Volume 6, Issue 3, Spring2010

A NEW METHOD OF INVESTIGATING THE STRUCTURE BY WEIGHT LOSS OF **POLYESTER FIBERS**

M. Dhinakaran*a, B.S. Dasaradan^b & V. Subramaniam^c

- a. Department of Textile Technology, Kumaraguru College of Technology, Coimbatore -641006
- b. Department of Textile Technology, PSG College of Technology, Coimbatore- 641004
- c. Department of Textile Technology, Java Engineering College, Tiruninravur, Chennai-600024 *a b c Tamil Nadu, India

ABSTRACT

An investigation on the study of structure of polyester fibers following drawing heat setting, hydrolytic and light fastness studied by weight loss following alkaline hydrolysis is reported. It is shown that weight loss is very sensitive to these treatments and can be considered as tool for determining the structure of polyester fibers.

Keywords: light fastness, heat set, hydrolytic, Polyester, weight loss

1. Introduction

Polyester fiber which is produced from polyethylene terephthalate has some unless deficiencies modified fabrics produced from polyester fibers lack in moisture absorption and prone to pilling. A number of techniques have been developed for altering their properties. They are being considered for medical textiles today. A considerable amount of work has been done on the weight loss of polyester fabrics in the past to improve their aesthetic properties [1, 2]. Waters [3] was the first to describe the alkaline hydrolysis of poly (ethylene terephthalate) varns. It was demonstrated that weight loss of polyester fibers could be used as a means of studying the fine structure of polyester fibers [4]. However the work was confined to only drawn and undrawn fibers and not for fibers which were heat set and subjected to hydrolytic and light fastness. Recently the

the effect of draw texturing parameters on the structural aspects of polyester textured yarn has been investigated [5] Polyester (poly ethylene terephthalate), PET both industrial and apparent grades fibers have been treated with various chemicals to modify their properties. The alkaline hydrolysis of polyester fibers has been studied for about 50 years and a great deal of research has to be carried out. Alkaline hydrolysis is affected by yarn geometry (Teli and Purkayastha [6], Dave et al [7], Goraffa [8], Halse et al [9, 10], heat setting temperature, Collins et al [1], (ICI [11]), Teli and Purkayastha [12], Hashimoto [13], filament cross section Houser [14], Collins et al [1], Solbrig and Obendorf [15], additives in the fibers (Sanders and Zeronian [16]. Collins et al [1]. Goraffa and [8].

Ellison et al [17]), and presence of co-

potential of alkaline hydrolysis in studying

monomers in the Latta [18], Guang et al [19] and McIntyre [20]. The effect of alkaline hydrolysis treatment on the degradation of polyester geotextiles has been investigated by Rahman [21]. Gorchakova et al [22] have studied the effect of structural modification of polyester fiber brought out by alkaline hydrolysis on the strength of non woven materials. East and Rahman investigated the effect of applied stress on the alkaline hydrolysis of geotextiles poly (ethylene terephthalate). Recently Rahman and East [24] studied titanium dioxide particles induced alkaline degradation of PET application in medical textiles. Sato [25], Kim et al [26] and Zeronian et al [27] and Jan et al [28] carried out work on alkaline hydrolysis and enzymatic treatments of different type of polyester fibers.

Thus most of the studies reported above are concerned with the application of alkali treated polyester fibers and fabrics to geotextile and medical textiles. There is, however a lack of information on the investigation of structure of polyester fibers using alkaline hydrolysis as a tool in various situations, and this paper reports the findings. It may be noted that for investigating the structure of fibers, expensive tools such as X-ray diffraction, microscopy, optical **FTIR** (Fourier Transform Infrared Spectroscopy) scanning electron microscope are used.

2. Materials and methods

The starting materials were polyester tow undrawn and drawn of 2.0 denier and 1. 2 denier x 51 mm bright polyester staple fibers. Polyester staple fibers of 1.2 denier x 51 mm were subjected to heat setting in an oven at temperatures ranging from 100 to 150 °C in steps of 10°C; the setting time was 2 minutes. In the case of hydrolytic treatment, the polyester staple fibers of 1.2 denier x 51 mm were treated with distilled water in a beaker for 30 minutes at temperatures 75°C, 80°C, 85°C, 90C°, 95°C, and 100°C respectively. The samples were dried and used for investigation. In the case of light fastness, polyester staple fibers of

1.2 denier x 51mm were subjected to light fastness studies by keeping them in direct sunlight for 1,3,5,7,9,11,13 and 15 days.

