

Evaluation of Mechanical Properties of Polypropylene/Polycarbonate/SEBS Ternary Polymer Blends Using Taguchi Experimental Analysis

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ABSTRACT: In this work, ternary polymer blends based on polypropylene (PP)/polycarbonate (PC)/poly(styrene-*b*-(ethylene-*co*-butylene)-*b*-styrene) (SEBS) triblock copolymer and a reactive maleic anhydride grafted SEBS (SEBS-*g*-MAH) at fixed compositions are prepared using twin-screw extruder at different levels of die temperature (235–245–255°C), screw speed (70–100–130 rpm), and blending sequence (M1–M2–M3). In M₁ procedure, all of the components are dry blended and extruded simultaneously using Brabender twin-screw extruder, whereas in M₂ procedure, PC, SEBS, and SEBS-*g*-MAH minor phases are first preblended in twin-screw extruder and after granulating are added to PP continuous phase in twin-screw extruder. Consequently, in M₃ procedure, PP and SEBS-*g*-MAH are first preblended and then are extruded with other components.

The influence of these parameters as processing conditions on mechanical properties of PP/PC/SEBS ternary blends is investigated using L9 Taguchi experimental design. The responding variables are impact strength and tensile properties (Young's modulus and yield stress), which are influenced by the morphology of ternary blend, and the results are used to perform the analysis of mean effect as well. It is shown that the resulted morphology, tensile properties, and impact strength are influenced by extrusion variables. Additionally, the optimum processing conditions of ternary PP/PC/SEBS blends were achieved via Taguchi analysis. © 2010 Wiley Periodicals, Inc. *J Appl Polym Sci* 116: 2312–2319, 2010

Key words: ternary polymer blends; mechanical properties; Taguchi analysis

INTRODUCTION

Blending two or more immiscible polymers is a well-known method to obtain new polymeric materials with synergistic and tailored properties. The final size and shape of the minor phase in polymer blend is a function of several factors as composition, viscosity ratio, interfacial tension, shear rate, elasticity, and processing conditions.¹ In most cases, blending of polymers without compatibilizers has not led to enhanced properties in final products. To alleviate the weak interfacial adhesion of immiscible blends, *in situ* compatibilization has been extensively used in recent studies. In both polypropylene (PP)/ polyamide-6 (PA-6) binary and PP/PA-6/Poly(styrene-*b*-(ethylene-*co*-butylene)-*b*-styrene) (SEBS) ternary systems, using maleated SEBS (SEBS-*g*-MAH) as a compatibilizer, strongly influenced the blend morphology and mechanical properties by variation in the degree of interfacial reaction between the succinic anhydride groups of the SEBS-*g*-MAH and the terminal amino groups of PA6.^{2–4} Recently, the study of ter-

nary blends has raised the attention of researches cause of wide range of variation in mechanical and morphological properties in these systems.^{5–17} It is observed that for the systems containing two minor phases, three distinct types of phase morphology have to be specified. For some ternary systems, one of the minor components formed an encapsulating layer around domains of another minor component, whereas in other systems, two minor components formed independent phases separately. The third type is the intermediate case, where mixed phases of the two components are formed without any ordered structures.^{5–11} To predict the tendency for one minor phase to encapsulate a second one, the alternative form of Harkin's equation have been used as follows:

$$\lambda_{BC} = \gamma_{AC} - \gamma_{AB} - \gamma_{BC} \quad (1)$$

where γ_{AC} , γ_{AB} , and γ_{BC} are the interfacial tension for each component pair, and λ_{BC} is defined as the spreading coefficient for the shell forming component B on the core of component C. The index A corresponds to the matrix continuous phase. If the λ_{BC} is positive, the B-phase will encapsulate the C-phase. Similarly,

