JOURNAL OF MATERIALS SCIENCE (2005)

Viscoelastic properties of poly(butylene terephthalate)/poly(ethylene naphthalate) blends

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Melt blends of poly(butylene terephalate) (PBT) and poly(ethylene naphthalate) (PEN) with 30 and 60 wt% PEN were prepared using a single screw extruder and an injection moulding machine. Stress relaxation tests for the specimens of PBT/PEN blends and the homopolymers were carried out using an Instron testing machine in an Instron environmental chamber. The Taguchi method of experimental design analysed how different levels of temperature, PEN content and initial stress affected the relaxation behaviour of PBT/PEN blends and homopolymers. From the response tables and analyses of main and interaction effects, it was shown that the most significant factor was temperature, followed by PEN content and then the initial stress. Consequently, high temperature, low PEN content and high initial stress speeded up stress relaxation rate of specimens. Interaction effects between factors were insignificant.

To fit the relaxation curves of the PBT/PEN blends and the homopolymers at different temperatures, PEN contents and the initial stresses, four different equations were attempted with Matlab$^{\text{TM}}$, which determined the coefficients of these functions using the experimental data of stress change with time. The simulated curves from the most suitable function among them were shown using the calculated coefficients to predict the relaxation behaviour of PBT/PEN blends (50% PEN) at temperatures of 30 and 60°C with an initial stress of 7 MPa. © *2005 Springer Science + Business Media, Inc.*

1. Introduction

The behaviour of a viscoelastic material lies between that of an elastic solid and that of a viscous fluid. In the case of a viscoelastic material subjected to a constant strain, the resulting stress decreases with time and this is known as relaxation as opposed to creep which indicates increasing strain at a decreasing rate when stress remains constant [1]. These phenomena could predict long-term time-dependent behaviour of a viscoelastic materials. A significant body of literature is available on the stress relaxation of polymeric materials with the changes of time at different temperatures [2–8]. Different annealing times above the glass transition temperature and various relaxation times have been applied for the relaxation behaviour of an amorphous poly(ethylene naphthalate) (PEN) by Cerrada and McKenna [2]. However, little work has so far been reported on PBT/PEN blends, which have been successfully produced in recent times [9–11]. These recent papers investigated the miscibility of the Nylon66/PEN and PBT/PEN blends by NMR, FTIR, XPS and SEM and mechanical and thermal properties by DMTA, DSC, tension and impact test. It is interesting to note that the enhancement of strengths in PBT/PEN blends has been attributed to close physical affinity rather than chemical reaction such as transesterification [9]. The present paper uses different levels of temperature, PEN content and initial stress to investigate the stress relaxation characteristics of PBT/PEN blends along with those of PEN and PBT homopolymers.

The mechanical responses of viscoelastic bodies, such as polymers, are poorly represented by either the spring model that represents the elastic property or the dashpot model that represents the viscous property. A better approximation results from the combination of the spring and dashpot elements, a simple example being the Maxwell Model [1] that connects a spring and a dashpot in series. In a stress relaxation experiment, if an instant stress is applied to a specimen to reach a level of strain, the stress changes to maintain a constant strain. The instantaneous strain is realised only in the spring element and the dashpot gradually undergoes

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DOI: 10.1007/s10853-005-1911-1

deformation forcing the spring element to relax accordingly. Thus the model shows the stress decreasing as a function of time, which describes the stress relaxation characteristics of different polymers reasonably well.

In the present study, four different equations for simulating relaxation curves of tested blends based on Maxwell model [12], have been tried using the experimental data of stress relaxation curves of PBT/PEN blends and homopolymers, and a prediction within a certain range of conditions has been attempted after acquiring the necessary coefficients in the equations.

2. Experimental details

2.1. Materials

Polymers used in this investigation were PEN purchased from Teijin Ltd. (PN-550) and PBT (Arnite T06-202) supplied by DSM Engineering Plastics. Pure PBT, pure PEN and PBT/PEN blends with 30 and 60 wt% PEN were dried at 70°C for 48 h in vacuum to remove moisture which would affect the properties of the materials. Mixtures were extruded with an Axon (Model BX-18-286) single screw extruder with 265–280°C die temperature and 70–80 rpm motor speed. After drying the pelletised materials at 70°C for more than 48 h in vacuum they were injection moulded using a BOY (Model 50M) automatic injection-moulding machine with the temperature of 265°C at nozzle and injection chamber and 40°C at mould before ejection. The shape of tensile specimens was manufactured in accordance with the ASTM standard D 638M (l: 200 mm, t: 4 mm, type 1). The moulded specimens were dried at 50°C for 48 h in an oven and put into desiccators at room temperature at least one hour before testing.

