DEGRADATION OF POLYESTER GEOTEXTILES IN ALKALINE SOLUTIONS UNDER APPLIED LOADING

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ABSTRACT

Alkaline degradation of polyster has been studied in various concentrations of sodium hydroxide, potassium hydroxide and lithium hydroxide at different temperature and load levels. The data obtained from sodium hydroxide treated polyester geotextiles were analyzed using an empirical formula developed by the first author. It was found that at a rate of hydrolysis of $2.8*10^{-4}\%h^{-1}$ in 0.1% NaOH at room temperature of 20°C, 2GT polyester fibres would survive 20 years without any applied load and in 0.01% NaOH concentration without any external loading conditions, the PET (2GT) geotextiles would survive approximately 200 years. The calculated lifetime using an empirical formula was 260 years in 0.01% NaOH when there was no applied load. The lifetime reduced drastically when the samples were under applied load. The sample lost almost 27% of their original breaking load within 120 days when the applied load was beyond the yield load. The calculated lifetime obtained from the empirical formula was compared with Arrhennius equation and ICI equation. The results were consistent with both the equations.

RÉSUMÉ

1. INTRODUCTION

Polyester (polyethelene terephthalate, PET both industrial and apparel grades) fibres have been extensively studied to understand their characteristics and properties as a function of processing and as a function of end use conditions. Extensive studies have been made on the effects of various chemicals on the final properties of polyester fibres. For geotextile applications, there is also the desire to understand the factors that may affect the durability of polyester when it is buried in soil. Hydrolysis is the primary means by which polyester fibres degrade.

The alkaline hydrolysis of PET has been studied for about 40 years and much more needs to be known before conclusions can be made with confidence. The factors upon which the rate of alkaline hydrolysis of PET varies depends on yarn geometry (Teli and Purkayastha, 1993; Dave et al. 1987; Gorrafa, 1980; Halse et al. 1990, 1987), heatsetting temperature (ICI, 1966; Teli and Purkayastha, 1993; Hasimoto, 1959; Collins et al. 1991), filament cross-section (Houser, 1983; Collins et al. 1991; Solbrig and Obendorf, 1991), additives in the fibres (Sanders and Zeronian, 1982; Collins et al. 1991; Gorrafa, 1980; Ellison et al. 1982) and presence of comonomers in the polyesters (Latta, 1993; Guang et al. 1993; McIntyre, 1985). However, when PET geotextile material is used for geosynthetic engineering applications, it would be expected to be under applied loading. Very little work has been done about the effect of load on polyesters and no work on the effect of load in alkaline hydrolysis.

2. DESCRIPTION OF TESTING MATERIALS

2.1 Yarns

Three different types of high tenacity geotextile grade poly(ethylene terephthalate) yarns were used and they were as follows:

(a) Trevira (HOECHST) high tenacity poly(ethylene terephthalate) coded as PET1, type-730, 1100dtex 200f.
(b) Tergal Tech (RHONE POULENC) poly(ethylene terephthalate) coded as PET2, type-156, 1100dtex 192f
(c) High tenacity PET from Far Eastern Textiles, coded as PET3, 1111dtex 192f.

2.2 Fabrics

Double jersey rib (1x1) knitted fabric from the above yarns was produced using V-bed universal knitting machine with needle gauge of 7 and stitch-cam setting of 12.

2.3 Chemicals

Various chemicals were used in the present investigation and the name, grades and suppliers of the chemicals are summarized in the Table 1.

Table 1. Chemicals used in the study.