The polyester fibers were immersed in fresh isopropylene alcohol at room temperature for about 15 minutes, and then washed with soap solution at 80°C, for 20 minutes to remove the spin finish present in the fibers. After the final hot and cold wash with water. the specimens were dried and conditioned for determination of weight loss in all the cases of polyester substrates. Alkaline hydrolysis was carried out in sealed flasks or beakers at 100°C & 130°C (1:40 bath ratio, hold time 45 minutes) as the case may be with mild agitation on a laboratory model High Temperature High Pressure (HTHP) dyeing machine. The concentration of sodium hydroxide used was 10%. To terminate the hydrolysis, samples were rinsed in de-ionized water to eliminate excess alkali and neutralized for 2 minutes in 0.1% hydrochloric acid (HCl) for 10 minutes. The specimens were then washed in de-ionized water until the rinsed water was neutral to litmus paper. The products were then dried at room temperature for 48 hours.

Weight loss % was calculated by the formula

$$X_1-X_2$$
 X_1
 X_1

Where, X_1 is initial weight, X_2 is final weight. The mean of 10 readings was considered.

Measurements of density of substrates were made at 25° C (\pm 2° C) in a density gradient column prepared with Carbon tetrachloride and Xylene.

3.0 Results and discussions

3.1 Influence of hydrolytic treatment on weight loss of drawn and undrawn polyester

Т

Α

Т

Table 1 shows the weight loss of undrawn and drawn polyester tow. The drawn fiber shows a lower value of weight loss compared to the undrawn fiber; this is in agreement with earlier findings [4]. This is due to the differences in crystallinity and orientation. It is evident that the rate of weight loss has been determined by the fine structure of the fibers. Since undrawn was the least oriented and crystalline fiber, it was expected that, this sample would hydrolyze

most rapidly. Drawing operation slowed hydrolysis considerably due to large increase in the order and orientation of the polymer chains; even though the drawn samples were much finer than the undrawn sample. Similar effects have been observed in the case of conventionally spun and drawn PET fibers before and after setting [1]. It is also apparent that the variability of drawn fibers is higher in comparison with undrawn fiber.

Table 1 - Weight loss of undrawn and drawn polyester tow

Sl. No	Type of polyester	Weight Loss (%)	Standard deviation (%)	CV%	Density (g/cc)
1	Undrawn	2.3	0.100	4.35	1.343
2	Drawn (Draw ratio 2.44)	0.78	0.0577	7.39	1.401

3.2 Influence of heat setting on weight loss of polyester fibers

The influence of heat setting on the weight loss is shown in Table 2. It is noticed that the weight loss shows a progressive reduction with an increase in temperature (Fig 1); this is in conformity with the reported work. [32] This is due to an increase in crystallinity of the fibers following heat setting. It is a well-known fact that upon heat setting, the crystallinity of the polyester fibers increases rapidly and

levels off thereafter. It is interesting to note that weight loss follows a similar pattern as that of crystallinity. With finer fibers, the available surface per unit weight would be greater and therefore the hydrolysis would be expected to proceed faster with such fibers.

A statistical test conducted showed that the mean values of weight loss were highly significant. It is also interesting to note that the variability of weight loss for treated fiber is lower than that of control.

Table 2 – Influence of heat setting on the weight loss of polyester fibers

Sl. No	Heat setting	Weight Loss (%)	Standard deviation (%)	CV%	Density (g/cc)
1.	Untreated control	0.9	0.1732	19.2	1.343
2.	100°C	5.33	0.1732	3.24	1.348
3.	110°C	4.93	0.1154	2.34	1.358
4.	120°C	3.93	0.0577	1.46	1.371
5.	130°C	2.93	0.1154	3.93	1.397
6.	140°C	2.46	0.0577	2.34	1.399
7.	150°C	1.90	0.1154	6.07	1.401

