

Heat Setting of Poly(butylene terephthalate)

Manisha R. MATHUR, Sanjeev R. SHUKLA, and Prafulla B. SAWANT

Department of Chemical Technology, University of Bombay, Matunga, Bombay 400019, India

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ABSTRACT: Poly(butylene terephthalate) (PBT) yarn was subjected to heat setting at different temperatures for various time periods under taut conditions. Properties such as breaking load, critical dissolution time, dyeing with disperse dyes, etc. were evaluated and related to the changes taking place in the internal fiber structure. A temperature of about 140°C and a time period of about 60 seconds was found to be optimum for attaining the dimensional stability without affecting the useful textile properties of PBT filament yarn significantly.

KEY WORDS Poly(butylene terephthalate) / Heat Setting / Dyeing /

Poly(butylene terephthalate) (PBT), a linear condensation homopolymer resembles in most of its physico-chemical and mechanical properties to poly(ethylene terephthalate) (PET). PBT undergoes a unique reversible crystalline transformation at low levels of applied stress and, similar to PET, it also exhibits triclinic unit cell. The transition is related to a conformational change of the butylene chain from a relaxed *gauche-trans-gauche* to a stretched *all-trans* form.

Kinetics of heat treatment of PBT fibers and its microstructure during crystallization have been studied by Peszkin and Schultz¹ using X-ray and birefringence techniques. Lue and Spruiell² have studied the influence of processing conditions on structure development and the mechanical properties of PBT filaments. Kanno³ has reviewed the PBT fiber properties such as heat setting characteristics and printing performance. PBT fiber is also becoming popular as a "Carrierless Dyeable Polyester" with a number of advantages over normal carrier dyeable PET fiber. The main drawback of PBT, as against PET, is that it exhibits greater shrinkage. Since heat setting is essential to impart the dimensional stability, the present work was aimed at developing optimum heat setting conditions for PBT filament yarn in relation to its different useful properties such as dye uptake and strength.

EXPERIMENTAL

Yarn

PBT Filament yarn Finecell, with following specifications was used: BB 75 Denier/15 Filament, Type G 300 HP, supplied by Teijin Ltd., Japan.

Dyes

Foron Brilliant Orange ERLI (C.I. Disperse Orange 25) ($MW=323$) and Foron Navy Blue S-2GLI (C.I. Disperse Blue 79) ($MW=639.92$), both supplied by Sandoz (I) Ltd. Lyocol OI, dispersing agent was supplied by Sandoz (I) Ltd. All other chemicals were of "Laboratory Reagent" grade.

A) Heat Setting of PBT Yarn

PBT yarn samples were heat set under zero stretch (taut condition) for different time periods (30, 60, and 90

seconds) at various temperatures between 100°C and 200°C with 10°C intervals.

B) Determination of Critical Dissolution Time (CDT)

The CDT values of the heat set PBT samples were determined in 1:1::phenol:1,1-2,2 tetrachloroethane solvent system according to the method suggested by Galil.⁴

C) Determination of % Crystallinity and Crystallite Size

X-Ray diffraction studies were carried out using Philips PW 1729 X-ray generator coupled to PW 1710 Diffractometer Control and the plots were obtained on PM 8203 A on-line recorder. The sample powder was irradiated by Ni-filtered Cu- K_{α} radiation. The scanning range was 5—40° and the speed was 0.02 degrees per second. The % crystallinity and crystallite size were then calculated.⁵

D) Dyeing with Disperse Dyes

Commercial disperse dyes were used for dyeing of the PBT samples in a Beaker Dyeing Machine. The dyebath contained disperse dye, acetic acid (1 g l^{-1}), and Lyocol OI (0.5 g l^{-1}). The shade dyed was 1%, keeping liquor ratio 50. The temperature of the dye bath was raised to 100°C and the dyeing was continued for 1 hour in a conventional manner. The dyed samples were rinsed with cold water and subjected to reduction-clearing with sodium hydrosulfite (2 g l^{-1}) and sodium hydroxide (2 g l^{-1}) at 65°C for 15 minutes to remove surface dye. Finally, the dyed samples were soaped, washed and dried.

The amount of dye taken up was estimated by extracting the dyed samples in chlorobenzene at 100°C for 30 minutes and determining the absorbance values of the extracts on Pye Unicam SP 8-400 UV/Vis Spectrophotometer at appropriate λ_{max} .