2.2. Experimental methods

An Instron testing machine (Model 5567) fitted with an Instron environmental chamber (Model 3119-006) was used to carry out the viscoelastic testing. After the environmental chamber was equilibrated to the desired temperatures (30 and 60°C), the specimen was gripped between two sets of jaws 130 mm apart and then was allowed to equilibrate to the conditions within the chamber for a period of 30–40 min. Once equilibrated, tensile stress was applied to the specimen by moving the upper grip at a constant rate (5 mm min^{-1} crosshead speed). When the desired initial stress had been reached, movement of the grip was halted and the decrease in stress with time (relaxation) was recorded for a period of 50 min.

The Taguchi method of experimental design is usually utilised to investigate the influence of factors on variability of a given response [13, 14]. However, in this work Taguchi method has been used purely to examine the main and interaction effects of a number of factors on the level of relaxation of PBT/PEN blends. The method was designed to analyse how different levels of temperature, PEN content and initial stress would affect the relaxation behaviour of PBT/PEN blends and homopolymers. The Taguchi method requires a quantifiable outcome to determine the effect of changes in different parameters and in this study the outcome selected was the stress after a period of 50 min as a percentage of initial stress. Two separate eight-run, two level experiments, each based on three factors, were used to analyse the relaxation results because three factors and interaction effects could be placed within eight trials. These constituted full factorial experimental designs, as every possible combination of the three factors at each of the two levels, were tried.

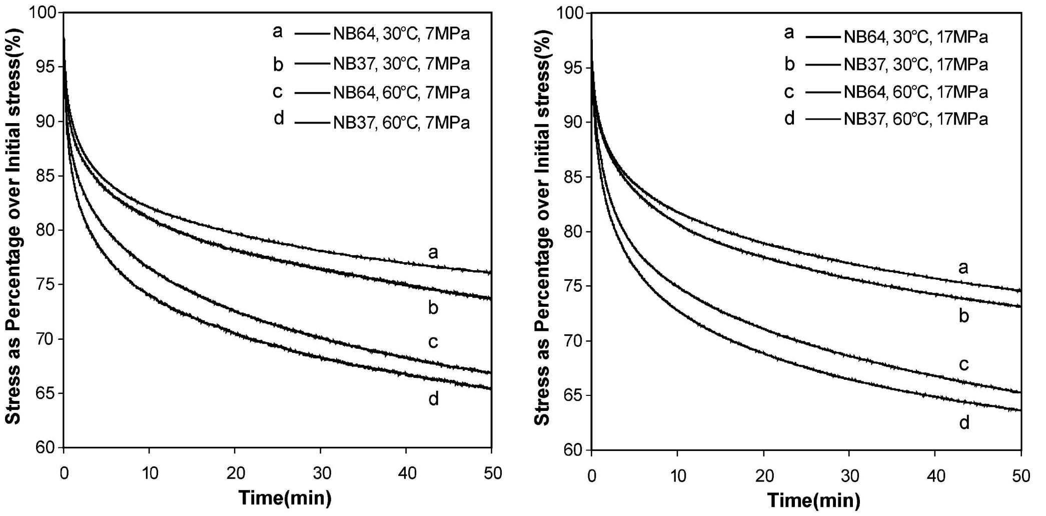
TABLE I. The different conditions in Series 1 as assigned by Taguchi method for analysing the relaxation behaviour of PBT/PEN blends

	A: PEN (wt%)		B: Temperature (°C)		C: Stress (MPa)	
Standard order trial number	30 Level 1	60 Level 2	60 Level 1	30 Level 2	17 Level 1	7 Level 2
1	✓		✓		✓	
2	✓		✓			✓
3	✓			✓	✓	
4	✓			✓		✓
5		✓	✓		✓	
6		✓	✓			✓
7		✓		✓	✓	
8		✓		✓		✓

TABLE II The different conditions in Series 2 as assigned by Taguchi method for analysing the relaxation behaviour of PBT/PEN homopolymers

