

## DEGRADATION STUDIES OF POLY(ETHYLENE TEREPHTHALATE) YARNS

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### ABSTRACT

Poly(ethylene terephthalate) multifilament yarn, textured yarn and staple were treated with potassium hydroxide. Time, temperature and concentration of base were selected as variables. Degradation of the products was investigated in terms of percentage loss in weight, viscosity measurements and percent elongation at break.

It was observed that textured yarn has greater loss in weight than either multifilament yarn or staple. However all products showed the same reduction in viscosity. As a result of treatment with base the mechanical strength of the textured yarn decreased with increasing base concentration and time.

### INTRODUCTION

Poly(ethylene terephthalate) (PET) is a well known synthetic polymer which was introduced in the electrical industry as insulating film or sheet and in the textile industry as fiber.

Various authors have stated that PET is stable to action of chemical reagents such as alkalies, salts and acids at low temperature. Fiber has no apparent loss in strength upon several hours exposure to dilute solutions of hydrochloric, acid sulfuric acid and nitric acids and sodium hydroxide at room temperature (Korshak et al. 1965). There are also several papers on the mechanism of hydrolytic, thermal degradation and the effects of chemical degradation on physical properties such as tensile strength, elongation at break (Mahon et al., 1959, Phol et al., 1951, Marshal et al., 1953, Carlsson et al. (1982).

Since various organic and inorganic bases were used in dyeing, after treatment and washing steps of fabric preparation the effect of bases on the properties of fibers must be investigated.

A comparative study of the reactivity of hydroxide and various alkoxide anions was made by C. G. G. Namboori and et. all previously (Namboori et al., 1968). It was found that regardless of the basicity of the anions the order of reactivity follows the nucleophilicity of the bases and steric retardation during the equilibrium process.

As a part of broader study on the degradation of polyethylene terephthalate, we have studied the effect of time, temperature, and base concentration on the properties of multifilament yarn, textured yarn and staple in the present paper.

## EXPERIMENTAL

### Materials:

Multifilament yarn, textured yarn and staple produced by a firm called SASA, were used in all experiments.

Potassium hydroxide (Merck), m-cresol (SIGMA) were reagent grade materials.

### Characterization of PET samples:

Intrinsic viscosity of samples were determined using Ubbelohde viscometer.

Tensile properties were measured by an Instron Tester at room temperature.

### Hydrolysis of PET samples:

PET samples 2.0 g were weighed accurately. 250 mL of potassium hydroxide solution was used for each experiment. Treatments were carried out in 250 cc round-bottomed flask fitted with a reflux condenser, and stirrer. The temperature was maintained by immersing the flask in a constant temperature bath. Specimens with various degree of degradation were obtained by changing the time of treatment concentration of potassium hydroxide solution and temperature. After the treatments, the samples were washed with an excess of water and 10–15 cc of 1% hydrochloric acid solution followed by an excess of water until the filtrate was neutral. After the suction extraction of water, the samples were dried under vacuum at 60°C overnight and reweighed to calculate percentage loss in weight. Viscosity measurements were done by dissolving the samples in m-cresol at 30°C.

## RESULTS and DISCUSSION

A series of experiments were carried out with different PET samples at a constant concentration of potassium hydroxide (%10) with treatment times ranging 15–180 min at 50°C. Plots of percentage loss in weight of samples vs time were shown in fig. 1. It should be noticed from the figure that degradation of PET samples is proportional to the time of treatment.