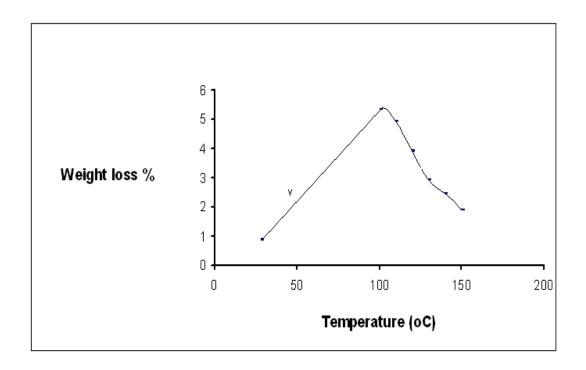


Figure 1 – Influence of heat setting on weight loss of PET fibers

3.3 Effect of hydrolytic treatment on weight loss

Table 3 shows the weight loss of polyester fibers following hydrolytic treatment at different temperatures. It will be seen that as the temperature of water increases, the weight loss increases progressively. Heat has a significant effect on polyester properties (Fig.2). The temperature scale may be divided into three significant points; these are room temperature, second-order transition temperature ranges and the crystalline melting temperature of the fiber in question. Morton and Hearle [31] explained the difference between first-order and second -order transitions. Melting is an example of first-order transition; that is, a change of state (from solid to liquid) accompanied by a latent heat and no change in volume. Discontinuities in specific heat and in the rate of change of volume with temperature are referred to by Morton and Hearle [31] as second-order transitions.

The technical significance of these secondtransition temperature is association with a change in mechanical properties. The larger increase in weight loss at temperature 80°C to 95°C may be attributed to exceeding to the glass transition temperature of the polymer. According to literature, the transition for polyethylene terephthalate occurs in the 75°C to 80°C Above this temperature, the range. amorphous region of the polymer becomes very mobile. For this reason, namely the relative volume increases, and the weight loss increases. The time of treatment has a significant effect on the amorphous region When the amorphous of the polyester. regions of the polymer become flexible and the molecular segment in this region become highly mobile, the water present may give the added effect of increasing molecular mobility. Due to this the weight loss shows an increase. It is also observed that the variability of weight loss for most of the treated fibers is low.

Table 3 - Effect of hydrolytic treatment on weight loss of polyester fibers

SL. NO	Hydrolytic Treatment	Weight loss (%)	Standard deviation (%)	CV%	Density (g/cc)
1.	Untreated control	0.9	0.05	5.55	1.343
2.	75°C	0.9	0.11	12.2	1.348
3.	80°C	1.9	0.11	5.78	1.358
4.	85°C	2.5	0.05	2.00	1.371
5.	90°C	2.9	0.05	1.72	1.397
6.	95°C	4.2	0.23	5.47	1.399
7.	100°C	4.3	0.06	1.39	1.401

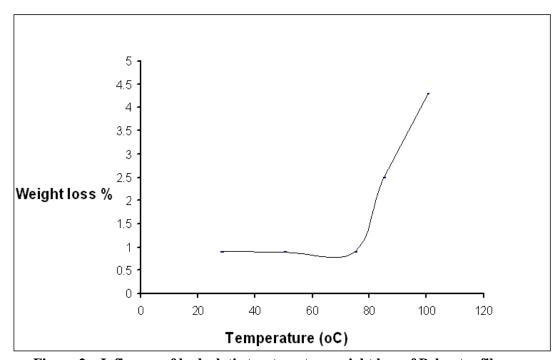


Figure 2 – Influence of hydrolytic treatment on weight loss of Polyester fiber

3.4 Effect of light fastness studies on weight loss

Values of weight loss of polyester fibers, which were subjected to light fastness studies, are set out in Table 4.

It will be seen that the weight loss percentage increases with an increase in exposure time. An increase in weight loss is a clear indication of polymer degradation (Fig.3). It is an established fact that the exposure of high polymers to the action of sunlight is known to result in a progressive

molecular chain scission at the exposed surfaces; this results in a reduction strength loss. Researchers have studied the actinic degradation of polyester on the basis of strength loss [29]. In this work, it is shown that weight loss can be taken as a measure of light fastness degradation. Rather than determining the strength, which is a time consuming process, the weight loss, which is cheaper and simpler, can be determined to detect the changes that occur in the fibers following light fastness degradation. Again the variation in weight loss is generally on the lower side for exposed fibers.