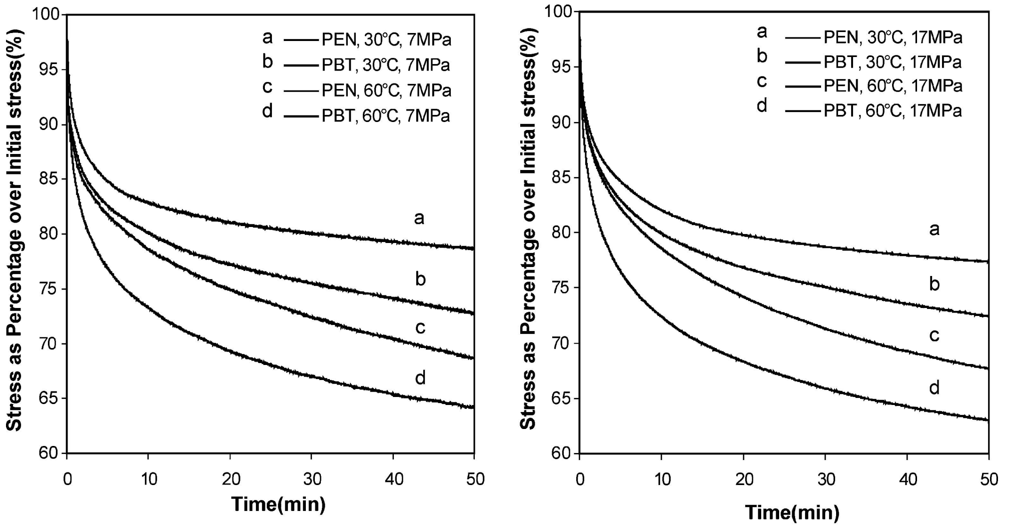
	A: Polymer		B: Temperature (°C)		C: Stress (MPa)	
Standard order trial number	PBT Level 1	PEN Level 2	60 Level 1	30 Level 2	17 Level 1	7 Level 2
1	✓		✓		✓	
2	✓		✓			✓
3	✓			✓	✓	
4	✓			✓		✓
5		✓	✓		✓	
6		✓	✓			✓
7		✓		✓	✓	
8		✓		✓		✓

The three factors used in Series 1 were temperatures of 30 and 60°C, initial stresses of 7 and 17 MPa, and PEN contents in PBT/PEN blends of 30 and 60 wt%, as shown in Table I. The three factors in Series 2 were the same as those in Series 1 except for the two pure homopolymers of PBT and PEN (Table II). The primary reason for using the same temperatures and initial stress in the first and second Series was to compare the stress relaxation percentage for different PEN contents. The initial stress level of 17 MPa was determined as approximately 25% of the maximum tensile stress of PEN. However, rather than using the 25% of the maximum tensile stress of PBT (i.e. 14 MPa), 7 MPa was used considering that close initial stresses might not be able to clearly identify the stress relaxation behaviour of various specimens. All level 1 factors in Tables I and II were determined in accordance with the expected lower final stress relaxation percentage between two levels of each factor, i.e. the low content of PEN, the high temperature and the high initial stress were assumed to result in the lower relaxation percent-



(a) (b)

Figure 1 Stress relaxation curves of PBT/PEN blends at different temperatures and initial stresses: (a) at 7 MPa (b) at 17 MPa [NB37: PEN 30 wt% and PBT 70 wt%; NB64: PEN 60 wt% and PBT 30 wt%].



(a) (b)

Figure 2 Stress relaxation curves of homopolymers at different temperatures and initial stresses: (a) at 7 MPa and (b) at 17 MPa.

age. The trials, listed in Tables I and II, were conducted in a random order to help prevent any unexpected factors, which might change with time, from distorting the analysis and resulting in misleading conclusions [13].

3. Results and discussion

3.1. Taguchi method of experimental design

All the stress relaxation curves obtained for the trials outlined in Tables I and II are shown in Figs 1 and 2. As expected, the trends of the relaxation characteristics of PBT/PEN blends are very similar to those of the homopolymers with an increase in temperature results in an increase in the amount and rate of stress relaxation.

In addition, the relaxation response at a given stress is in accordance with the weight content of PEN: a higher proportion of PEN results in a slower stress relaxation. According to the previous tensile tests, of the tensile strength at yield among different kinds of specimens PEN has the highest value (PEN: 67.9 MPa, PBT: 54.7 MPa). This agrees well with the trend found in each of the relaxation curves. The relaxation rate at 17 MPa is faster than that at 7 MPa, while the effects of different initial stresses are small in comparison with other factors.

The response tables for Series 1 and 2 are shown in Tables III and IV respectively. The layout for these response tables has been obtained from Lochner and Matar [13]. The response values used in these tables are the stress magnitudes after a period of 50 min as the percentages of initial stresses. These tables are used as a means for calculating the effects of each factor and also the interaction effects between the various factors. The main effects are calculated in the columns labelled A, B, and C, while the interaction effects are calculated in the columns labelled AB, AC, and BC. All these effects are calculated by subtracting the Level 1 average from Level 2 average. For example, the effect for temperature (factor B) in Table III is (74.4%) − (65.3%) = 9.1%. It should be noted that some of the values shown in Tables III and IV have been rounded to three significant figures and therefore may not give exactly the same answers when recalculated.