RESULTS AND DISCUSSION

PBT filament yarn Finecell was heat set under taut condition and the effect of heat setting on various fiber properties was analyzed. Both time and temperature of heat setting were found to influence the strength of PBT yarns. The results plotted in Figure 1 indicate that with

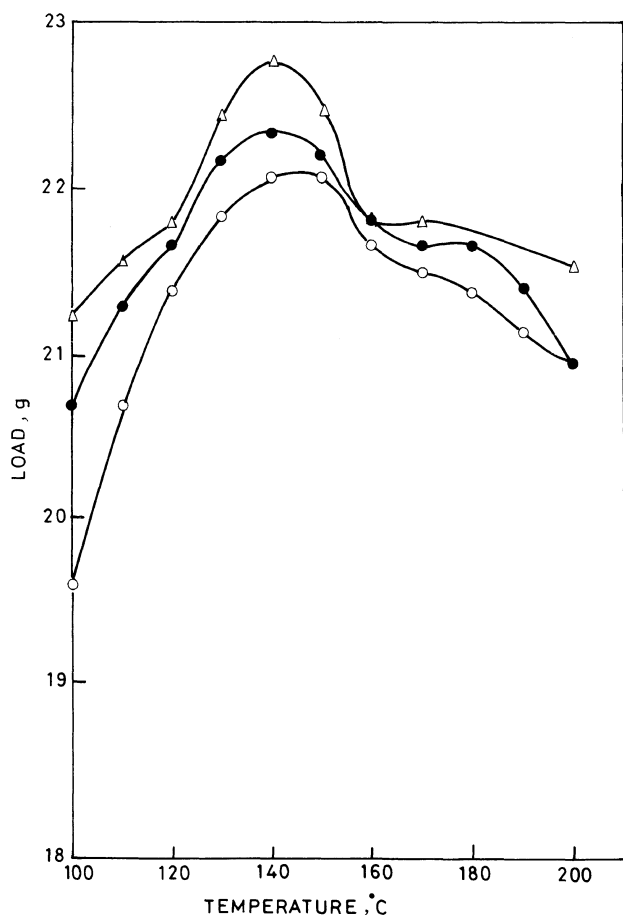


Figure 1. Plot of breaking load vs. heat setting temperature: —○—, 30 seconds; —●—, 60 seconds; —△—, 90 seconds.

rise in temperature of heat setting upto 140°C, the breaking load of PBT yarn increased gradually and further decreased upto 200°C at all the three heat setting time periods.

From the plots in Figure 2 it may be observed that upto 140–150°C temperature of heat setting, the CDT values increased only marginally and thereafter a very large increase in the CDT values with rise in temperature of heat treatment upto 200°C took place.

The data in Table I indicates that the heat setting temperature of 100°C decreased the % crystallinity of PBT insignificantly at any duration of treatment. At a treatment temperature of 150°C, the % crystallinity decreased considerably and at 200°C it registered a further substantial decrease.

As regards the crystallite size, as observed for the two different planes (010) and (100) of the triclinic crystal of PBT, the heat setting at 100°C slightly decreased it in (010) plane as compared to the original unheatset sample. In the (100) plane, however, the dimensions remained the same. The (010) plane lies in a direction parallel to the fiber axis in which the molecular chains, and hence the crystallites, are oriented. With further rise in temperature of heat setting upto 200°C, however, the crystallite size increased substantially in both the planes. Heat setting at 200°C increased the crystallite size in the (010) plane to similar levels at both the treatment periods of 30 and 90 seconds.

The heat setting treatment causes many changes in the internal structure of the fiber which are associated