Table 4 - Effect of light fastness studies on weight loss of polyester staple fibers

SL. NO	Light Fastness Treatment (days)	Weight loss (%)	Standard deviation (%)	CV%	Density (g/cc)
1.	1	1.56	0.115	7.37	1.343
2.	3	2.93	0.115	3.92	1.342
3.	5	4.53	0.115	2.53	1.342
4.	7	6,76	0.057	0.84	1.340
5.	9	8.53	0.058	0.67	1.343
6.	11	10.36	0.057	0.55	1.344
7.	13	13.53	0.577	4.26	1.342
8.	15	13.96	0.058	0.415	1.343

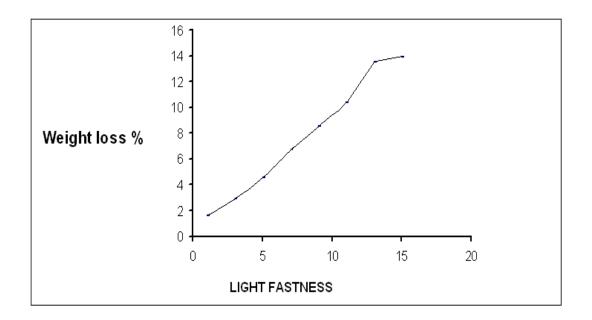


Figure 3 – Influence of light fastness studies on Weight Loss of polyester staple fibers

Т

M

4. Conclusions

The following conclusions been reached:

- 1. Weight loss of polyester drawn fiber following alkaline hydrolysis was significantly lower than that of undrawn fiber.
- 2. Weight loss of polyester fiber subjected to heat setting and then alkaline hydrolysis increased first at 100°c and thereafter fell.
- 3. Weight loss of polyester subjected to hydrolytic treatment showed a then an increase with increase in temperature.

4. As for as the effect of light fastness studies of polyester is concerned, the weight loss showed an increase up to 13 days of exposure following alkaline hydrolysis.

In view of the fact that weight loss of polyester fiber following alkaline hydrolysis has been followed to reveal change in this followed structure it is subjected that this may be used as a tool for investigating the structure.

5. References

- 1. M.J. Collins, S.H. Zeronian, and M. Semmelmeyer, "The use of aqueous alkaline hydrolysis to reveal the fine structure of poly (ethylene terephthalate) fibers", journal of applied polymer science, vol.42, 1991, pp 2144-2162
- 2. S.H. Zeronian and M.J. Collins, "Surface modification of polyester by alkaline Treatments' Text Progress, Textile Institute, Manchester, Vol. 20, 1989, pp1 24.
- 3. E. Waters, "The Dyeing of Terylene Polyester Fibres", Journal of Society of Dyers & colourists, Vol.66, No.12, 1950, pp 609 -615
- 4. S.A. Holmes and S.H Zeronian "The uses of sodium hydroxide hydrolysis to study the fine structure of undrawn and drawn high speed spun poly (ethylene terephthalate) fibers" J. Macromol, Sci, Phys Applchem., A 32 (9), 1994, pp 1147-1168.
- 5. J. Hayavadana, and V. Subramaniam. "Draw texturing effects" Textile Asia, Vol 33, 2001, pp 56-57.
- M.D. Teli and A. Purkayastha, "Effect of Substrate Geometry on Alkaline Hydrolysis" American Dyestuff Reporters, Vol 80, No 4, 1991 pp 78-80.
- 7. J. Dave, Rajkumar and H.C. Srivastava, "Studies on modification of Polyester fabrics by alkaline hydrolysis", Journal of Applied Polymer Science, Vol.33, 1987, pp 455-477.
- 8. A.A.M. Gorrafa, "Caustic treatment of polyester filament fabrics", Textile Chemistry and Colorist, Vol. 12, No.12, 1980, pp 33 37.
- 9. Y. Halse, R.M. Koerner and A.E. Lord, "Effect of high alkalinity levels on geotextiles II NaOH (alkaline hydroxide) solution", Geotextile and Geomembaranes, Vol.6, No.4, 1987, pp 295 305.