TABLE III. Response table for Series 1: L1 and L2 represent levels 1 and 2

Random order trial number	Standard order trial number	Percentage of initial stress at 50 min (%)	A: PEN (wt%)		B: Temperature (°C)		C: Stress (MPa)		AB		AC		BC	
			30	60	60	30	17	7						
			L1	L2	L1	L2	L1	L2	L1	L2	L1	L2	L1	L2
4	1	63.64	63.6		63.6		63.6			63.6		63.6		63.6
1	2	65.38	65.4		65.4			65.4		65.4	65.4		65.4	
8	3	73.13	73.1			73.1	73.1		73.1			73.1	73.1	
5	4	73.73	73.7			73.7		73.7	73.7		73.7			73.7
6	5	65.26		65.3	65.3		65.3		65.3		65.3			65.3
3	6	66.87		66.9	66.9			66.9	66.9			66.9	66.9	
2	7	74.58		74.6		74.6	74.6			74.6	74.6		74.6	
7	8	76.05		76.1		76.1		76.1		76.1		76.1		76.1
Total		558.63	275.9	282.8	261.1	297.5	276.6	282.0	279.0	279.7	278.9	279.7	280.0	278.7
Number of values		8	4	4	4	4	4	4	4	4	4	4	4	4
Average		69.83	69.0	70.7	65.3	74.4	69.2	70.5	69.7	69.9	69.7	69.9	70.0	69.7
Effect			1.7		9.1		1.4		0.2		0.2		-0.3	

TABLE IV Response table for Series 2: L1 and L2 represent levels 1 and 2

Random order trial number	Standard order trial number	Percentage of initial stress at 50 min (%)	A: Polymer		B: Temperature (°C)		C: Stress (MPa)		AB		AC		BC	
			PBT	PEN	60	30	17	7						
			L1	L2	L1	L2	L1	L2	L1	L2	L1	L2	L1	L2
4	1	63.00	63.0		63.0		63.0			63.0		63.0		63.0
1	2	64.20	64.2		64.2			64.2		64.2	64.2		64.2	
8	3	72.42	72.4			72.4	72.4		72.4			72.4	72.4	
5	4	72.76	72.8			72.8		72.8	72.8		72.8			72.8
6	5	67.65		67.7	67.7		67.7		67.7		67.7			67.7
3	6	68.56		68.6	68.6			68.6	68.6			68.6	68.6	
2	7	77.34		77.3		77.3	77.3			77.3	77.3		77.3	
7	8	78.61		78.6		78.6		78.6		78.6		78.6		78.6
Total		564.54	272.4	292.2	263.4	301.1	280.4	284.1	281.4	283.2	282.0	282.6	282.5	282.0
Number of values		8	4	4	4	4	4	4	4	4	4	4	4	4
Average		70.57	68.1	73.0	65.9	75.3	70.1	71.0	70.3	70.8	70.5	70.6	70.6	70.5
Effect			4.9		9.4		0.9		0.4		0.2		−0.1	

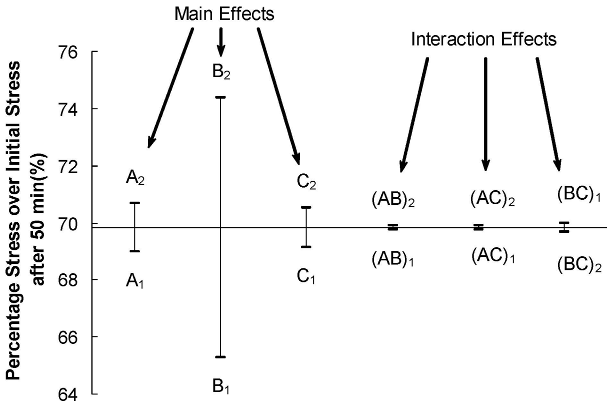


Figure 3 Estimated main and interaction effects of the three factors in Series 1: "A" represents PEN content, "B" represents temperature and "C" represents initial stress. The subscripts "1" and "2" represent low and high levels respectively.

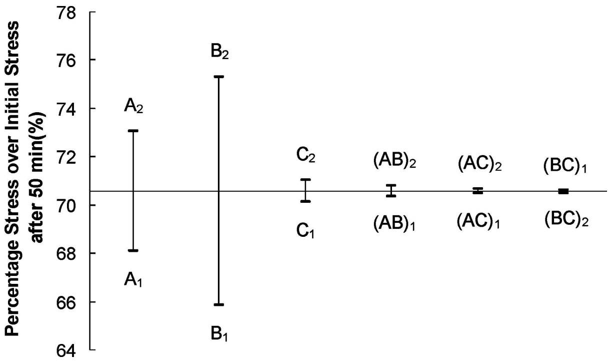


Figure 4 Estimated main and interaction effects of the three factors in Series 2: "A" represents homopolymers, "B" represents temperature and "C" represents initial stress. The subscripts "1" and "2" represents low and high levels respectively.