$$\lambda_{CB} = \gamma_{AB} - \gamma_{AC} - \gamma_{BC} \quad (2)$$

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When λ_{CB} is positive, the component C will encapsulate component B. However, if both λ_{CB} and λ_{BC} are negative, component C and B will tend to form two separate dispersed phases within the matrix component A. In the intermediate region, where $\lambda_{BC} \approx 0$, stack morphology may result in which component B only partially eliminates the interface between component C and the matrix.³ Guo et al. have applied their model to different ternary blends and have compared the predicted morphologies with experimental results.⁵ In addition, they have successfully converted the phase structures of ternary blends from one type to other using interfacial active block copolymers. Luzinov et al. have shown that the morphology of the ternary polystyrene (PS)/styrene-butadiene-rubber (SBR)/polyethylene (PE) blend is influenced by the weight ratio of the minor component (SBR and PE) and stress transfer from the matrix through the shell to the core occurs when the ratio of the core size to thickness of the SBR layer is high enough.⁶⁻⁸ Also upon increasing the viscosity of polyolefin's, the size of the cores and SBR domains including them increases. In the case of dispersed phase morphology, the hard core in the core-shell phases has no major effect on the mechanical properties, whatever the matrix is (PE or PS). The effect of melt viscosity ratio on morphology of ternary blends is very complex. Kim et al. described that for polyolefin ternary polymer blends with core-shell morphology when two minor phases have the same composition, the minor phase with lower viscosity encapsulate the one with higher viscosity.⁹ Ha et al. reported for PP/ethylene-octene copolymer (mPE)/high-density polyethylene (HDPE), fibrils were observed when the dispersed phase viscosity (mPE/HDPE) was less than that of PP.¹⁰ Nemirovski et al. suggested that for some ternary blends (thermoplastic/thermotropic) core-shell morphology (A will encapsulate B in matrix C) was observed when both thermodynamic (a positive spreading coefficient) and kinetic effects (a dispersed phase viscosity ratio smaller than one) act simultaneously.¹¹ Moreover, some processing conditions such as feeding sequence, extrusion rate, and temperature profile along the screw can significantly affect the mechanical and morphological properties.^{15,18} In another research, the morphology of high-density polyethylene(HDPE)/PS/poly(methyl-metacrylate)(PMMA) ternary blends prepared by twin-screw extrusion is investigated as a function of composition, minor phase viscosity ratios, sequence of addition, and compounding production rate by Favis and co-workers.¹⁵ The results have been shown that within explored processing conditions, the final morphology in the studied system is primarily governed by interfacial free energy considerations. It is found that the yield stress and Young's modulus of the PET/

PC/E-GMA-MA ternary blends decreased with increasing copolymer content at room temperature, but no significant blending sequence effect was observed.¹⁸ The results are analyzed based on toughening mechanisms during the fracture of toughened ternary blend using combined cavitations and matrix shear yielding mechanisms. As a marked result, the number and size of core structures can influence the mechanical properties and morphology of ternary blends.^{16,17} In PP/HDPE/EPDM/EP quaternary blends containing more than 30 wt % of EPDM, the impact strengths have been extensively improved.¹⁹ The higher the PP content in this system, the larger the EPDM/HDPE ratio needed for achieving good impact toughness at low temperature. According to the mentioned literature reviews, the investigation of effective parameters in ternary blend is completely different from one system to another. In this study, the effect of processing conditions including screw speed, temperature profile, and mixing sequence are investigated for PP/PC/SEBS ternary blend using twin-screw extruder. To evaluate the effects of processing conditions on mechanical properties and morphology evolution, the experiments are designed using Taguchi method of experimental design. The blends are compatibilized with SEBS-g-MAH to achieve good adhesion between three phases based on previous studies.²⁻⁴ Attention will be focused to the phase morphology and its effect on the mechanical properties via manipulation of processing conditions.

EXPERIMENTAL

Materials

The materials used in this research were: (i) an isotactic polypropylene homo-polymer (PP), SEETEC H5300 supplied by LG chemical company (Korea) (MFI: 3.5 g/10 min, 230°C, 2.16 kg), (ii) Polycarbonate (PC), Makrolon 2858 purchased from Bayer Co (Germany) (MFI: 10 g/10 min, 300°C, 1.2 kg), (iii) Poly(styrene-*b*-(ethylene-*co*-butylene)-*b*-styrene) (SEBS) triblock copolymer, Kraton™ G1652 supplied by Shell Chemicals (29% styrene; molecular weight : styrene block 7000, EB block 37500), and (iv) Maleic-anhydride grafted SEBS (SEBS-g-MAH) triblock copolymer, Kraton™ FG1901x supplied by Shell Chemicals (29% styrene, nominal weight percent of grafted maleic anhydride: $1.8 \pm 0.4\%$).

