Effect of glycerine & alkaline hydrolysis on polyester fabric properties

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Abstract

Surface modification of polyester is well known to a chemical processing man by weight reduction process. The treatment will impart comfort characteristics to polyester and also the treatment has proved as replacement for silk.

This study deals with pre-treatment of polyester with swelling agent followed by alkaline hydrolysis. Glycerine was selected as pre-treatment solvent with 15% and 25% concentration treated at 60°c. Pre-treated materials were base hydrolysed at 130°c. With 5% and 8% NaOH. The results have been discusse4d with respect to effect of pre-treatment on weight loss of PET during alkaline hydrolysis. A general trend is observed with respect to higher concentration and temperature of pre-treatment followed by higher caustic concentration and temperature in favour of the physical properties of the weight loss increased with higher concentration pre-treatment and higher substrate hydrolysis temperature with glycerine.

Warp and weft bending length has registered a decreasing trend in the glycerine pre-treated samples at 60°c and alkaline hydrolysis at 130°c.

Sharp rise in GSM following pre-treatment and hydrolysis (Glycerine pre-treated samples) have indicated the action of glycerine on PET.

Crease recovery in warp and weft has registered the increasing trend with concentration and temperature of pre-treatment and hydrolysis at higher levels.

Fall in drape coefficient values following pre-treatment and base hydrolysis at higher levels of concentration and temperature have clearly indicated the softness imparted to fabric due to caustic action on PET.

The effect on pre-treatment and base hydrolysis conditions on compression behaviour is an interesting feature. The compressibility increased (EMC) with the increase in caustic concentration and temperature of hydrolysis.

1. Introduction

Polyester has had a phenomenal growth in the past decade as compared to other textile fibres. Morton Hearle and others published recently a commemorative review of 50 years research on polyester. Polyester has distinguishing features like melting point, high tensile strength, good elastic properties and possibility for grafting etc. However it suffers from features like good moisture absorption capacity, hydrophobic, static problem ETC., There are many ways of modifying the basic structure so as to achieve the desired objectives from apparel to aerospace. If mentioned, polyester is the only fibre enjoying its preference will be not an exaggeration.

It was in 1950 ICI disclosed the action of NaOH on polyester since then there has been many efforts to modify the basic structure.
Following are the approaches, which can be suitable for achieving desired goal:

i. Producing very fine fibres in the order of micro denier, converting into cloth and giving to woven fabric.
ii. Producing the woven cloth in normal deniers and finishing it in jet dyeing machine.

Among the two approaches it is the second approach widely employed as it uses a cheaper chemical and doesn't call for any specialized equipments.

Surface modification of polyester includes treatment with a base and the net result is that the substrate loses its weight. The process is referred as weight reduction technically and as scouring in industrial circles. The process imparts polyester a silky feeling and thus improves lustre of the fabric. Zeronian and Collins have brought a monogram on surface modification of polyester by alkaline treatments gave multi-directional thoughts for polyester process.

During the last 5 years an attempt has also been made to study the mechanical properties of filament and fabric by various researches (Vasanta, Hayavadana and McIntyre). Matsudira and Matasou have presented an extensive work on fabric low stress properties in a simple way.

The study included the effects of alkali on different polyester staple fibres varying in substrate and geometry (Mohansundaram and Hayavadana, and Gawish). The study emphases the effect of geometry on weight loss and on other mechanical properties. Although polyester surface modification has been known for the pat four decades, there exist certain unfilled gaps that need to be covered to address the current problem in polyester processing.

2. Literature Review

2.1 Introduction:

The consumption of polyester filament apparel section is increasing rapidly in India due to availability of indigenous high quality materials.

Polyester hardly needs introduction to a layman. It was in 1950 a research conducted by two English chemists viz John R.Whinfeild and James T.Dickson. Polyester was manufactured in different substrate forms but mainly from PTA route.

2.2 Production of silk like polyester:

Whenever the word research is heard japans never go unnoticed. Recently the toray company has come out with the production of silk like polyester. Following are the methods available for producing the silk like polyester.

- Fibre modification.
- Yarn modification.
- Fabric modification.

2.2.1 Fibre modification: Polyester can be modified by changing cross section, surface properties, structure and denier. As far as cross section is concerned fibres with trilobal, pentagonal, irregular shape can be produced by bicomponent fibre technique. However recently reliance industry have successfully produced dope dyed polyester.

By using certain chemical agent like sulpho-iso-pthalate, surface modification can be done. A new silky material has been developed by copolymerisation of polyester with benzoate. By producing finer fibres in the range 0.3-0.03 decitex fibres silky effect can be obtained.