A graphical representation of the estimated main and interaction effects for Series 1 and 2 are illustrated in Figs 3 and 4 respectively. The averages at the bottom of the columns in Tables III and IV labelled "Percentage of Initial Stress at 50 min" are the values at which the *x*-axis intercepts the y-axis in Figs 3 and 4 respectively. In Fig. 3, A_1 represents the "Level 1" PEN content (30 wt%) and A_2 represents the "Level 2" PEN content (60 wt%), while in Fig. 4 A_1 represents pure PBT and A_2 represents pure PEN. The values used for these points are the averages of the corresponding columns as calculated in the response tables. Similarly, B_1 and B_2 represent Level 1 and 2 temperatures respectively, while C_1 and C_2 represent Level 1 and 2 initial stress respectively for both Figs 3 and 4.

The results shown in Figs 3 and 4 suggest that for both set of data high temperature leads to the greatest amount of stress relaxation. It appears that the factor with most influence on the relaxation behaviour in Series 1 is B (temperature), while the remaining factors are less effective and interaction effects have little influence by comparison (Fig. 3). Similarly, in Series 2, it appears that factors B and A have the most influence on stress relaxation (Fig. 4). The smaller influence of A (PEN content) in Series 1 is because the properties of blends are between those of PBT and PEN, which results in stress relaxation rates between two extremes [9, 10].

TABLE V Example of how the AB interaction for Series 1 is calculated

Standard order trial no.	Factors: A: PEN Composition (%)	Factors: B: Temperature (°C)	Percentage of initial stress at 50 min (%)	Average
1,2	30[A_1]	60[B_1]	63.6, 65.4	64.5
3,4	30[A_1]	60[B_2]	73.1, 73.7	73.4
5,6	60[A_2]	30[B_1]	65.3, 66.9	66.1
7,8	60[A_2]	30[B_2]	74.6, 76.1	75.3

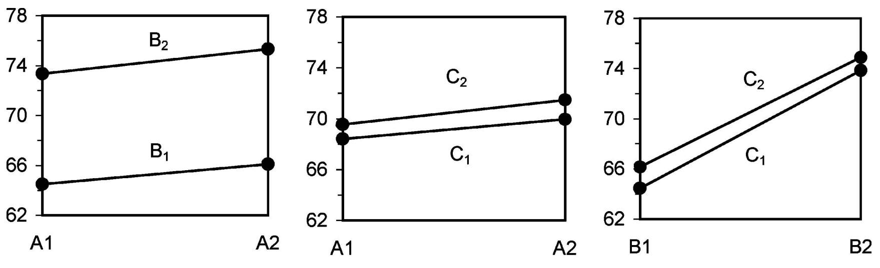
Interactions between factors can be described as the influence of one factor on the level of another factor. The interaction effects plotted in Figs 3 and 4 merely indicate which interactions are of most significance. In order to observe what the actual interactions are, they need to be plotted on individual graphs. Interaction effects for A and B, A and C, and B and C for Series 1 and 2 are shown in Figs 5 and 6 respectively. An example of how the points for the graph in Fig. 5a representing the AB interaction for Series 1 were calculated is shown in Table V. The average values in the last column of this table are the end points for the two lines in Fig. 5a. All of the interaction effects plotted in Figs 5 and 6 were calculated in a similar manner.

The nearly parallel lines in Figs 5a, b and c indicate that there is no strong interaction between any of the factors in Series 1. The short bars plotted for the interaction effects in Fig. 3 also reflect this. However, it is of interest to note that Fig. 5a indicates that at the low and high PEN content levels (A_1 and A_2), some change in response (stress relaxation) occurs due to change of temperatures (a big gap between B_1 and B_2) with little change according to the other factors. Similarly, Fig. 6 (Series 2) also shows some effects due to change of temperature (B) and little effect on the response due to a change in homopolymers and initial stress. Therefore, temperature appears to be most influential factor on the relaxation behaviour of both blends and homopolymers with little interaction among the factors.

From the point of materials science it is important to check the possibility of transesterification between PEN and PBT and the change of crystallinity of the materials during material processing, which will influence the final properties of the blends. From NMR analysis it was found that no significant transesterification reaction had taken place under the conditions that were used (a short residence time during extrusion and injection moulding) [9]. The increase of crystalline phase of the blends by annealing at 200°C was also examined and the moulded specimens used in this study showed almost amorphous phase [15]. Hence, it may be concluded that the relaxation behaviour reported in this paper was influenced by physical interaction rather than any chemical reaction such as transesterification or hydrolysis of the component polymers.