Experimental design by Taguchi method

The application of design of experiments requires careful planning prudent layout of the experiments and expert analysis of result. The Taguchi method replaces factorial design with the more suitable

TABLE I
Variation Fashion of Three Independent Parameters

Parameters	Symbol	Unit	Nominal levels
Temperature of die	A	(°C)	235-245-255
Screw speed	B	(rpm)	70-100-130
Blending sequence	C	–	M ₁ -M ₂ -M ₃

partial factorial method based on orthogonal arrays. Because partial factorial design is a subset of full factorial method, so as to determine the reliability and accuracy of the experimental results, standard statistical method of analysis of variance was exploited. In this method, the variance of data is more interest and directly would give an evaluation of the accuracy results.²⁰ The most appropriate orthogonal array to meet this requirement is a 9-trial experimental (L9). In this design, three independent variables are statistically changed at three different levels on the basis of process knowledge.²¹ In recent years, there are some applied researches on polymer-based processes using L9 method.^{22,23} In fact, by using this experimental analysis, the responding variable can be optimized on basis statistical–mathematical calculations via L9 design of Taguchi method.

Blend preparation

As it is mentioned, to investigate the effect of independent parameters on responding variable for specified experimental runs, Taguchi analysis is a suitable method for reducing the number of runs and evaluating the variation trends based on statistical mathematical analysis. The three independent processing variables in this study are die temperature, screw speed, and blending sequence, which are considered on the basis of literature studies^{1,15,18} using L9 Taguchi design. These changing variables were prepared in nine ternary blends with the same compositions (70 wt % PP, 15 wt % PC, 7.5 wt % SEBS, 7.5 wt % SEBS-g-MAH) in Brabender corotating twin-screw extruder (diameter of screw = 2 cm, length/diameter ratio = 40). The temperature profile of Barrel in five heat zones are altered based on die temperature: A1 profile: 210-215-220-225-230°C; A2 profile: 220-225-230-235-240°C; A3 profile: 230-235-240-245-250°C. Blending sequence is consisting of three different procedures M₁, M₂, and M₃. In M₁ procedure, all of the components are dry blended and extruded simultaneously using Brabender twin-screw extruder. In M₂ procedure, PC, SEBS, and SEBS-g-MAH minor phases are first preblended in twin-screw extruder and after granulating are added to PP continuous phase in twin-screw extruder. Consequently, in M₃ procedure, PP and SEBS-g-MAH are first preblended and then are extruded with other components. Table I represents the nominal independent parameters.

The results of Taguchi L9 design for the mentioned parameters are listed in Table II.

The responding variables are impact energy and tensile properties (Young's modulus and yield stress), which are influenced by the morphology of ternary blends. After analyzing Taguchi method, the optimized conditions will be achieved.

Mechanical properties

After melt blending of designed compounds in twin-screw extruder, the blends were quenched in cooling water bath and pelletized in a granulator. Dried blends were molded to form tensile and impact specimens using an ENGEL injection molding machine. The Barrel temperature profile was 180°C (hopper) to 240°C (nozzle), and the mold temperature was maintained at 40°C. Tensile stress–strain data were obtained using Galdabini testing machine in the rate of 50 mm/min according to the ASTM D-638. Moreover, Izod impact strength was done for notched specimens according to ASTM D-256 using Zwick pendulum-type tester.

Morphological studies

To evaluate the effect of particle size and the type of resulted morphology on the mechanical properties of PP/PC/SEBS ternary blends, scanning electron microscopy (SEM) micrographs were obtained using AIS-2100 SEM supplied by SERON Company through fracture surface of impact specimens. Before doing SEM, the samples were fractured in liquid nitrogen and consequently were etched by cyclohexane for 24 h to remove SEBS and SEBS-g-MAH minor phases. Then, the etched samples were gold sputtered to make the samples conductive.

