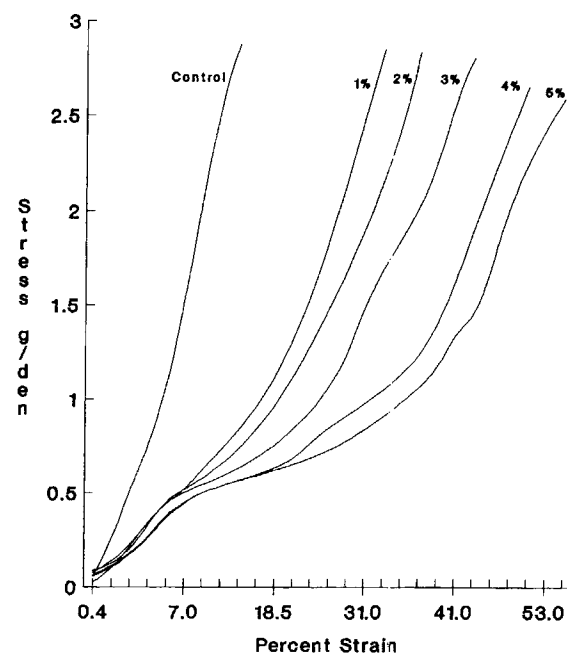

Figure 5   Stress–strain behavior of polyester spun yarn.

Table II   Initial Modulus of Polyester Yarn

Percent Concentration w/v	Flat Filament Yarn		High Twist Yarn		Spun Yarn	
	Initial Modulus g/den	Percent Reduction in Initial Modulus	Initial Modulus g/den	Percent Reduction in Initial Modulus	Initial Modulus g/den	Percent Reduction in Initial Modulus
0	63.76	—	41.69	—	20.01	—
1	16.00	74.91	30.52	26.79	14.12	29.44
2	16.90	73.49	28.47	31.71	14.12	29.44
3	16.00	74.91	27.46	34.13	14.12	29.44
4	15.29	76.02	26.44	36.58	10.09	49.58
5	13.65	78.59	25.42	39.03	10.09	49.58

stress, the frequency of an absorption band will shift to longer wavelengths, and as a result, the band will undergo sharp distortion. This shift in frequency is proved to be a linear relationship with stress. Further, the authors have found that, of all the skeletal ir-bands, the ir-band of 973 cm^{-1} is more suitable for investigating the stress–strain relationship of polyester. This band represents the transisomers of the PET that are associated with either the crystalline units or with the extended units by interlamellar links.[8]

Effect of Treatment on Strength

As discussed in the foregoing section, the strength of treated yarn decreases with an increase in concentration of TCAMC treatment. The extent of loss varies from low to medium and depends on the type of polyester yarns used. The TCAMC treatment causes major changes in the shape of the stress–strain curves of PET yarns. A sudden change in the shape of the curves even at 1% treatment level showed very high interacting power of the solvent with the polyester. Depending on the extent of PET–TCAMC interactions, a large range of solvent-induced structural changes may take place in the yarn.

The high interacting power of the TCAMC reagent with PET can be explained by the structural model of the PET polymer, which quantitatively describes the relationship between the polymer structure and mechanical properties. It is known that the PET polymer consists of microfibrils that are arranged in columns, and in between these microfibrils oriented noncrystalline domains are present. Disordered domains are also present in between the well-oriented crystallites. Prevorsek[9] has found that oriented noncrystalline domains consisting more or less of extended polymer chains exist between microfibrils. Peterlin[10] also postulated similar theory and concluded that the extended amorphous domains are considered to be one of the essential elements of fiber structure with regard to strength and elongation properties. The author further concluded that these domains are plasticized during solvent–polymer interaction. The extent of plasticization depends on the interacting power of the solvent with the polymer, the solubility parameter values of the two systems, and the treatment environment such as time, temperature, etc.