2.2.2 Yarn Modification: Methods of modification of yarn with the aim of obtaining silk like effect are classified into following groups:

i. Combining polyester fibres of different characteristics.
ii. Blending of polyester with other fibres.
iii. Employing silk like twist.

2.2.3 Fabric Modification: In the fabric stage silk like hand can be obtained by employing anyone of the following three methods:

i. By changing the geometrical properties.
ii. By the use of softening agents.
iii. Weight reduction of polyester by NaOH treatment.

2.3 Review on weight reduction of polyester by alkaline hydrolysis:

Alkaline hydrolysis of polyester gives consideration from both industrial and research point of view. Following paragraph give an idea about weight reduction phenomena.

2.3.1 Hydrolysis : The ester hydrolysis is represented as follows:

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R-CO-O-R + H-O-R → R-C-OH + ROH
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Alkaline hydrolysis of an ester is also referred as saponification. Because it is the type of reaction used in the manufacture of soap the base catalyzed reaction goes to completion because the acid formed during hydrolysis reach irreversibility with the alkaline catalyst. At least one equipment of alkali is required from each equipment of ester, which is hydrolysed.

2.3.2 Nature of action of alkali on PET: In the saponification process NaOH with PET is initiated by the hydroxyl ion attack on the electron deficient carbonyl carbon atom of ester linkages. The result is that a compound namely di-sodium salt of terephthalate is formed. The chains are removed and hence the substrate looses its weight.

2.3.3 Influencing parameters: The reduction in weight during the hydrolysis by the rate of hydrolysis, which in turn is influenced by various parameters known as prime treatment conditions type and amount of catalyst, fibre type and heat treatments.

Among the various factor of prime treatment conditions it has been observed that temperature is one of the governing factors with a profound effect.

The rate of hydrolysis of polymer can be enhanced by adding certain quaternary ammonium compounds which acts as catalyst. According dorset among the various compounds CTMAB will give approximately 25% weight loss at 90°c for 30 min. It was also observed by Goraffa that the research reports have shown that among, the various alkalies tried, NaOH is found to be more powerful.

2.4 Effect of weight reduction on fabric property:

2.4.1 Nature degradation: Several studies have indicated that the action of caustic is limited to surface only as found from the density measurements .it was also found that if the material is heat set then the effect will be observed from core to surface .

2.4.2 Effect of moisture absorption: NaOH attack the surface and forms scars. Due to these pits moisture absorption improves. The studies have shown that moisture absorption from 0.4 to 1.5.

2.4.3 Effect on fabric properties drape coefficient: Weight loss improves the aesthetic properties of the fabric and drape coefficient decreases as weight loss increase.

2.4.4 Effect on crease recovery: Higher the weight loss higher is the crease recovery angle.

2.4.5 Effect on bending length: Caustic softens the surface and imparts a silky feeling to the fabric. In other words bending length decreases as fineness increase.

2.4.6 Effect on compressibility: Compressibility is affected by the fineness and in turn weight loss. Compressibility decreases following the load increase due to weight reduction phenomena.

3. Materials and Methods

This chapter deals with materials and methods selected for the study. The selection of process of process parameters and materials for the study was on the basis of ranges usually chosen in industry. The fabrics were of **Twill weave** produced on semiautomatic looms with necessary modifications.

4.1 Materials:

The details of the materials selected for present study is given in the table 4.1. The chemicals used for pretreatment and hydrolysis along with the parameters like concentration and temperature are given in the table. All the chemicals used were of laboratory grade.

4.2 Methods:

4.2.1 Removal of spin finish: In all the cases the spin finish was removed using 0.5% soap solution at 100 °c for 1 hr with 1:40 w/v MLR. The samples were washed in de-ionised water and dried and cooled in dessicator.

4.2.2 Pretreatment of PET fabric: In all the cases with 1:5 MLR the samples were pretreated with 15% and 25% concentrated glycerine. Pretreatment was carried out at

60°c. The treatment time was 1 hr. samples following pretreatment were washed and then dried.

4.2.3 Alkaline hydrolysis: Pre-treated samples were loaded into closed beakers of laboratory model HTHP beaker dyeing machine. Samples were hydrolysed at 130 °c with 5% and 8% **caustic soda** respectively. The materials following hydrolysis were washed thoroughly with water to remove the traces of NaOH.