RESULTS AND DISCUSSION

Taguchi analysis of PP/PC/SEBS ternary blend

The experimental results for nine different trial conditions are listed in Table III. These experimental

TABLE II
Processing Conditions for Preparation of PP/PC/SEBS Ternary Blends Based on Taguchi L9 Method

Run no.	A (°C)	B (rpm)	C
1	235	70	M ₁
2	235	100	M ₂
3	235	130	M ₃
4	245	70	M ₂
5	245	100	M ₃
6	245	130	M ₁
7	255	70	M ₃
8	255	100	M ₁
9	255	130	M ₂

TABLE III
Experimental Results for PP/PC/SEBS

Run no	Yield stress (MPa)	Young's modulus (MPa)	Impact strength (J/m)
1	28.25 ± 0.74	1113.23 ± 57.71	88 ± 2
2	27.39 ± 0.26	1113.175 ± 39.765	81.5 ± 5.5
3	28.3 ± 0.85	1094.05 ± 37.9	114 ± 1
4	28.29 ± 0.315	1060.205 ± 55.185	67.75 ± 0.25
5	28.45 ± 0.53	1130.45 ± 48.03	115 ± 3
6	27.98 ± 0.67	1176.81 ± 163.76	102.3 ± 3.29
7	29.33 ± 1.17	1366.92 ± 171.69	95.25 ± 2.75
8	28.98 ± 0.53	1299.99 ± 106.62	87.5 ± 0.5
9	29.17 ± 0.31	1307.416 ± 30.29	94 ± 2.94
Pure PP	32.3 ± 0.39	1167 ± 22.6	25.66 ± 0.471

results have been set up to investigate the influence of processing conditions (die temperature, screw speed, and blending sequence) on the mechanical properties of PP/PC/SEBS ternary blend. The mechanical results of pure PP sample were added to Table III as 10th specimen to make an evidence to study the order of importance.

To investigate the role of processing condition on the resulted mechanical properties, the mean effect of each factor must be taken into account. Figure 1 shows the effect of die temperature on yield stress, Young's modulus, and impact strength, which all totally summarized as mechanical properties.

Figure 1(A) indicates the influence of temperature on the yield stress of the PP/PC/SEBS ternary blend.

As it can be seen, the yield stress of ternary blend steadily increases by increasing the temperature. This behavior can be attributed to interfacial adhesion between the phases in ternary blend, whereas the yield stress is known to be highly dependent on the interfacial adhesion among the other mechanical properties. Pukanaszky and coworkers^{24,25} showed that to obtain complete interfacial adhesion between phases, the yield stress of a blend must be obeyed by the mixture law as follows:

$$\sigma_{yb} = \sigma_{ym}(1 - \varphi_d) + \sigma_{yd}\varphi_d \quad (3)$$

where σ_{yb} , σ_{ym} , σ_{yd} , and φ_d are yield stress of the blend, yield stress of the matrix, yield stress of the dispersed phase, and volume fraction of the dispersed phase, respectively. In another words, the maximum deviation from mixture law [Eq. (3)] is indicating very poor interfacial adhesion and vice versa. In our study, the yield stress of pure PP and PC are 32.3 and 66 MPa, respectively. As the composition of other ingredients are low compared with PP and PC, the yield stress of the blend must be achieved ~ 35.9879 MPa. As can be seen, the experimental results in Table III show a minor negative deviation from the theoretical yield stress calculated by mixture law from Eq. (3), which is remarked in 7, 8, 9 series of this table. This observation is completely confirmed with SEM evaluations. Figure 2 represents the morphology of the 70/15/7.5/7.5