Name of the chemical	Grade	Supplier
Sodium Carbonate	99.5%	Vickers
Sandosin NI	99.0%	Sandoz
Sodium hydroxide pellets	AnalaR	BDH
Sodium hydroxide solution	ACS (1M)	Aldrich
Potassium hydroxide pellets	AnalaR	BDH
Lithium hydroxide	99%	Vickers

2.4 Preparation of Materials

Raw grey yarn and fabric were prepared without any prior treatment. The same materials were also prepared but were scoured. The scouring of the fabric was carried out in a rotary wash wheel with 550 ml of scoured solution in the dyeing canister using a rotation speed of 40±2 revolutions per minute. It was carried out using the following recipes:

Scouring Technique (S1):
Na_2CO_3 : 2g/l
Sandosin: 2 g/l
Temperature: 60°C
Time: 30 minutes

Scouring Technique (S2):
Na_2CO_3 : 2g/l
Sandosin: 2 g/l
Temperature: 80°C
Time: 30 minutes

2.5 Preparation of Samples for Hydrolysis Under Load

In order to apply load on the yarn, a winding stainless steel tube of 16 cm long and 10 cm outer diameter was used (Figure 1). The tube contained 40 holes of 1.0 mm diameter in 4-lines and there were two 2.0 mm diameter holes at both ends of the tube which could take 6.BA screws. One end of the two-meter long of grey yarn was tied in one end of the tube. The other end of the yarn was subjected to loading through the attached dead weights. The yarn was then wounded along the length of the tube under load. Once the winding of the yarn reached the other end of the tube, the yarn was wrapped around a screw fitted in the 2 mm hole at the other end of the tube. This provided a locked-in load in the yarn prior to alkaline exposure.

2.6 Experimental Methods

All the hydrolysis experiments were carried out in a rotary dyeing machine (ROTADYER) in water and oil bath using plastic and stainless steel dyeing tubes. The samples were treated with 100 ml alkaline solution of different concentrations (1 to 10%) and under a range of temperatures for a predetermined period of time. After the completion of each treatment, the alkaline treated samples were washed with distilled water followed by acid treatment to neutralize the remaining alkali inside the sample. The sample was dried for 48 hours at room temperature and the percentage weight loss was measured using the following formula:

$$\text{Weight Loss} = \frac{W_1 - W_2}{W_1} \times 100\% \qquad [1]$$

where W_1 is the weight of the original sample and W_2 is the weight of the hydrolyzed sample. The mechanical properties of the individual filament were measured on an INSTRON-1026 Universal Tester. The rate of displacement was 50 mm/min and the maximum capacity of the load cell was 500g.

3. RESULTS AND DISCUSSIONS

3.1 Reactivity of Various Metal Hydroxide on Polyester Geotextiles

Table 2 showed the relative reactivity of aqueous NaOH, KOH and LiOH for different treatment time at 80°C. It can be seen from this table that the reactivity of different alkali hydroxide is quite different when treatment was carried out at 80°C. The relative reactivity was established and it was found that the relative rates of reactions for LiOH, NaOH and KOH are in the ratio of 2.2:1.4:1.0 (LiOH: NaOH:KOH). This behaviour of the relative reactivity was not consistent when treatment was carried out at elevated or lower temperature, different scoured samples or alcoholic solution of these alkali used. For example, it was found that at 100°C the ratio of relative reactivity of aqueous NaOH and KOH was 1.0 and at 20°C the value was 1.13. The scouring techniques were also affected the relative reactivity and reported elsewhere (Rahman, 1997).

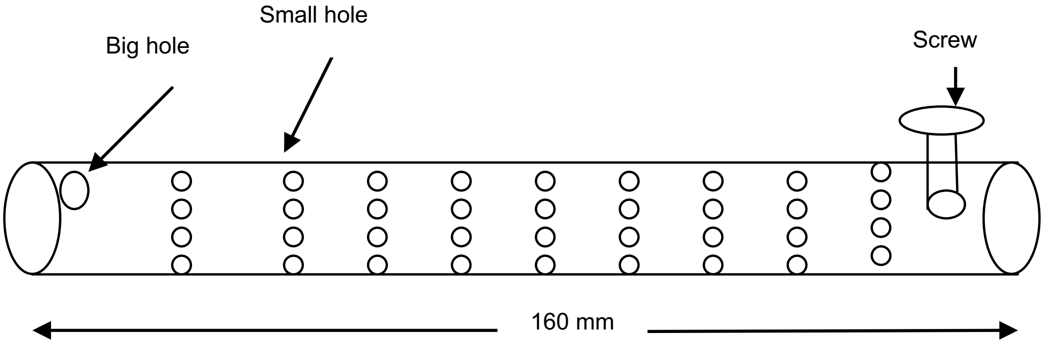

Figure 1. Schematic diagram of yarn treatment equipment.