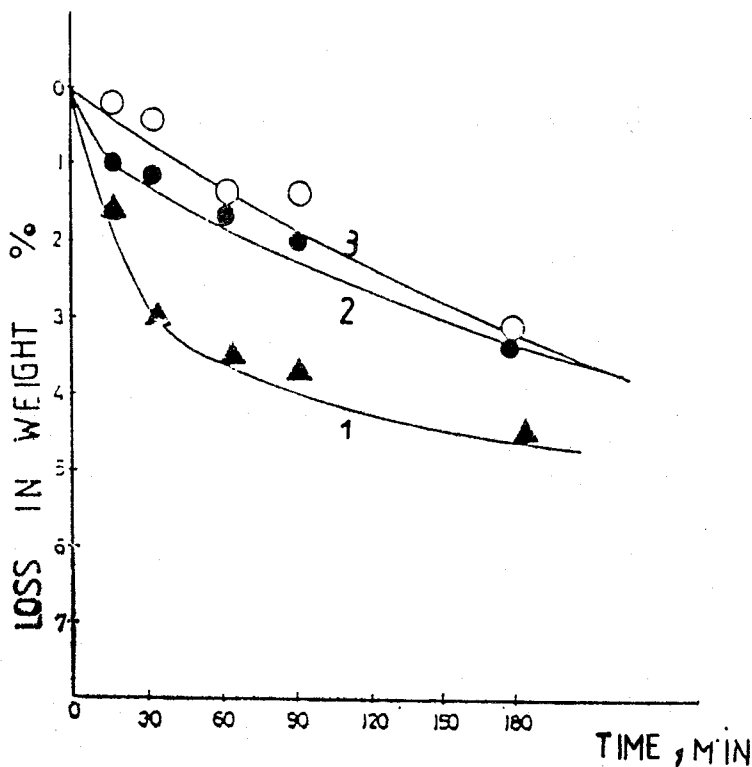
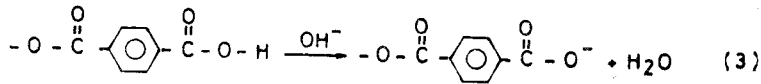
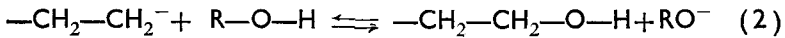
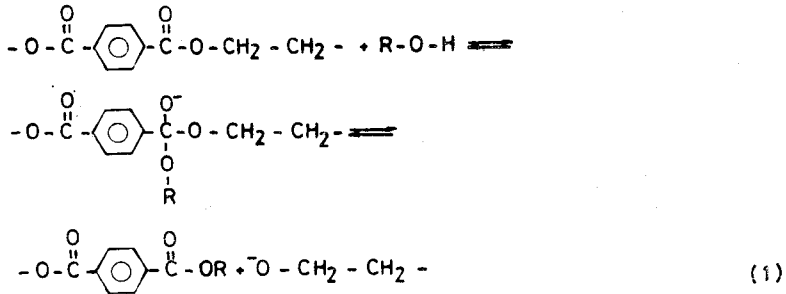


Figure 1. Loss in weight of poly (ethylene terephthalate) with time

- 1- textured yarn
- 2- multifilament yarn
- 3- staple.

Although a complex polyester PET is susceptible to basic hydrolysis similar to simple esters, the base attacks the electron deficient carbonyl, forming an intermediate anion with the choice of reversing the reaction or forming the product (Nambori et al, 1968).



According to eq. 1 the overall hydrolysis depends on the first equilibrium reaction and the base regenerated in solution as in eq.2. In saponification the overall reaction is irreversible since once the acid is formed it is immediately converted to carboxylate ion eq.3 which is not further attacked by base. Hence the reaction goes to completion in the direction of hydrolysis.

The attack of base removes the molecules present on the surface of the fiber as short chains, these are further hydrolyzed to the salt of terephthalic acid or to its esters. As the treatment time increases number of short chains removed increases, so loss in weight of samples increases. Although similar losses in weight were observed for staple and yarn, textured yarn showed the highest reduction in weight. This can be attributed to the formation of oligomers during the texturing processes.

The effect of treatment time on the intrinsic viscosity of the samples were given in table I. As it is seen from the table reduction in viscosity is not large and almost the same trend is observed for all of the products. This result also supports the assumption that base attacks the groups on the surface of fiber.

Table I. Change in intrinsic viscosity of PET samples with time  
Intrinsic Viscosity  $[\eta]$ .