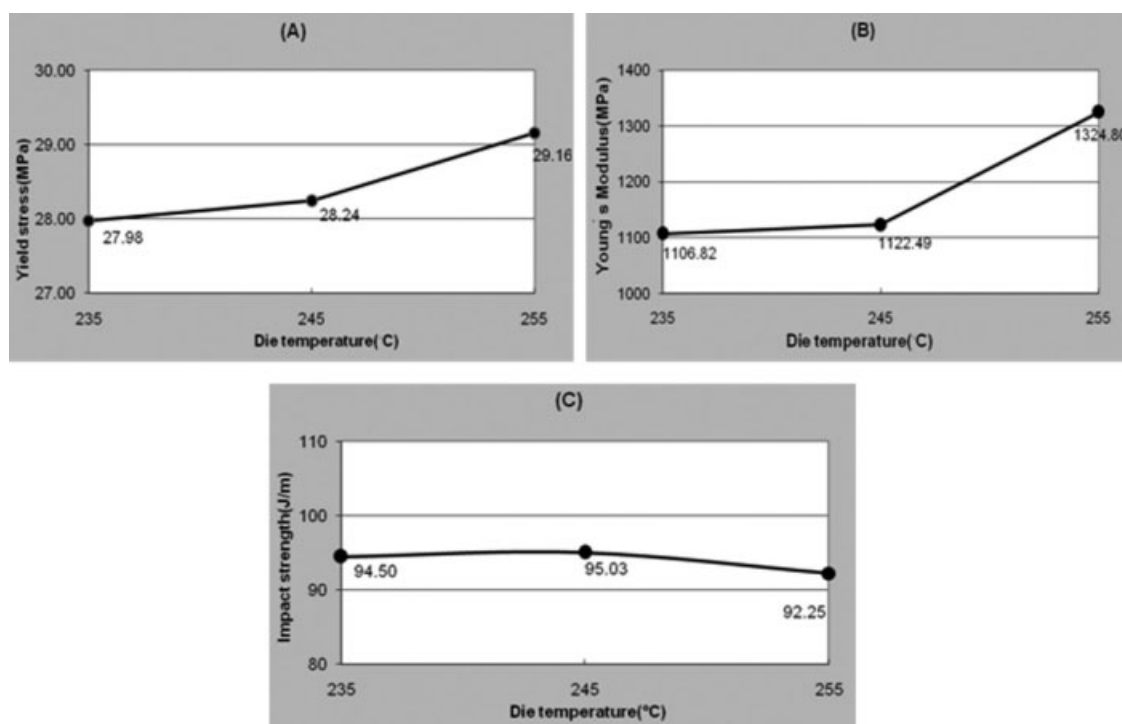


Figure 1 Mean effect of die temperature on the mechanical properties of PP/PC/SEBS ternary blends (A) yield stress, (B) Young's modulus, and (C) impact strength.