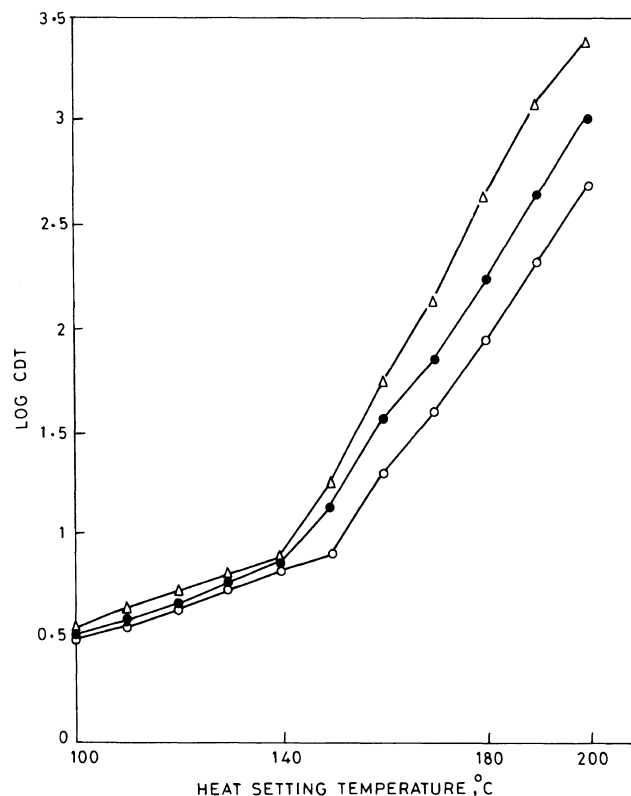


Figure 2. Plot of log critical dissolution time (CDT) vs. heat setting temperature: —○—, 30 seconds; —●—, 60 seconds; —△—, 90 seconds.

with mobilization, rearrangement and stabilization of the chain molecules in the new conformation which is more stable thermodynamically. By the process of heat setting, majority of the stresses present in the fine structure of the fiber are removed thereby imparting dimensional stability to it. The quantum and the nature of these changes are dependent on the time and temperature conditions of heat setting as well as on the history of fiber manufacture.

In the heat setting temperature range of 100–140°C, the transformation of small crystallites to the real big crystallites is extremely less and it is a temperature region which predominantly orients the existing crystallites along the fiber axis. It is well known that the breaking load increases with orientation of crystallites along the fiber axis. This phenomenon caused increase in the breaking load with rise in heat setting temperature upto 140°C. At higher heat setting temperatures above 140°C, the tendency of smaller crystallites to melt enhances rapidly thereby providing a very high degree of freedom to the chain molecules to allow them to come closer to each other and form bigger crystallites. Also, crystal perfection takes place at the boundary layers of the crystalline and amorphous phases. Due to this, the amorphous portion in the fiber structure virtually tends to increase thereby decreasing the breaking load.

The CDT values showed a marginal increase in the heat setting temperature range of upto 140°C. Such changes have been reported earlier in the case of PET fibers by Groeninckx *et al.*^{6,7} The linear relationship between log CDT and the crystallite size also showed a sudden break at a temperature of about 140°C (Figure

Table I. Effect of heat setting conditions on crystallinity and crystallite size of PBT yarn^a

Heat setting temp/°C	Crystallinity of sample heat set/%		Crystallite size of sample heat set/Å			
	30 s ^b	90 s ^c	30 s		90 s	
			D_{010}	D_{100}	D_{010}	D_{100}
100	77.65	76.80	50.44	28.09	53.80	29.12
150	70.48	69.78	100.87	62.66	114.80	80.95
200	57.52	52.12	161.50	108.67	161.40	116.46

^a Control PBT: crystallinity/% = 77.40; crystallite size/Å, D_{010} = 62.07; D_{100} = 27.15. D_{010} = crystallite size in the direction normal to (010) plane; D_{100} = crystallite size in the direction normal to (100) plane. ^{b, c} Heat setting time.