Table 4.1: Geometrical properties

Status of materials	Threads per cm		Linear density (denier)		Crimp %	
	Wp	Wf	Wp	Wf	Wp	Wf
Control	106	60	36	34	5	4
Finished			36	34		

Table 4.2: Sample code and treatment details

Sample code	Pretreatment temp °C	Glycerine concentration%	Alkaline hydrolysis temp (°C)	NaOH concentration%
0	control	------	------	--------
1	60	15	T	--------
2	60	15	130	5
3	60	15	130	8
4	60	25	T	---------
5	60	25	130	5
6	60	25	130	8
T – Testing sample without alkaline hydrolysis				

The specimens were then washed with de-ionised water and dried and cooled in desiccators.

4.3 Testing:

The specimens were conditioned at standard atmosphere 65 ± 2% RH and 25 ± 2% as per IS: 6359-1971**.**

4.3.1 Determination of geometrical properties:

4.3.1.1 Yarn count: Yarn count was determined and an average of 10 measurements is reported in denier as per **IS 1315.**

4.3.1.2 Fabric weight per sq.mt: The weight per sq.mt of the fabric was determined as per IS: 1964-1970. An average of 5 measurements was expressed as weight per sq.mt.

4.3.1.3 Yarn crimp: The crimp of yarns (warp and weft) unravelled from the test fabric was measured on Shirley crimp tester as per **IS: 3442-1966**.Measurements were taken in both warp and weft ways. The average was given in % using formula.

$$\% \text{ crimp} = (L2 - L1)/L1 * 100 \quad \text{------------------}\rightarrow (4.1)$$

Where L1 = length of yarn in the fabric.

L2 = Stretched length of yarn.

4.3.1.4 Thread spacing: The parameter is defined as the average distance of the yarn between two consecutive threads and calculated mathematically as follows:

T = 1/epc or 1/ppc

Where epc = ends per cm.

Ppc = picks per cm.

4.3.1.5 Thickness: Thickness gauge was used to measure the compression .An average of at least 20 observations is reported. Compression was compared using the formula

EMC = (To-Tm)* 100/Tm -----------------→ (4.2)

To = Thickness at 0.5 gf/sq.cm.

Tm= Thickness at 2 gf/sq.cm.

4.3.2 Testing of mechanical properties:

4.3.2.1 Measurement of Bending Length: Shirley stiffness tester working on cantilever principle was used and the average of 5 replications is reported. The experiment was conducted as per IS: 6490-1971.

4.3.2.2. Determination of Drape Coefficient: Fabric drapability was measured on drape meter as per **IS: 8357-1977** and the average of at east 5 readings were reported. The drape coefficient was calculated using the formula:

Drape coefficient = (As-Ad)/ (AD-Ad) ----------------→(4.3)

Where AD= Area of the large disc.

Ad = Area of the smaller disc.

AS = Projected area of the draped fabric.

4.3.2.3 Measurement of crease recovery: Fabrics were tested for crease recovery tester on Shirley crease recovery tester in warp and weft directions as per **IS: 4681-1968.** The results are reported as an average of 5 observations.

5. Results and Discussions

5.1 Effect on weight loss:

Table5.1shows the weight loss % values of PET pre treated and hydrolysed fabric. With the increase in concentration of glycerine from 15%-25% the weight loss at 130° during hydrolysis is 5.05%. The results are in concomitant with several. This is mainly due to action loss % is also higher. This may be due to the basic fact that caustic action is severe at higher concentration .figure5.1 shows the effect of pre treatment conditions on alkaline hydrolysis.

5.2 Effect on geometrical properties:

It's clear from table 5.3 that the thread density has increased by 8.62 % for warp and for weft it is 10% following pre-treatment and base hydrolysis. The increase in thread spacing is noticeable with higher concentration and higher temperature in pre-treatment as well as base hydrolysis. The percentage increase in thread density is mainly due to the weight of the substrate

5.2.1 Effect on GSM:

It is clear from table5.2 that GSM shows a decreasing trend with the increase in solvent concentration and solvent pre-treatment temperature. Table shows a maximum of 8.7%change in GSM value at 130°c and 8% NaOH concentration along with 25% of solvent and 60°c pre-treatment temperature. Lower values of GSM are mainly attributed to attributed to higher weight loss% .it is obvious that as the pre-treatment temperature and caustic concentration. The chains of molecules on surface fibre are continuously depleted and hence the lower GSM value the trend is similar to the investigations as reported by many research workers.