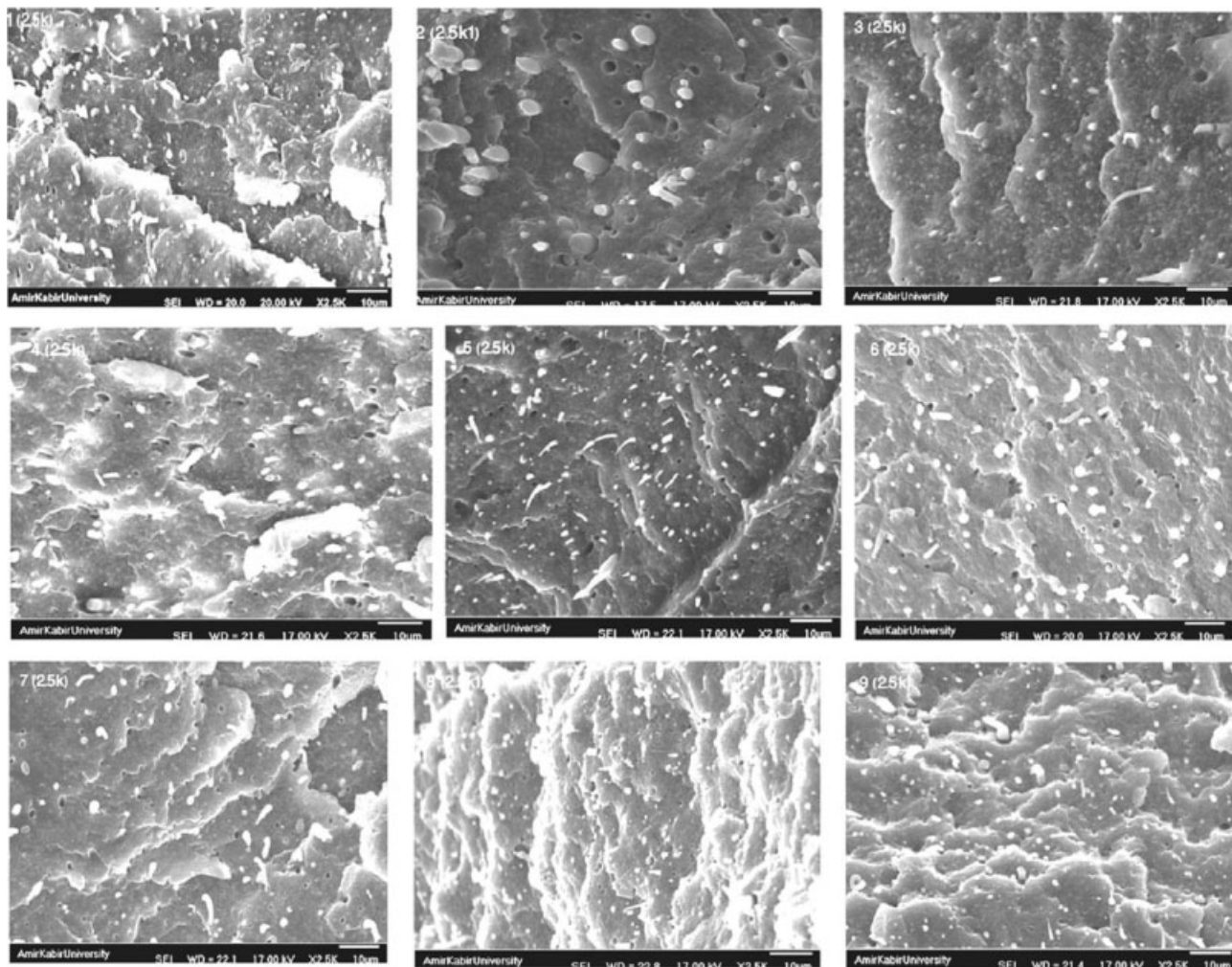


Figure 2 SEM micrographs of PP/PC/SEBS/SEBS-g-MAH at different processing conditions according to Table II.

blend of PP/PC/SEBS / SEBS-g-MAH according to L9 design listed in Table II.

As it can be seen, SEM micrographs of the etched impact fractured samples reveal the existence of composite droplets of SEBS and PC embedded in PP matrix with SEBS and SEBS-g-MAH shells around the PC and also individual droplets of PC and SEBS. According to the explanation given for the temperature effect, for the compound No. 8, the dispersion of droplets is finer than that of 2 and 5 compounds and smaller particle size of droplets can be distinguished. Figure 1(B) shows the influence of temperature on the Young's modulus of PP/PC/SEBS ternary blends. As it can be seen, the modulus of ternary blends increases slightly from 235 to 245°C and sharply from 245 to 255°C. This observation may be attributed to orientation of PC as a reinforcement in ternary blend. Increasing in temperature causes decreasing in viscosity, and so the probability of PC orientation during the process in twin-screw extruder increases, and this matter causes modulus

increases in PP/PC/SEBS ternary blend. The effect of die temperature on impact strength is shown in Figure 1(C). The impact strength fixes nearly with increasing temperature from 235 to 245°C and slight decrease in impact strength with enhancing the temperature from 245 to 255°C. This slight decay in impact strength may be attributed to molecular weight reduction of PP because of the thermomechanical degradation at high temperature. Figure 3 shows the effect of screw speed on mechanical properties. As it is shown in Figure 3(A) firstly, the yield stress decrease with increasing the screw speed and then increases slightly with screw speed. Figure 3(B) shows the effect of screw speed on Young's modulus. This behavior is expected because of the fact that the crystallinity of PP would be decreased with increasing the screw speed via reduction of molecular weight and consequent thermomechanical degradation of matrix. This effect is compensated partly by increasing the stiffness of blend because of orientation of PC, and thus Young's modulus increases from 70 to 100