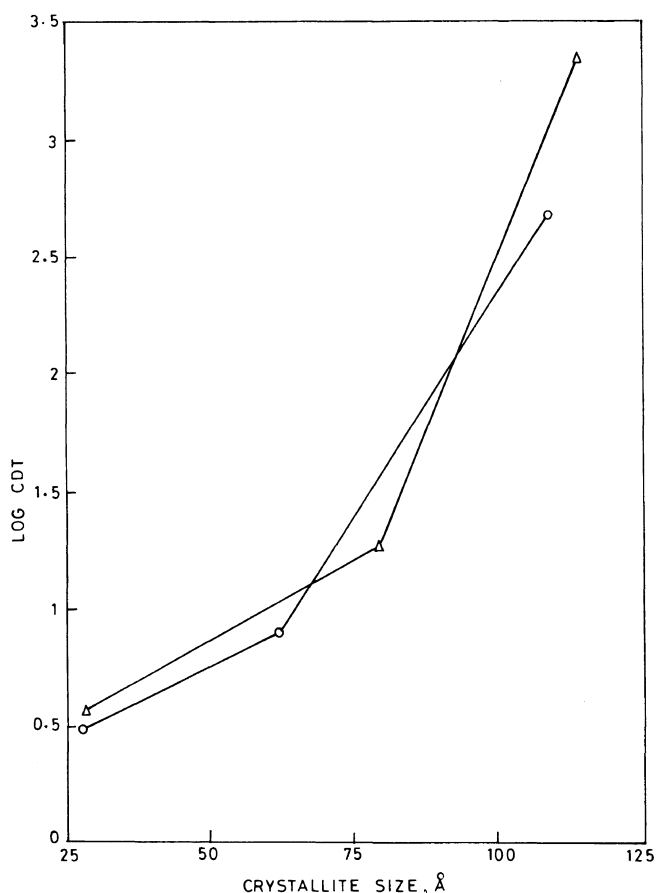


Figure 3. Plot of log CDT vs. crystallite size in the direction normal to (100) plane: —○— 30 seconds; —△— 90 seconds.

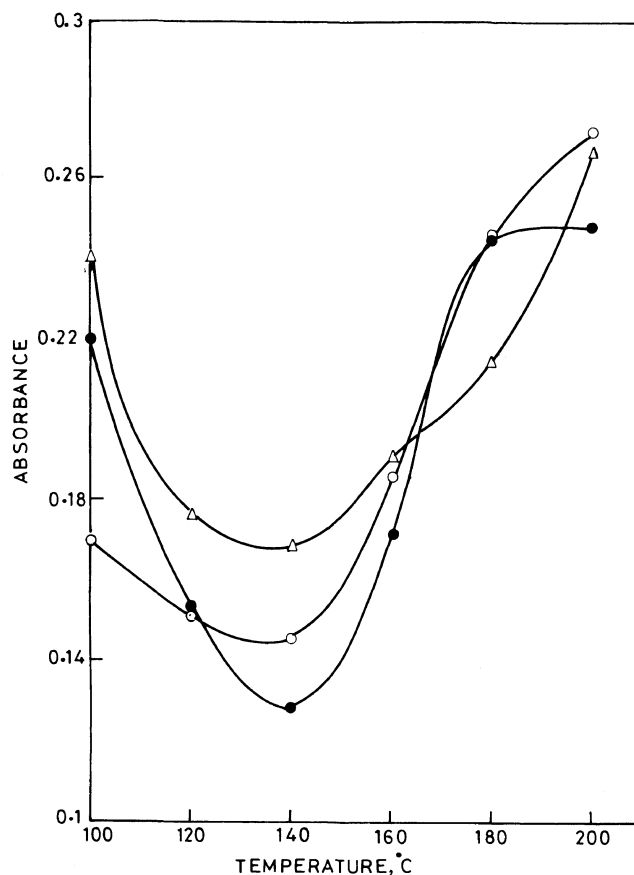


Figure 4. Plot of absorbance vs. heat setting temperature for C.I. Disperse Orange 25: —○—, 30 seconds; —●—, 60 seconds; —△—, 90 seconds.

3). Beckmann⁸ had an opinion that upto this point nucleation or the formation of tiny crystals takes place and that after this specific point the conditions are suitable for sudden growth in the crystal size. The larger crystals take more time to dissolve than the smaller ones.

The heat set PBT samples were subjected to dyeing with two disperse dyes having different molecular weights, using the conventional method of dyeing. The plots of absorbance shown in Figures 4 and 5, for the dyeings with C.I. Disperse Orange 25 (low molecular weight = 323) and C.I. Disperse Blue 79 (high molecular weight = 640), respectively, indicate that the dye uptake reached a minimum for a sample heat set at about 140°C and then increased with further rise in the heat setting temperature for both the dyes. The overall dye uptake values were considerably low for the high molecular weight dye due to the hindrance caused to the bulkier

dye molecules to penetrate the fiber structure.