- Y. Halse, R.M. Koerner, A. Eiles and Lord "Effect of high levels of alkalinity of geotextiles I calcium hydroxide solutions", Geotextile and Geomembaranes, Vol. 5, No. 4, 1986 pp 261 – 282.
- 11. I.C.I. Industrial Fiber Manual, Section B2/1-3, 1966.
- 12. M.D. Teli and A. Purkayastha, "Effect of Fabric Geometry on Alkaline Hydrolysis of Polyester: Part II", American Dyestuff Reporter, Vol. 82, No. 2, 1993, pp 34 40.
- 13. T. Hashimoto, "Alkali Treatment of Tetron (Poly ethylene Terephthalate) fibers II Some observations on the mechanism of solubility of tetron in aqueous or alcoholic solution", Journal of Society of Textile Cellulose Industry, Japan, Vol. 15, No.10, 1959, pp 794 799.
- 14. K.D. Houser "Caustic Reduction of Polyester Fibers", Textile Chemistry and Colorist, Vol. 15, No.4, 1983. pp 37 -39.
- 15. C.M. Solbrig and S. Kay Obendorf, "Alkaline hydrolysis of titanium dioxide delustered poly (ethylene Terephthalate) Yarns", Textile Research Journal, Vol.61, No.3, 1991, pp 177-181.
- 16. E.M. Sanders and S.H. Zeronian, "An anlaysis of the moisture related properties of hydrolyzed polyester", Journal of Applied Polymer Science, Vol. 27, 1982, pp 4477 4491.
- 17. M.S. Ellison, L.D. Fisher and S.H. Zeronian "Physical properties of polyester fibers degraded by aminolysis and alkaline hydrolysis "Journal of Applied Polymer Science, Vol. 27, No.5, 1982, pp 247 251.
- 18. B.M. Latta, "Improved tactile and sorption properties of polyester fabrics through caustic treatment", Textile Research Journal, Vol. 54, No.11, 1984, pp 766 775.

Article Designation: Scholarly 7 JTATM

А

M

- 19. L.Guang, G. Lixia, and S. ong, J. China J.E. McIntvre. "Handbook of Fiber Technology, Science and Chemistry" Edited by M. Lewin, Marcel Dekker Inc, New York, Vol.4, 1985, pp 1 -71.
- 20. M. Rahman "The Alkaline Degradation of Polyester Geotextiles, University of Leeds, U.K. 1997.
- 21. M. Gorchakava, B.A. Lzamailov and V.A. Gartsueva, "Effect of structural modification of Polyester fibers on the strength of non woven materials ", Fiber Chemistry, Vol. 31, No.4, 1999, pp 315 -317.
- 22. G.C. East and Mashiur Rahman, "Effect of applied stress on the alkaline hydrolysis of geotextile poly (ethylene Terephthalate). Part 1: temperature." Polymer, Vol.40, No.9, 1999, pp 2281-2288.
- 23. Mashiur Rahman and G.C. East, "Effect of applied stress on the alkaline hydrolysis of poly (ethylene Terephthalate) at 40°c: relevance to medical textiles." Journal of Applied Polymer Science, Vol.102, No.5, 2006, pp 4814 - 4822.
- 24. M Sato, "Deterioration of filaments and films of polyethylene terephthalate with enzvme of Cladosporium Cladosporiodes FERM J-8", Sen-I Gakkaishi, Vol. 39, No 5, 1983, pp 209-219.
- 25. E.S. Kim, C.H. Lee and S.H. Kim "Effects of Pre Treatment Reagents on the Hydrolysis and Physical Properties

- Text Univ, Vol. 10, 1993 pp 34. of PET fabrics, Journal of Applied Polymer Science Vol. 112, No 5, 2009, pp 3071-3078.
- 26. S.H. Zeronian, M.K. Inglesby, Ning Pan, D. Lin, G. Sun ,B. Soni, K.W. Alger and J.D. Gibbon, "The fine structure of bi-component fibers. "Journal of Applied Polymer Science, Vol. 71, No. 7, 1999, pp 1163 – 1173.
- 27. S.S. Jan, J.L. Ven, G.A. Shyu, C.L. Wuu and C. Chen." Alkaline hydrolysis of Poly(ethylene Terephthalate) fabric in the presence of organic solvents ", American Dyestuff Reporters, Vol. 81, No. 10, 1992, pp 32-40.
- 28. Mashiur Rahman and Marolo Alfaro, "Degradation of Polyester Geotextiles in alkaline solutions under loading" 57th Canadian Geotechnical Conference. Québec, Canada, 2004.
- 29. R.W. Singleton, R.K. Kunkel and B.S. "Factors influencing Sprague. evaluation of actinic degradation of fibers", Textile Research Journal., Vol. 35, 1965, pp 228-237.
- 30. W.E. Morton and J.W.S. Hearle, The Physical Properties of Fibers. The Textile Institute, Manchester, U.K. 1994.
- 31. S.H. Niu and Wakida "Effect of Heat setting temperature on Alkali Hydrolysis of Poly(ethylene terephthalate) Fibers", Textile Research Journal, Vol. 63, 1993, pp 346-350.

Т

Т

M