Table 2. Weight loss (%) of aqueous NaOH, KOH and LiOH in relaxed condition at 80°C (scoured PET1, scouring technique-2).

Treatment time (hour)	Conc. Of NaOH (M)	Weight loss (%) in NaOH	Conc. Of KOH (M)*	Weight loss (%) in KOH	Conc. Of LiOH (M)	Weight loss (%) in LiOH
1	1.21	1.8 ± 0.47	1.20	1.28± 0.35	1.19	2.2 ± 0.17
2	1.21	3.5 ± 0.42	1.20	2.5± 0.06	1.19	5.1 ± 0.25
4	1.21	6.8 ± 1.2	1.20	4.9 ± 0.04	1.19	11.1 ± 0.48
6	1.21	10.3 ± 0.05	1.20	7.8 ± 0.04	1.19	16.2 ± 0.14
8	1.21	13.8 ± 0.04	1.20	9.8 ± 0.22	1.19	24.0 ± 1.49
Rate of hydrolysis (R_H)% h^{-1}		1.74 ± 0.03	1.20	1.2 ± 0.22	1.19	2.64 ± 0.29

* Adjusted from established data (Rahman 1997)

3.2 Alkaline Hydrolysis in Relaxed Conditions

Tables 3, 4, and 5 show the percentage weight loss of PET1. These results reflect the conditions for a range of temperatures and treatment times in aqueous sodium hydroxide solution. The weight loss increases with increasing treatment time and temperature. For a given temperature, the weight loss was linearly related with treatment time. These three tables also showed the predicted weight loss (W_P) calculated from the ICI formula (ICI, 1966) which is given below:

$$W_P = \frac{0.47\ t\left(x^2 + 6\,x\right)}{1.082^{100-T}\ \sqrt{d}} \qquad [2]$$

where W_P is the weight loss (%), t is treatment time (hour), x is concentration of NaOH (%), T is temperature (°C), and d is decitex of fibre (g/10^4 m). The agreement of actual weight loss (W_A) and the predicted weight loss (W_P) from the ICI formula was very good, which is quite surprising considering the fact that this formula was published 35 years ago and was meant for apparel polyesters. The weight loss of PET2 and PET3 also closely followed the ICI formula (Rahman, 1997).

The activation energy was calculated from Arrhenius plot based on the 8 hour period for weight loss given in Table 3. This gives a value of 14.7±1.1 Kcal/mol or 61.2±4.6 KJ/mol. The activation energy calculated from the ICI formula is 17.9 Kcal/mol over the temperature range of 50°C to 80°C. This value is consistent with those found in similar studies. Yamamoto et al. (1984) reported that the activation energy calculated for PET film hydrolysis was 12.8 Kcal/mol and for fibrous (non-woven) material the value was 15.5 Kcal/mol. For polyester fabric hydrolysis, the activation energy was 14.7 Kcal/mol as reported by Radillo et al. (1993).

3.3 Effect of Load on Alkaline Hydrolysis

The load on the yarn was produced by winding the yarn under different load levels. It was calculated from the breaking load of untreated PET and is also expressed as a percentage of the original breaking load of the yarn. The breaking load of the yarn was measured by TEXTECHNO STATIMAT with a gauge length of 100mm, load cell capacity of 100N and displacement rate of 50 mm/min.

Table 3. Weight loss (%) of the PET1 scoured fabric, 9.92% NaOH.