In the initial range of heat setting, as shown in Table I, the % crystallinity has decreased and the crystallite size has increased with temperature. Generally, with a decrease in crystallinity, the dye uptake should show an increase as the accessibility of the fiber is supposed to increase. Figures 4 and 5, however, show a decrease in the dye uptake (as measured by absorbance) of treated samples. This may be attributed to the fact that the value of % crystallinity decreases due to reorganization of structure in which the smaller crystallites tend to orient themselves along the fiber axis and in the process of doing so increase the crystallite size. The dye is taken up by the fiber in the amorphous region as well as on the surface of the crystallites. With increase in the crystallite size, the surface area decreases and this factor predominantly decreases the dye uptake. Therefore, on

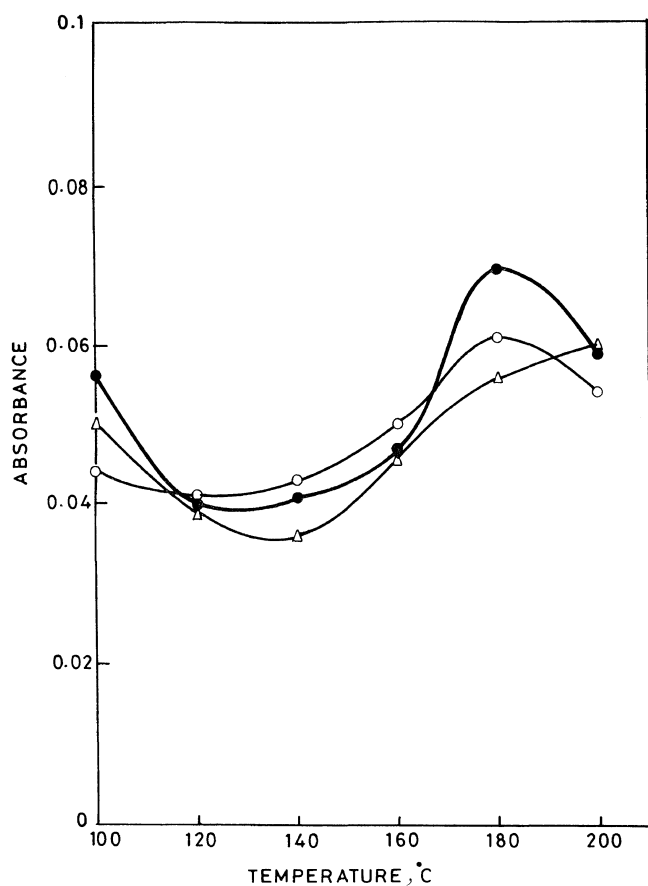


Figure 5. Plot of absorbance vs. heat setting temperature for C.I. Disperse Blue 79: —○—, 30 seconds; —●—, 60 seconds; —△— 90 seconds.

the whole, the effect appears to decrease the dye uptake of samples heat treated upto 140°C. Thus, the dyeing studies also support the contention that the changes taking place in the fiber structure with the changes in heat setting temperature are of different nature upto 140°C and thereafter.

Since the PBT fibers are dyed only at boil, in contrast to PET fibers which are dyed at a much higher temperature of 130°C, the heat set PBT yarns were subjected to treatment in boiling water for 1 minute under slack condition to observe their dimensional stability. These results are given in Table II. Thus, after the taut heat setting at 120°C for 60 seconds or more, no shrinkage took place indicating that the yarn attained the

Table II. Effect of heat setting conditions on shrinkage of PBT in boiling water

Heat setting temp/°C	Shrinkage of PBT heat set/%		
	30 s ^a	60 s ^b	90 s ^c
100	6.25	5.56	5.70
110	3.57	2.17	2.08
120	1.07	0.00	0.00
130	0.00	0.00	0.00
140	0.00	0.00	0.00

^{a, b, c} Heat setting time.

dimensional stability at and above the heat setting temperature of 120°C.

Heat setting should be carried out at a temperature at which the dimensional stability is fully achieved by a synthetic thermoplastic fiber. It may be observed that at 120°C and 140°C for 90 seconds treatment although the dye uptake values do not differ significantly, the rise in breaking load is significant. Also, after about 140°C, the fiber starts undergoing major changes in the fine structure as indicated by the crystallite size and CDT values.

Hence, taking into account all the parameters related to the changes in fine structure, it is suggested that the heat setting be carried out at about 140°C for a duration of about 60 seconds.

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