The solvent-induced crystallization takes place when the solubility parameter values of both the solvent and the polymer are close to each other.[11,12]

Table III   Yield Point of Polyester Yarns

Percent Concentration w/v	Flat Filament		High Twist Yarn		Spun Yarn	
	Yield Stress g/den	Yield Strain %	Yield Stress g/den	Yield Strain %	Yield Stress g/den	Yield Strain %
0	2.29	4.3	0.76	2.5	0.48	2.4
1	0.68	4.3	0.56	2.5	0.34	2.4
2	0.66	4.3	0.53	2.5	0.34	2.4
3	0.64	4.3	0.53	2.5	0.34	2.4
4	0.59	4.3	0.51	2.5	0.34	4.2
5	0.57	4.3	0.51	2.5	0.34	4.2

Table IV   Effect of Reagent on Plasticity of Polyester Yarns

Percent Concentration w/v	Flat Filament Yarn Plastic Flow %	High Twist Filament Yarn Plastic Flow %	Spun Yarn Plastic Flow %
0	—	4.00	—
1	9.35	6.00	7.80
2	10.20	6.50	8.40
3	14.45	7.00	9.60
4	18.70	9.50	18.60
5	24.65	10.00	22.50

Besides, it is generally agreed[13,14] that in most cases solvents do not penetrate in to the crystalline regions in polymers and, therefore, do not significantly affect the strength of the polymer. The changes in strength and other mechanical properties of solvent-treated PET yarn is a consequence of the increase in chain mobility, resulting in internal structural rearrangement of the polymer chains.[15]

Based on the above discussions, it appears that the TCAMC reagent penetrates into the polymer matrix and attacks the oriented amorphous domains and breaks the intermolecular bonds, leaving the crystalline domains unaffected. The strength drop recorded at extreme treatment for HTFY is only around 4% and about 10% for SPY samples. However, significant strength loss (24%) is recorded in the case of FFY at 5% treatment, indicating that at higher concentration the reagent is capable of penetrating into the crystalline region of the PET and breaks down various secondary forces present in it after attacking the amorphous region.

Influence of Reagent on Elongation

The treated polyester samples, irrespective of the state, showed higher deformation and gradually in-

creases as the concentration of the reagent increases (Table I). In general, the extension of any fiber is controlled by length, the configuration of chain molecules, the energy content, and the number of chemical bonds that hold them together in the amorphous regions of the fiber. It is reported[16] that a high elastic material, after being extended to several times its original length when released, will return to the original length, exhibiting little or no permanent deformation as a result of the extension. This may perhaps be the behavior of crystalline solids that cannot normally be extended to more than a very small fraction of their original length without undergoing breakage. On the other hand, some polymers can undergo large deformation without significant fracture up to certain level, but do not completely return to their original length upon removal of the deforming stress. However, they possess elastic response at smaller strains referred as Hookean region. This phenomenon is seen in the TCAMC-treated polyester yarns. It is known that the chain molecules in a fiber are held together by lateral forces due to covalent bonds, hydrogen bonds, and Van der Waals forces. The flexibility of the chain molecules depends on the holding power of these lateral forces. It appears that the TCAMC re-

Table V   Work of Rupture of Polyester Yarns

| Percent Concentration w/v	Flat Filament Yarn Work of Rupture (g wt cm)	Percent Increase	High Twist Yarn Work of Rupture (g wt cm)	Percent Increase	Spun Yarn Work of Rupture (g wt cm)	Percent Increase
0	239.26	—	160.12	—	262.00	—
1	356.76	49.11	174.19	8.79	516.23	97.03
2	360.24	50.56	182.78	14.15	609.12	132.49
3	389.92	62.97	187.36	17.01	632.95	141.58
4	414.72	73.33	189.75	18.50	832.82	217.87
5	452.33	89.05	192.50	20.22	1023.85	290.78
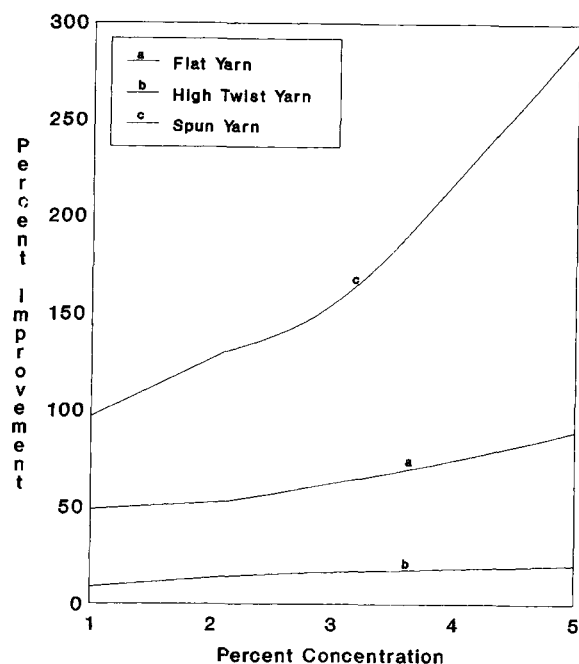
Figure 6   Work of rupture of treated yarns.

agent produces some measure of freedom for molecular movements by relaxing the aforementioned lateral forces, and ultimately, higher extensibility resulted. The degree of molecular relaxation due to treatment is progressively increasing with the concentration of reagent and recorded very high in the case of SPY and low for HTFY samples.