Time (min)	Yarn	Staple	Textured Yarn
15	0.67	0.67	0.66
60	0.65	0.64	0.65
90	0.62	0.58	0.57
180	0.576	0.54	0.55

The next step in the investigation was to determine the effect of base concentration. Multifilament yarn and textured yarn samples were treated with different concentrations of potassium hydroxide (10% - 40%) at 50°C for 1 h. Change in percentage loss in weight and intrinsic viscosity were shown in fig. 2 and table II. Increases in potassium hydroxide concentration increased the loss in weight, caused reduction in viscosity.

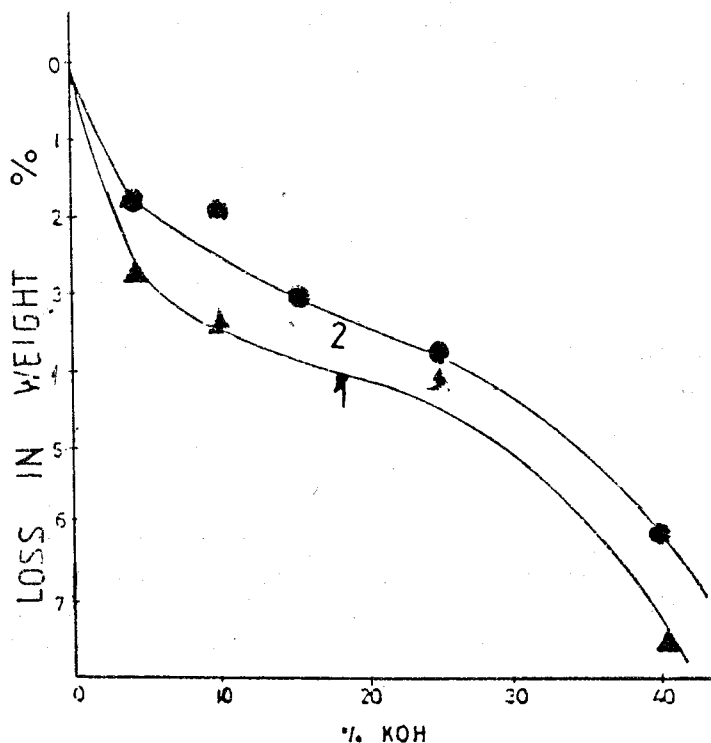


Figure 2. Loss in weight of poly (ethylene terephthalate) with potassium hydroxide  
1- textured yarn  
2- multifilament yarn.

Table II. Change in intrinsic viscosity with potassium hydroxide concentration.

*Intrinsic Viscosity* [ $\eta$ ]

KOH %	Multifilament Yarn
3	0.67
10	0.65
15	0.60
25	0.57

In the present work the effect of temperature was investigated by treating the samples with 10 % KOH for 1h at temperatures ranging 30°C–80°C. The results were tabulated in table III. The percent weight loss was found to depend considerably on the temperature.

Table III. Effect of temperature on the degradation of PET.

Temperature (°C)	Weight loss %
30	1.46
50	1.56
80	4.48

Changes in percent elongation at break with potassium hydroxide concentration and time were shown in fig. 3.4. Since textured yarn is more affected on treatment with hydroxide, mechanical tests were done using this yarn. Decreased elongation at break has been observed on treatment.

## CONCLUSION

It was found that the loss in weight of poly (ethylene terephthalate) fibers on treatment with potassium hydroxide increases with base concentration, time and temperature. The loss in weight and reduction in viscosity were in order of textured yarn > multifilament yarn > staple.