Table 5.1: weight loss percentage:

Sample code	Weight of the samples in gms	Weight loss %
0	41.55	---------
1	41.5	0.12
2	40.01	3.7
3	39.45	5.05
4	41.47	0.19
5	40.51	2.5
6	40.15	3.36

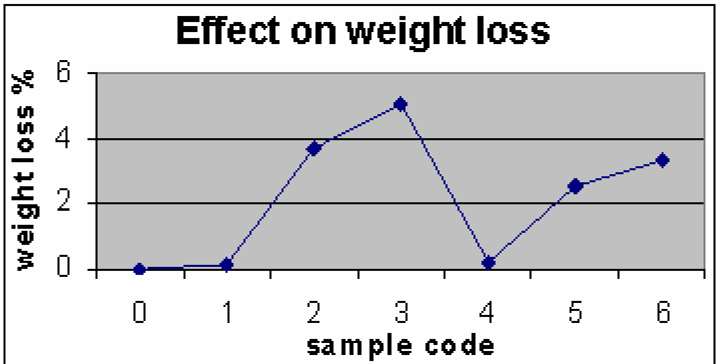

Figure 5.1

Table 5.3: Thread density:

Sample code	Thread density		Shift %	
	Wp	Wf	Wp	Wf
0	58	38	---	------
1	59	31	1.72	3.33
2	60	32	3.44	3.33
3	61	33	5.17	10
4	61	32	6.89	10
5	63	33	8.62	6.66
6	63	32	5.27	3.33

Table 5.2: GSM of treated fabrics:

Sample code	GSM	% shift
0	**114**	**------**
1	**110**	**3.5**
2	**112**	**1.75**
3	**109**	**4.38**
4	**113**	**0.877**
5	**108**	**5.2**
6	**104**	**8.7**

5.3 Effect on fabric stiffness:

It's clear from the table5.4 that the bending length values in warp and weft way have registered a decreasing trend as the concentration and temperature of pre-treatment and hydrolysis increase. Decrease in bending length have also indicated that the fabric has also been imparted a soft feel. The decrease in the bending length has also attributed to the weight loss. Figure5.3 shows the plot of bending with pretreament and hydrolysis conditions.

5.4 Effect on drape coefficient:

Table 5.5 shows steep decline in drape coefficient values of pre-treated fabrics has clearly established the change in the surface texture of the fabric. This is also supplemented by % increase in EMC values. Figure5.4 shows the plot of drape coefficient with process conditions.

5.5 Effect on crease recovery:

Table5.6 shows increase in crease recovery angles following base hydrolysis has indicated the significantly of solvent pre-treatment and base hydrolysis. The shift in the crease recovery values is mainly due to higher weight loss percentages of fabric. Figure 5.2shows the change in the crease recovery angle with process conditions

Table 5.4: Bending length of treated fabrics:

Sample code	Bending length		% shift	
	Wp	Wf	Wp	Wf
0	2.88	2.77	-----	------
1	2.52	2.48	12.4	10.2
2	2.41	2.36	16.2	14.6
3	2.40	2.31	16.6	16.4
4	2.53	2.46	12.3	11
5	2.46	2.38	14.7	17.5
6	2.32	2.32	19.3	15.9

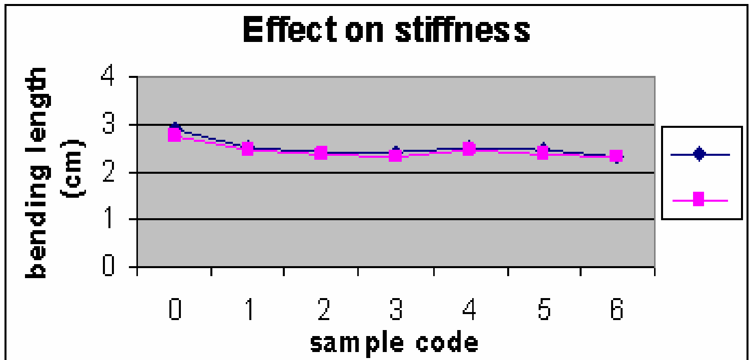

Figure 5.3

Table 5.5 Drape coefficient of treated fabrics:

Sample code	Drape coefficient	% shift
0	0.48	-------
1	0.44	8.9
2	0.38	20.2
3	0.33	30.8
4	0.44	8.11
5	0.42	12.6
6	0.32	33.3