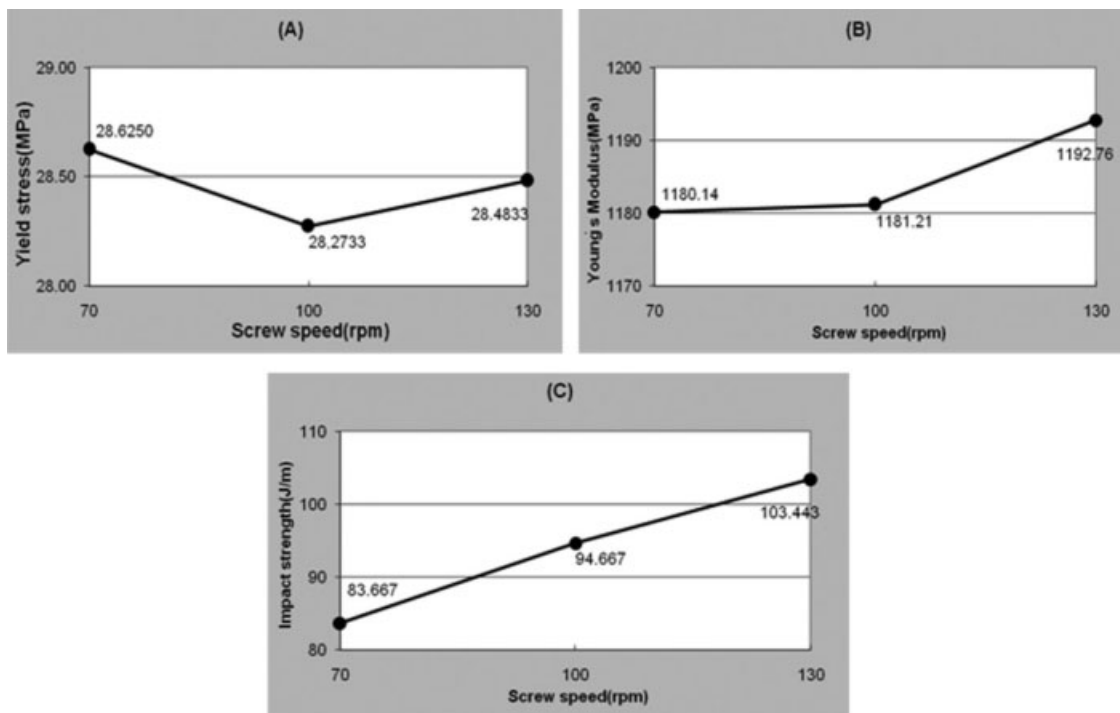


Figure 3 Mean effect of screw speed on the mechanical properties of PP/PC/SEBS ternary blends (A) yield stress, (B) Young's modulus, and (C) impact strength.

rpm and then the orientation of PC is governed at 130 rpm so that the Young's modulus increased sharply.

In Figure 3(C), the effect of screw speed on impact strength is shown. It can be seen that the impact resistance of the blend increases steadily by increasing