Treatment time (hour)	Weight loss (%) at 60°C			Weight loss (%) at 70°C			Weight loss (%) at 80°C		
	(W_A)	(W_P)	(W_P–W_A)	(W_A)	(W_P)	(W_P-W_A)	(W_A)	(W_P)	(W_P – W_A)
1	2.4	1.4	-1.0	3.8	3.0	-0.8	6.1	6.6	+0.5
2	4.2	2.7	-1.5	6.3	6.0	-0.3	11.6	13.2	+1.6
4	7.6	5.5	-2.1	11.5	12.0	+0.5	21.7	26.5	+4.8
6	11.8	8.2	-3.6	14.7	18.0	+3.3	33.0	39.7	+6.7
8	13.4	11.0	-2.4	23.5	24.0	+0.5	47.2	52.9	+5.7
Rate of hydrolysis (R_H)% h^{-1}	2.0±0.27			3.1 ±0.50			5.8± 0.30		

Table 4. Weight loss (%) of the PET1 scoured fabric, 3.99% NaOH.

Treatment time (hour)	Weight loss (%) at 60°C			Weight loss (%) at 80°C			Weight loss (%) at 100°C		
	(W_A)	(W_P)	$(W_P – W_A)$	(W_A)	(W_P)	$(W_P – W_A)$	(W_A)	(W_P)	$(W_P – W_A)$
1	0.3	0.4	+0.1	2.4	1.7	-0.7	8.4	7.4	-1.0
2	0.4	0.7	+0.3	3.4	3.3	-0.1	16.4	14.8	-1.6
4	1.0	1.4	+0.4	6.0	6.6	+0.6	31.9	29.6	-2.3
6	1.3	2.1	+0.8	8.0	9.9	+1.9	41.0	44.4	+3.4
8	2.2	2.7	+0.5	11.3	13.2	+1.9	57.0	59.2	+2.2
Rate of hydrolysis $(R_H)\% \ h^{-1}$	0.2± 0.04			0.40 ±0.40			7.7± 0.70		

Table 5. Weight loss (%) of PET1 scoured fabric at room temperature, 9.92% NaOH.

Treatment time (hour)	Weight loss (%) (W_A)	W_{P20}*	W_{P20} - W_A	W_{P23}*	W_{P23} – W_A
447	37.6 ± 1.7	27.9	-9.7	35.3	-2.3
792	53.0 ± 2.9	46.3	-6.7	58.7	5.7
Rate of hydrolysis $(R_H)\% \ h^{-1}$	$(7.3 \pm 1.0)*10^{-2}$				

* Calculated from ICI formula at 20°C and 23°C

Table 6. Weight loss, breaking load and tenacity data of PET1 grey yarn treated at room temperature, 120 days treatment for different stress level, 1.03% NaOH.

Treatment condition	Property Changes				
	Weight loss (%)	Breaking load (cN)	Breaking load loss (%)	Tenacity (cN/tex)	Tenacity loss (%)
Control filament	--	45.9 ± 2.8	--	83.5 ± 5.1	--
Relaxed condition	3.75	44.4 ± 1.8	3.3	84.0 ± 3.3	0.6
12.3%*	3.81	39.6 ± 1.9	13.8	74.8 ± 3.5	10.4
18.4%*	4.06	37.3 ± 2.5	18.7	70.8 ± 4.6	15.2
24.5%*	4.41	33.7 ± 2.7	26.6	64.2 ± 5.0	23.1

* Percentage of the breaking load

Table 7. Breaking strain, work of rupture and modulus data of PET1 grey yarn treated at room temperature, 120 days treatment for different stress level, 1.03% NaOH.

Treatment condition	Property Changes					
	Breaking strain (%)	Breaking strain loss (%)	Work of rupture N.cm	Work of rupture loss (%)	Modulus (cN/tex)	Modulus loss (%)
Control filament	19.1 ± 1.8	--	9.2 ± 1.5	--	741.4 ± 56.1	--
Relaxed condition	20.9 ± 0.8	+9.4	10.1 ± 0.8	+9.8	696.1 ± 30.0	6.1
12.3%*	18.0 ± 1.3	5.8	7.3 ± 0.9	20.7	648.7 ± 36.2	12.5
18.4%*	16.1 ± 1.2	15.1	6.2 ± 1.2	32.6	646.5 ± 57.7	12.9
24.5%*	15.8 ± 1.0	17.3	5.4 ± 0.9	41.3	628.0 ± 76.5	15.3

Table 8. Breaking load data of PET1 treated at room temperature for different stress levels, 10.09% NaOH.