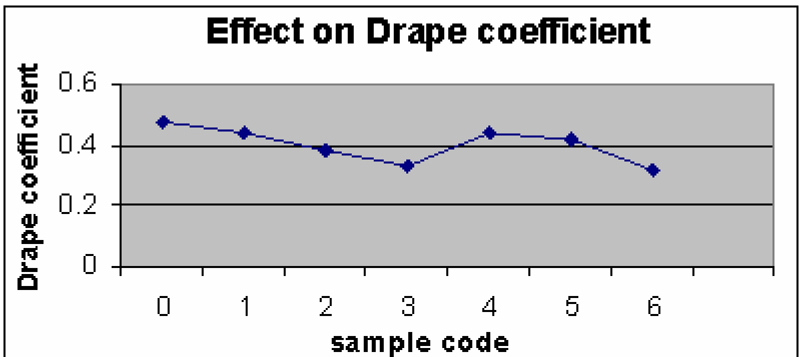

Figure 5.4

Table 5.6: Crease recovery of treated fabrics:

Sample code	Crease recovery		% shift	
	Wp	Wf	Wp	Wf
0	92.8	94	-----	-----
1	124	130.4	-42.2	-45.9
2	129.4	135.4	-39	-44.4
3	132	137.2	-33.6	-38.7
4	120.4	124.4	-34	-43.4
5	124.4	137	-29.7	-42.5
6	136.6	140.8	-47.1	-49.7

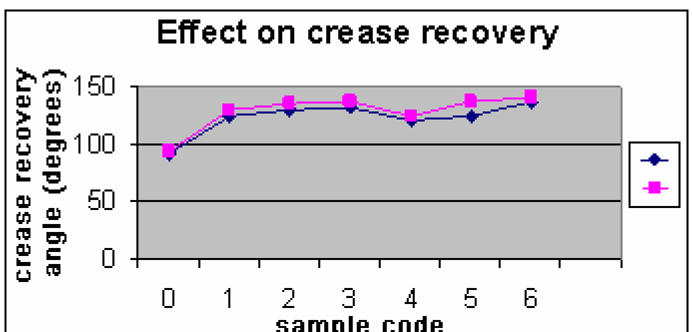

Figure 5.6

5.6 Effect on compressibility:

Figure5.5 shows the decreasing trend in thickness values as the compression load increase. It is obvious that following weight reduction the fabric has become soft and is thus able to be compressed. Increase in the EMC values have clearly indicated that the fabric can be used as a dress material. Higher values of EMC may be due to the fact that parameter like temperature has influenced the hydrolysis process significantly

Table 5.7: Thickness and compressibility of treated fabrics:

Sample code	Thickness (0.01 mm)	% shift
0	.525	-----
1	.552	36.6
2	.567	40.3
3	.564	39.6
4	.606	50
5	.551	36.3
6	.501	24

Sample code	Thickness (t 0)	Compression (Tm)	EMC (%)
0	0.525	0.489	7.36
1	0.552	0.448	23.21
2	0.567	0.470	20.63
3	0.564	0.430	31.16
4	0.606	0.453	33.77
5	0.551	0.446	23.54
6	0.501	0.416	20.40

6. Conclusions

Following are the conclusions drawn from the study conducted:

- The pre-treatment conditions have influenced the base hydrolysis i.e., increase in concentration and temperature of pre-treatment the higher the shift in the properties.
- Higher weight losses are observed at higher concentration and temperature of pre-treatment and base hydrolysis.
- Bending length of pre-treated fabrics samples has registered decreasing trend with increasing in concentration and temperature.
- Reduction in drape coefficient values following pre-treatment and hydrolysis have indicated the softness being imparted following pre-treatment and hydrolysis.
- Increases in crease recovery angles have confirmed the change in fabric texture.
- Higher values of compressibility with weight loss justified the action of caustic on PET.

7. Scope for future work:

Investigation on surface modification of 100% polyester or polyester contained union fabrics may be carried out on the following lines.

- Fabric in polyester(blend) fabrics with solvent like TCAMC, TCE/Phenol, formaldehyde can be considered with a view to study the pre-treatment effect on fabric low stress mechanical properties.
- Selection of PET warp and jute weft fabric and treatment with NaOH and KOH at different levels can be considered to find the response of PET and jute for caustic treatment on fabric handle.
- Effect of solvent pre-treatment on repeated hydrolysis can also be investigated.

- Effect of boiling water shrinkage on solvent pre-treated PET fabric can be investigated with a view to study fabric mechanical properties.
- The effect of primary treatment conditions on alkaline oxidation with copper sulphate padding and hydrogen peroxide bleaching can be studied on using 67/33 p/c blends. The effect of oxidation can be highlighted can be on fabric mechanical properties.

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