## ACKNOWLEDGEMENTS

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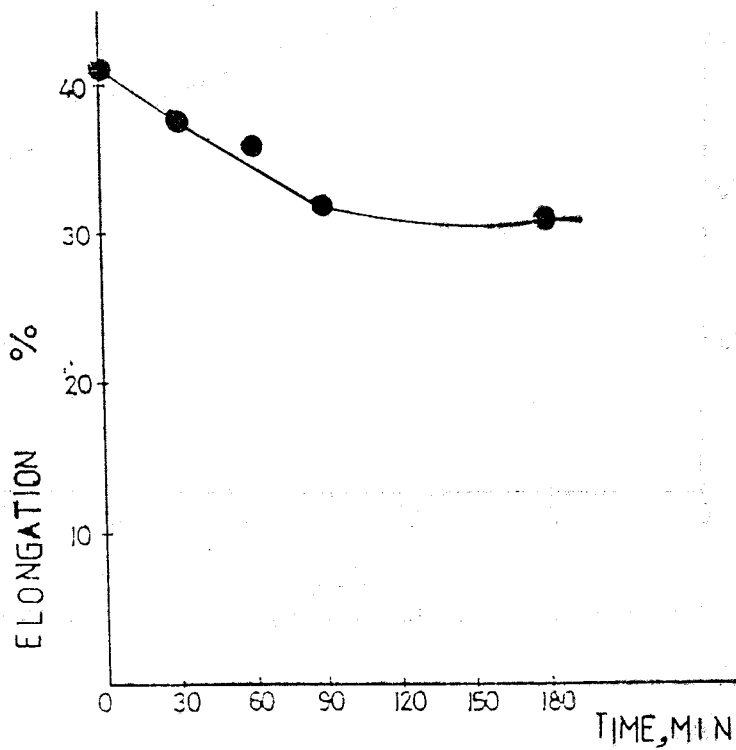


Figure 3. Effect of the extend of reaction time on percent elongation of textured yarn.

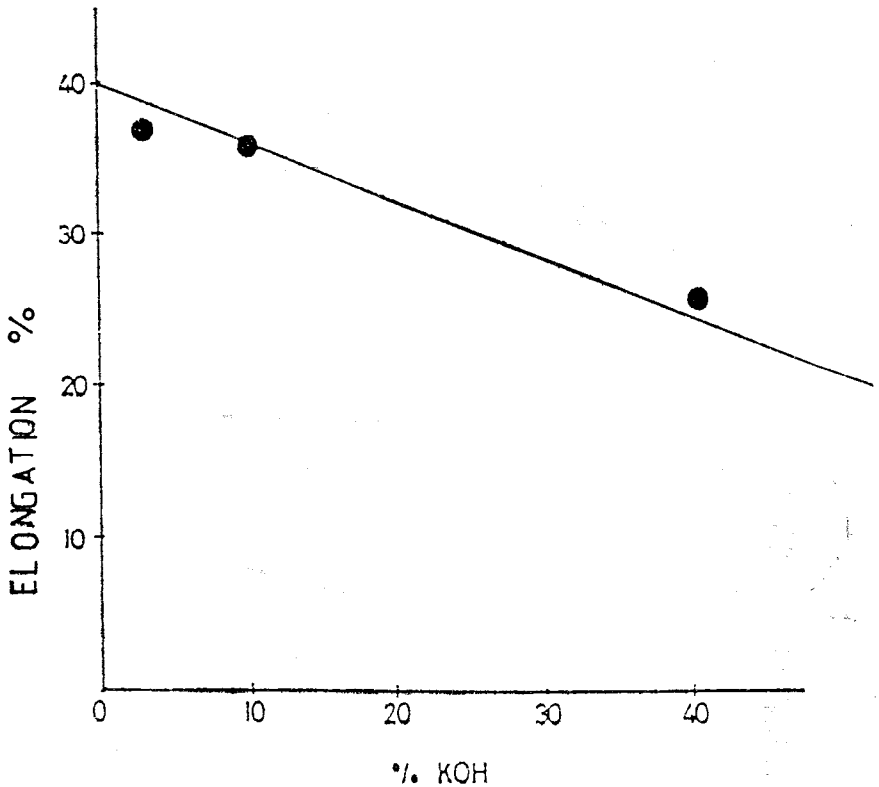


Figure 4. Dependence of percent elongation of textured yarn on potassium hydroxide concentration.

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