Treatment condition	Treatment time (days)					
	10		20		30	
	Breaking load (cN)	Breaking load loss (%)	Breaking load (cN)	Breaking load loss (%)	Breaking load (cN)	Breaking load loss (%)
Control filament	48.6 ± 3.2	--	48.6 ± 3.2	--	48.6 ± 3.2	--
Relaxed condition	40.9 ± 2.4	16.4	35.9 ± 4.1	26.1	30.2 ± 2.5	37.9
1.2%*	38.2 ± 3.1	21.4	27.6 ± 3.4	43.3	27.4 ± 1.9	43.7
6.1%*	37.8 ± 4.2	22.1	28.3 ± 3.5	41.8	24.9 ± 4.1	48.7
12.3%*	33.8 ± 3.6	30.4	29.1 ± 3.1	40.1	21.7 ± 3.9	55.4
18.4%*	26.9 ± 6.7	44.6	--	--	--	--
24.5%*	16.7 ± 5.2	65.7	7.3 ± 2.7	85.0	*D	D*

* Percentage of the breaking load

3.3.1 Weight Loss Measurements

Tables 6 and 7 showed the weight loss of PET1 alongside mechanical properties treated with 1.03% sodium hydroxide at room temperature for 120 days for different load levels. The weight loss of the loaded fibres was slightly higher than the relaxed fibres. The weight loss for relaxed fibres was 3.75% whereas for the loaded fibres (highest load) the weight loss recorded was only 4.41%. The increased weight loss for the loaded fibres could be due to the decreased linear density of the fibres caused by elongation during loading.

3.3.2 Mechanical Properties

Tables 6 and 7 also showed the changes of mechanical properties treated with 1.03% sodium hydroxide and Table 8 showed the breaking load of yarns treated with 10% sodium hydroxide. The percentage loss of breaking load for the relaxed samples is similar to the percentage weight loss. The percentage loss in breaking load for the loaded samples is higher than that of the relaxed samples. At the highest load level (at 24.5% of the breaking load) the effect is so severe that the fibres failed to survive for 30 days at room temperature (see Table 8). Usually if there is no other factor involved, the breaking load of alkaline hydrolyzed polyester filaments should decrease proportionately to the weight loss of the fibres. Others have stated (ICI, 1966) that the percentage loss of breaking load can be one to two times the percentage weight loss. However, in the presence of solvents such as DMF, DMSO, DMA in the NaOH solution bath (Jan et al. 1992) and amine treated polyesters (Ellison et al. 1982), the breaking loads decreased more than proportionally to the weight loss. So, the implication was that the alkaline hydrolysis under load was no longer a surface reaction and that under loading conditions, internal attack was taking place. The hydroxide ions must diffuse inside the fibres, producing further chain scission which causes extra damage to the fibres. Surface cracking of the fibres occurred under loading condition in alkaline solution as reported by East and Rahman (1999). Tenacity, breaking strain, and work of rupture decrease as the load increases during alkaline hydrolysis (see Tables 6 and 7). The effect is even catastrophic for the highest load levels. In some cases a decrease in modulus was observed only for the highest loaded samples.

3.3.3. Statistical Analysis

The results of alkaline hydrolysis showed a small effect at lower load levels, followed by a much larger effect at high loads. In an attempt to clarify the data, empirical relationships were examined. Statistical analysis of the experimental results was carried out using SPSS Multiple Linear Regression Release 6.0 (Norusis, 1993). The independent variables of the treatment time (t), applied load (P_A), T^2, $(P_A)^2$, TP_A were incorporated in the model together with the breaking load as dependent variables. A stepwise method was employed to develop the optimum model. It was found that, for a given concentration of sodium hydroxide and temperature of treatment, the breaking load can be best determined by the following equation:

$$P_P = \beta_0 + \beta_1 + \beta_2 (P_A)^2 \qquad [3]$$

where P_P is the predicted breaking load (cN), t is treatment time (day), and P_A is the applied load (g).