Changes in Initial Modulus

The TCAMC treatment influences the initial modulus of the PET yarns as seen in Table II. The slope of the stress–strain curve in the elastic region represents the modulus of yarn. The modulus of the yarn is affected by the contribution of the modulus of the crystallites and the type of low transfer between the phases. Structurally, all synthetic fibers like nylon and polyester consists of three phases: crystalline phase, amorphous phase, and extended chain interfibril domains. This extended chains are the strongest element of the fiber structure and have an adverse effect on fiber modulus, as discussed previously.[9,10] It can be seen from Table II that the percent reduction in modulus is high in FFY, and higher concentration treatments do not significantly influence the percent reduction further. Similar reduction is also observed in the other two types of yarns in which the reduction recorded in the case of HTFY is linear with the treatment concentration. The trend observed for SPY is same as FFY, but the modulus declined by 50% only at extreme treatment.

Table VI   Statistical Analysis of Breaking Strength of Polyester Yarns

Percent Concentration w/v	Flat Filament Yarn				High Twist Filament Yarn				Spun Yarn			
	Sample Variance	Standard Deviation	Standard Error	CV%	Sample Variance	Standard Deviation	Standard Error	CV%	Sample Variance	Standard Deviation	Standard Error	CV%
0	0.02	0.14	0.03	3.40	0.01	0.09	0.12	3.16	1.64	1.28	0.23	4.95
1	0.01	0.10	0.02	2.80	0.01	0.07	0.01	2.47	1.44	1.20	0.22	4.69
2	0.00	0.06	0.01	1.72	0.00	0.06	0.01	2.14	2.10	1.45	0.26	5.69
3	0.00	0.03	0.00	0.88	0.00	0.05	0.01	1.79	1.77	1.33	0.24	5.28
4	0.01	0.04	0.00	1.19	0.01	0.09	0.01	3.24	2.20	1.48	0.27	6.21
5	0.01	0.07	0.01	2.22	0.02	0.13	0.02	4.76	2.08	1.44	0.26	6.17

Table VII   Statistical Analysis of Breaking Elongation of Polyester Yarns

Percent Concentration w/v	Flat Filament Yarn				High Twist Filament Yarn				Spun Yarn			
	Sample Variance	Standard Deviation	Standard Error	CV%	Sample Variance	Standard Deviation	Standard Error	CV%	Sample Variance	Standard Deviation	Standard Error	CV%
0	6.55	2.56	0.47	7.27	8.97	3.00	0.42	7.83	0.42	0.65	0.12	4.81
1	8.97	3.00	0.55	4.92	6.00	2.45	0.35	5.63	1.56	1.25	0.23	4.01
2	5.69	2.39	0.44	3.75	3.24	1.80	0.25	3.86	2.09	1.45	0.26	3.96
3	4.28	2.07	0.38	3.10	2.59	1.61	0.23	3.43	3.98	1.99	0.36	4.74
4	3.77	1.94	0.35	2.58	9.58	3.10	0.44	6.51	5.45	2.33	0.43	4.75
5	7.41	2.72	0.50	3.23	13.49	3.67	0.52	7.66	6.65	2.58	0.47	4.61

Penetration of the reagent leads to solvent–polymer interaction and plasticization nature, thereby decreasing the modulus. The rate of plasticization is more in the case of FFY.

Changes in Yield Behavior

The elastic property of the treated yarns was determined in terms of yield point and the corresponding yield stress and yield strain (Table III). Yield point is considered as the point where the yarn transists from an elastic behavior to a plastic behavior. Different theories like the Whole theory of the WLF equation and Eyring theory of viscosity indicate the changes in molecular configuration that must occur for an overall yield to take place. All the molecular theories of yield have revealed that the prime cause of plasticity in polymers is the shear component of the tensile stress that is capable of inducing flow.[17] Treatment with 1% TCAMC changes the yield stress to a larger extent, after which the changes are marginal. This indicates that the penetration of the reagent modified the PET structure, irrespective of the concentrations used for the modification. This modification induces the plastic region in the stress–strain behavior in all the cases. The stress–strain behavior of yarn is characterized by three regions, namely a Hookean region followed by plastic region, which shows flow behavior and a strain-hardening region. In the plastic region, the molecular chains become oriented, and thereby irreversible elongation resulted. It can be seen from Table IV that the FFY and the SPY control samples do not show any plastic flow region, whereas the HTFY shows little plastic flow region. Treatment with TCAMC modified the structure, and all the samples possess plastic flow regions and recorded as highest at 5% treatment.