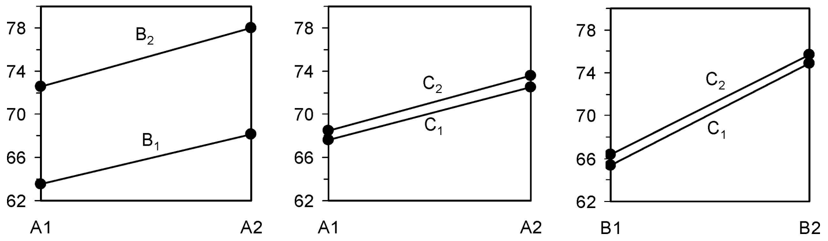
3.2. Fitting functions to the relaxation curves

Four different equations with a gradual increase in complexity are used to fit the relaxation curves of the



(a) AB Interaction (b) AC Interaction (c) BC Interaction

Figure 5 Interaction effects for Series 1 where "A" represents PEN content, "B" represents temperature and "C" represents initial stress.



(a) AB Interaction (b) AC Interaction (c) BC Interaction

Figure 6 Interaction effects for Series 2 where "A" represents homopolymers, "B" represents temperature and "C" represents initial stress.

PBT/PEN blends and the homopolymers at different temperatures and PEN contents, and the experimental data are used to predict stress terms (σ) as stresses change with time. The functions used are:

$$\sigma(t) = \sigma_o e^{mt} \quad (1)$$

$$\sigma(t) = \mathrm{M}e^{mt} + (\sigma_o - \mathrm{M}) \quad (2)$$

$$\sigma(t) = \mathrm{M}e^{mt} + (\sigma_o - \mathrm{M})e^{nt} \quad (3)$$

$$\sigma(t) = \mathrm{M}e^{mt} + \mathrm{N}e^{nt} + (\sigma_o - (\mathrm{M} + \mathrm{N})) \quad (4)$$

where, t is the time in seconds, σ_o is the stress at the beginning of the relaxation test, and M, N, m and n are coefficients which are determined using Matlab$^{\mathrm{TM}}$ to obtain a function which has best fit to the experimental data. Note that even though the same letters are used to represent the coefficients in the different equations, their numerical values are different for each equation.

An example of Equations 1 to 4 fitted to the stress relaxation curve of the PBT/PEN blend (40/60 wt%) in an environment at 60°C and 7 MPa is shown in Fig. 7. The coefficients for these equations are given in Table VI. From Fig. 7, it is clear that Equation 1 that follows the simplest form of Maxwell model, provides the worst fit to the experimental data. This is not surprising at all considering the fact that these simple models often fail to describe the time-dependent behaviour adequately, particularly for the linear polymers of sufficiently high molecular weight having at least two major transitions (glass to rubber and rubber to liquid). Furthermore, the decay of stress by given the Maxwell model is often much more rapid than the corresponding stress decay exhibited by the real polymers in either transition region. In order to overcome these deficiencies, multi-element models (connected in series and parallel) are often used [1, 16–19] although they require more rigorous analytical and experimental evaluations to obtain the most accurate fit. In the present study, it appears from Fig. 7 that it is necessary to go the levels of Equations 3 or preferably 4 and use double time constants to adequately describe the relaxation data obtained from experiments.

To some extent, the coefficients for Equations 3 and 4 are related to the temperature and PEN contents of the PBT/PEN blends during testing. The coefficients for the four equations are determined from the experimental data obtained from the stress relaxation tests carried out with initial stresses of 7 and 17 MPa including the data for PBT/PEN blends and homopolymers. However, in Fig. 8, the coefficients of Equation 4 only are plotted against PEN content and temperature at 7 MPa, and the other similar results are omitted for brevity. Linear regressions are done to the data points for constant temperature to help identify any trend.

When Equation 4 is fitted to the relaxation data of Series 1 and 2 a linear relationship with temperature and PEN content is observed for the "M" coefficient. Figs. 8a shows that an increase in temperature results in an increase in the "M" values of Equation 4, whereas an increase in PEN content results in a decrease of "M". However, Fig. 8b shows that the "N" coefficient increases with an increasing PEN content at 60°C, whereas it shows a reverse trend at 30°C. There is

TABLE VI Coefficients for the equations fitted to the experimental relaxation data of the PBT/PEN blend (40/60 wt%) at 60°C and an initial stress of 7 MPa as shown in Fig. 7