The following equation was obtained for PET1 grey yarn when treated with 10.09% NaOH at room temperature at different stress levels for 10, 20 and 30 days (Table 8):

$$P_P = 45.9 - 0.63\,t - 6.26 \times 10^{-6} (P_A)^2 \qquad [4]$$

($R^2 = 0.96$, S = 2.63, 95% confidence limit = ± 5.73). R^2 is the "coefficient of determination", which has a value =1 when the correlation is perfect; S is the standard deviation about the regression and 95% confidence limits for the predicted breaking load P_P are given by $\pm t_{0.025}$ S, where $t_{0.025}$ is obtained from the probability points of the t distribution tables using n-2 (=12) degree of freedom.

Table 9. Breaking load (cN) data of PET1 treated at room temperature (from Equation 4).

Treatment condition (P_A)	Treatment time (day)								
	10			20			30		
	P_E	P_P	$P_E - P_P$	P_E	P_P	$P_E - P_P$	P_E	P_P	$P_E - P_P$
Relaxed condition ('0' load)	40.9	39.55	1.35	35.92	33.22	2.7	30.16	26.90	3.26
1.2%* (100g)	38.18	39.49	-1.31	27.56	22.16	-5.6	27.38	26.83	0.55
6.1%* (500g)	37.83	38.98	-0.15	28.27	31.65	-3.38	24.94	25.33	-0.39
12.3%* (1000g)	33.83	33.30	0.50	29.12	26.97	2.15	21.65	20.65	1.0
24.5%* (2000g)	16.67	14.53	2.14	7.29	8.20	-0.91	*	1.88	

* Percentage of breaking load

Using Equation [4] a straight line is obtained for percentage loss of breaking load in alkaline solution with treatment time, where the loss of breaking load is the square of the applied load. Accordingly, Table 9 contained the experimental breaking load (P_E) obtained from Table 8 with corresponding predicted breaking load (P_P) from Equation [4].

3.3.4 Lifetime Prediction

Given the rate of hydrolysis determined at room temperature for PET1 geotextiles in 10% NaOH, $7.3*10^{-2}\%h^{-1}$ (Table 5), it is possible to make some predictions as to the lifetime of PET in various alkaline media. Such a prediction implies a rate of hydrolysis of $2.8*10^{-4}\%h^{-1}$ at 0.1% NaOH, a more realistic alkaline concentration for geotextiles applications. If we assume that the time to lose 50% of the breaking load is the same as the 50% of the weight of the fibre, then the time for the material to lose 50% breaking load at 0.1% NaOH will be $50\% \div 2.8*10^{-4}\%h^{-1} \approx 20$ years and in 0.01% NaOH the lifetime would be ≈200 years for scoured (S1) PET1 fabric.

If, alternatively, we take the Empirical Equation [4] for PET1, $P_P = 45.9 - 0.63t - 6.26 * 10^{-6} (P_A)^2$, then we can also estimate the lifetime for 50% loss of breaking load for zero applied load. If applied load (P_A) is zero, then for $P_P = 22.9$ (cN), t = 36 days, this is in 10% NaOH; presuming the same reduction in rate as above, for 0.1% NaOH, t = 26 years for grey polyester geotextiles. In the literature, several opinions can be found about the lifetime of polyester geotextiles that varies from 16.5 years to 214 years (Horrocks and D'Souza, 1992). These different opinions may be due to the different types of polyesters and various alkali metal hydroxides used in the experiments.

4. CONCLUSIONS

If the condition of geotechnical sites are neutral, perhaps polyester would be the best among the competing geotextiles. Even at a high load levels, polyester may survive for its anticipated lifetime. However, if these materials are exposed to alkaline solutions, the combined action of loads and alkali exposure should be taken into account in estimating the appropriate mechanical properties for engineering design.

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