Changes in Work of Rupture

The toughness of the treated materials is represented in Table V and Figure 6. Comparing the work of rupture of treated yarns with their respective control ones, it is obvious that very high energy is required to break the SPY samples. Similar higher improvement in work of rupture is also seen in FFY. However, there is no appreciable improvement in the case of HTFY, perhaps due to the lower elongation observed after TCAMC treatment. It is interesting to note (Fig. 6) that the percent increase in work of rupture of SPY is more or less linear with the treatment concentrations and recorded as high as 290% at extreme concentration. This trend perhaps correlated with the elongation property of the treated yarns because the work of rupture is pro-

Table VIII   Statistical Estimation Analysis of Increase in Breaking Elongation of Pretreated Yarns

| Percent Concentration (w/v) | Increase in Elongation at 95% Confidence Limit | | | | | |
| | Flat Filament Yarn | | High Twist Yarn | | Spun Yarn | |
	Mean	Range	Mean	Range	Mean	Range
1	25.80	24.36–27.24	5.18	4.09–6.27	17.68	17.16–18.19
2	28.60	27.32–29.88	8.33	7.35–9.31	23.14	22.56–23.72
3	31.52	30.32–32.73	8.70	7.74–9.66	28.55	27.78–29.31
4	39.77	38.43–41.12	9.30	8.09–10.51	35.55	34.67–36.44
5	48.99	47.63–50.36	9.58	8.24–10.91	42.49	41.52–43.46

portional to the elongation behavior, irrespective of the nature of yarn.

Statistical Significance of Experimental Results

As stated, in the three types of yarn more than 30 samples were subjected to the strength–elongation test. The results were analyzed statistically and presented in Table VI for breaking strength and Table VII for elongation at the break. It can be seen (Table VI) that in the case of FFY samples the amount of variation from the average of all the treated samples, irrespective of the concentration of reagent, is lower than the corresponding control ones, justifying the greater uniformity of the treatment. Similarly, the relative dispersion of the treated yarns is also smaller than the untreated yarn. A similar observation is also noticed in the HTFY samples up to 3% concentration. At higher concentrations, although there is nil difference in variability at 4% level, both the standard deviation and variability and CV% of the treated samples are slightly higher than the control. In SPY, it is seen that both the standard deviation and CV% are gradually increasing with the treatment concentrations.

In the breaking elongation (Table VII), although the sample variance recorded is slightly high, the ultimate CV% of all the treated samples is well below the CV% of the corresponding control yarns. The descriptive statistics show that the variability of tensile strength and elongation data of the treated samples is within the acceptable limit.

In order to find out the range in increase in breaking elongation at 95% confidence limits, an estimation analysis was done between control and 5 min treated samples, as suggested by Leaf[18] and the results are given in Table VIII. For instance, the increase in breaking elongation at 95% confidence limits for 5% treated FFY yarn is in the range of 47.6% to 50.4%.

CONCLUSIONS

The TCAMC treatment causes morphological changes in the poly(ethylene terephthalate) type polyester yarns. The treatment increases the elongation and reduces the tenacity of yarn at various extents, depending on the treatment environments adopted and the type of yarn used. The relative increase in elongation observed in spun yarn is 315%, with a strength loss of a mere 10%, whereas, the high twist yarn registered 25% elongation and 4% strength drop at 5% treatment for 5 min duration. In the case of flat yarn, although the decrease in tenacity is as high as 24% at 5% concentration, the 4% treatment exhibited around 19% loss with an appreciable improvement of 114% elongation. The changes effected in the polymer matrix proved high interacting power of the reagent with the polyester and also justified that the solubility parameter value of the reagent is close to that of polyester polymer. The reagent influences the initial modulus and yield stress of the treated yarns, which are inversely proportional to the treatment concentrations. Another interesting feature observed in the treated yarns is the very high increase in work of rupture. The increase noticed in spun yarn is about four times greater than the control at 5% treatment for 5 min duration. Similar higher improvement is also seen in flat yarn, but not in high twist yarn. A statistical analysis has been carried out on the variability of the test results.

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