	Coefficients			
Equations	M	m	N	n
$\sigma(t) = \sigma e^{mt}$		-1.7×10^{-4}		
$\sigma(t) = \mathrm{M}e^{mt}+(\sigma-\mathrm{M})$	31.0	-2.8×10^{-3}		
$\sigma(t) = \mathrm{M}e^{mt}+(\sigma-\mathrm{M})e^{nt}$	21.7	-9.9×10^{-3}		-5.6×10^{-5}
$\sigma(t) = \mathrm{M}e^{mt}+\mathrm{N}e^{nt}+(\sigma-(\mathrm{M}+\mathrm{N}))$	15.9	-2.7×10^{-2}	18.9	-7.9×10^{-4}

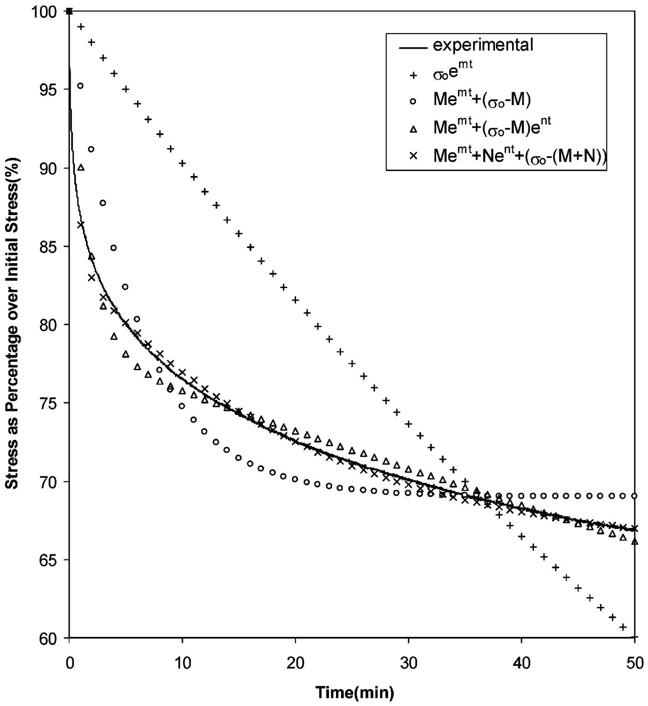


Figure 7 Different equations fitted to the relaxation curve of PBT/PEN blend (40/60 wt%) at 60°C and an initial stress of 7 MPa.

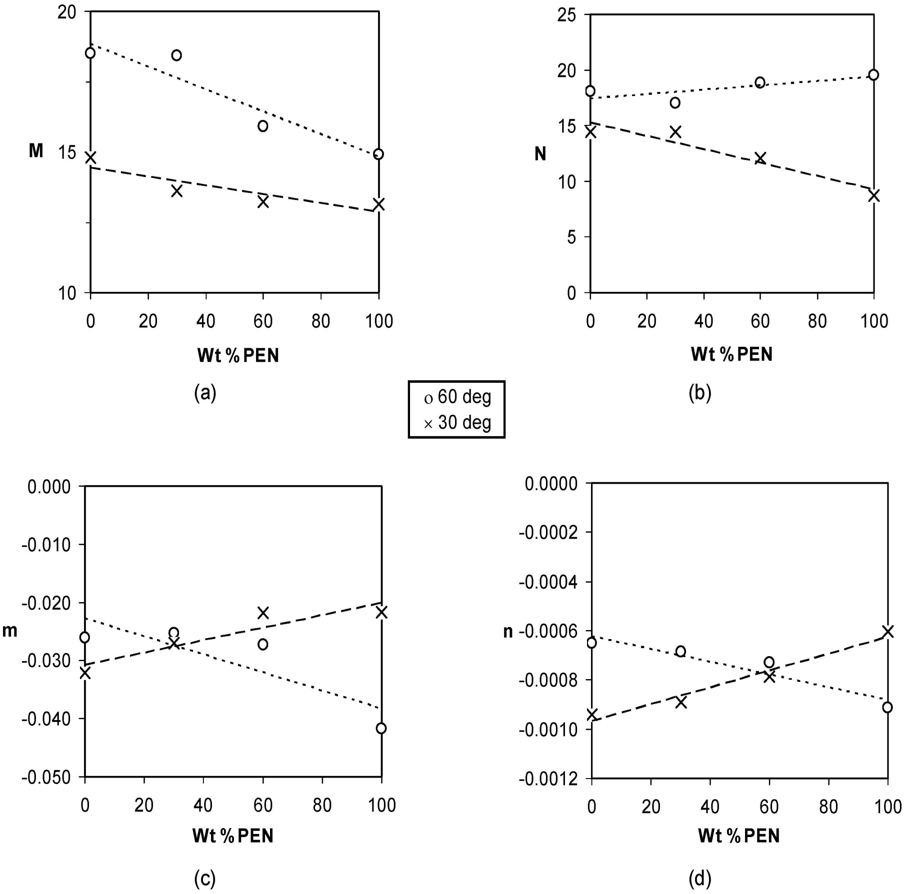


Figure 8 (a) "M" coefficient, (b) "N" coefficient, (c) "m" coefficient, and (d) "n" coefficient in Equation 4 versus PEN content at two different temperatures (30 and 60°C) and an initial stress of 7 MPa.

reasonable evidence to suggest that the "N" coefficient generally increases with an increasing temperature. The "m" and "n" coefficients in Figs 8c and d show similar trends to those of "N" coefficient. In general, "m" and "n" are increase with an increasing PEN content at 60°C, whereas they are decrease with an increasing PEN content at 30°C (Table VII). However, no clear trend with temperature is shown by the "m" and "n" coefficients in Fig. 8c and d. Despite this, the values for "m" and "n" coefficients at a given temperature and PEN content could be predicted within a certain range.

Using Fig. 8, the coefficients of Equation 4 can be estimated with a reasonable amount of certainty to provide an estimate of the relaxation behaviour of

TABLE VII The coefficients used for simulation of the stress relaxation curves of PBT/PEN blends (50 wt% PEN content) at temperatures of 30 and 60°C, and an initial stress of 7 MPa

	Temperature	
Coefficients	60°C	30°C
M	16.8	13.7
N	18.4	12.3
m	-3.0×10^{-2}	-2.6×10^{-2}
n	-8.0×10^{-4}	-7.4×10^{-4}

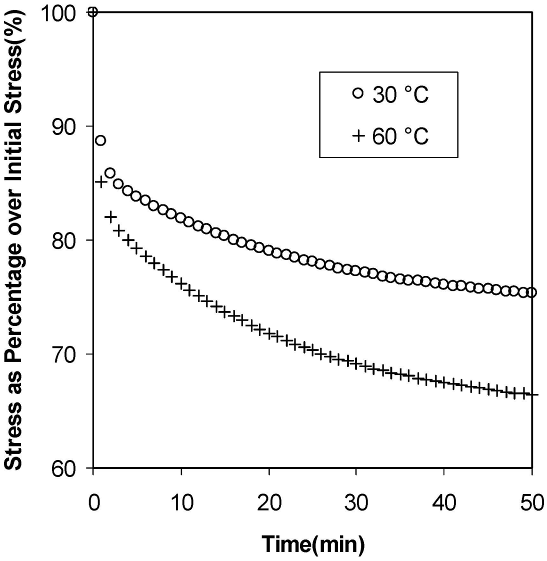


Figure 9 The predicted stress relaxation curves of PBT/PEN blends (50 wt% PEN content) at temperatures of 30 and 60°C, and an initial stress of 7 MPa.

PBT/PEN blends at any temperature from 30 to 60°C and any PEN content. For coefficients, such as "m" and "n" for Equation 4, the average values of their ranges, Figs 8c and d, would likely to be sufficient. Similarly, coefficients "M" and "N" for Equations 4, can be interpolated from Figs 8a and b for any PEN content and an initial stress 7 MPa. Fig. 9 shows the simulated stress relaxation curves for PBT/PEN blends with 50 wt% PEN content and an initial stress 7 MPa are simulated by using Equation 4 and the averaged constants for two different temperatures of 30 and 60°C.

4. Conclusions

The response table and a graphical representation of main and interaction effects for the PBT/PEN blends show that the most significant factor is temperature; as expected, an increase in temperature accelerates the stress relaxation, as in the case of PBT and PEN homopolymers. However, a less pronounced effect of PBT or PEN weight content is observed in the cases of PBT/PEN blends in comparison to the homopolymers, Figs 1 and 2. The initial stress is the least effective factor for both the blends and the homopolymers, and the interaction effects among the factors are negligible. For describing the stress relaxation data, double exponential forms given in Equations 3 are 4, which may be derived from the multi-element linear solids are necessary and Equation 4 containing an additional term gives a much better fit. It is significant that the "M" coefficient of Equation 4 decreases linearly (Figs. 8a) with wt% PEN at both 30 and 60°C. The "N" coefficient is increasing with increasing temperature, but no clear trend with temperature is shown in the "m" and "n" coefficients. Coefficients "M" and "N" interpolated from the experimental data in Figs. 8a and b, and the average values of coefficients "m" and "n" from Figs. 8c and d can be utilised to simulate stress relaxation curves of PBT/PEN blends at temperatures of 30 and 60°C and 50 wt% PEN content, at 7 MPa initial stress.

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Received 29 May 2003
and accepted 4 January 2005