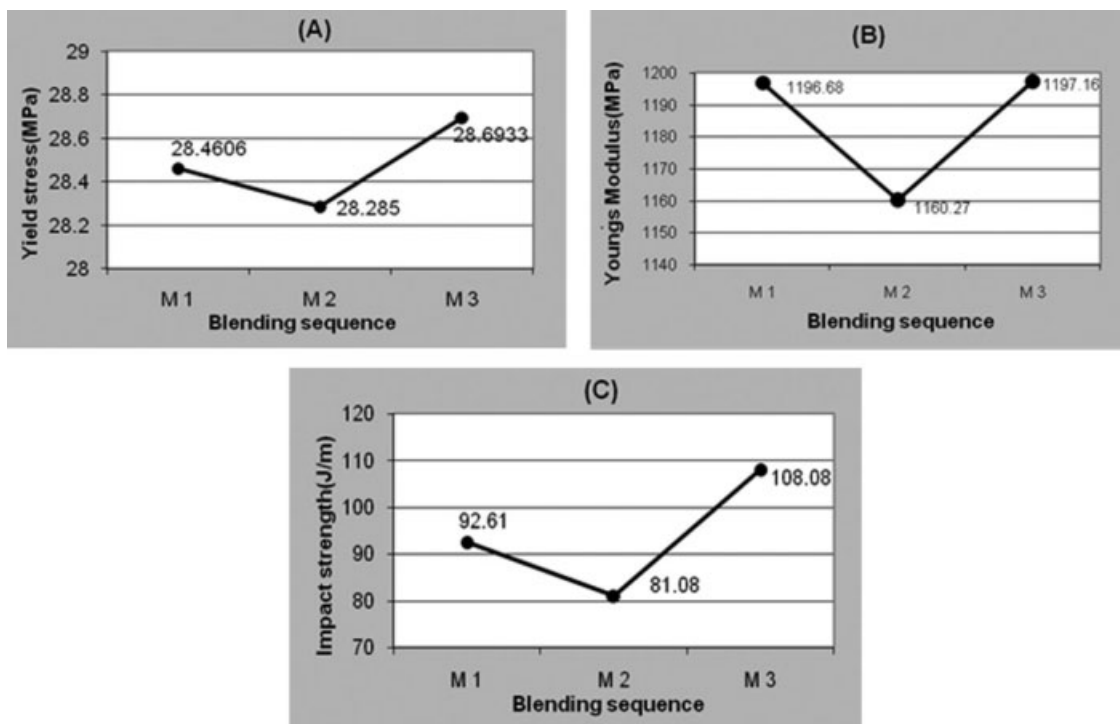


Figure 4 Mean effect of blending sequence on the mechanical properties of PP/PC/SEBS ternary blends (A) yield stress, (B) Young's modulus, and (C) impact strength.

the screw speed. This behavior can be attributed to better droplet breakup and dispersion of dispersed phase with increasing the mixing intensity in twin-screw extruder. Also, this effect is visible in SEM micrographs (Fig. 2) of 1, 6, 8 compounds. As it can be seen in Figure 2, the composite droplet break up is easier in high screw speed. In addition, Figure 4 shows the mean effect of blending sequence on mechanical properties.

As it can be seen in Figure 4(A), the yield stress is slightly higher for M3 blending sequence. This observation can be associated with better dispersion of compatibilizer in matrix and good interfacial adhesion between phases in ternary blend. This may be due to primary dispersion of compatibilizer in matrix and capability of compatibilizer to interfacial bonding with matrix. Figure 4(B) shows that the blending sequence had an optimal effect on Young's modulus. Figure 4(C) demonstrates that the blending sequence has major effect on impact strength. This observation can be attributed to the M3 blending criteria, which the probability of locating the compatibilizer at interface of PP and PC is high because in this protocol the compatibilizer is well dispersed in matrix and the resulted master batch (PP + SEBS-*g*-MAH) was added to disperse phases (PC + SEBS). This probability is very low in blending sequence of M2 because the compatibilizer was mixed with minor phase, and the mobility of compatibilizer toward interface is restricted. SEM test revealed that blending sequence has significant effect on the micro structure of blends and consequent mechanical properties (yield and impact strength). SEM micrographs of 1, 2, 3 compounds in Figure 2 show that in M1 and M3 blending sequence, the particle size of composite droplets is smaller and particle size distribution is finer than that of M2 blending sequence. In M2 blending sequence, the trapped compatibilizer in minor phases cannot migrate to interface and interfacial bonding of matrix and dispersed phases is very poor. In other words, in M3 blending sequence (no. 3, 5, 7 compounds) because of well dispersion of compatibilizer in matrix consequence by addition of master batch (PP + SEBS-*g*-MAH) to the disperse phases (PC + SEBS), the morphology of these compounds is finer with respect to other criteria. In M2 blending sequence (no. 2, 4, 9 compounds), the probability of locating the compatibilizer at the interface of PP and PC is very low because in this case, the compatibilizer would be trapped in disperse phase (PC + SEBS + SEBS-*g*-MAH) first and then this preblend is added to PP matrix.

Optimization of processing conditions

The predicted mechanical properties as a result of Taguchi analysis are shown in different cases in Table IV.

TABLE IV
Predicted Mechanical Properties of PP/PC/SEBS Ternary Blends via Taguchi Analysis

Processing conditions	Mechanical properties		
	Impact strength (J/m)	Young's modulus (MPa)	Yield stress (MPa)
$T = 255^{\circ}\text{C}$ $N = 130$ rpm M3 blending sequence	115.926	1345.32	29.4156
$T = 255^{\circ}\text{C}$ $N = 130$ rpm M1 blending sequence	100.452	1344.83	29.1256
$T = 255^{\circ}\text{C}$ $N = 70$ rpm M1 blending sequence	96.1489	1332.70	29.5572

The predicted results of Taguchi analysis show that the optimum processing conditions to produce the PP/PC/SEBS ternary blend to achieve higher tensile properties and impact strength simultaneously is as follows: $T = 255^{\circ}\text{C}$, $N = 130$ rpm, M3 blending sequence.

CONCLUSIONS

This research is used to highlight the role of processing conditions, namely die temperature, screw speed, and blending sequence on the morphological and mechanical properties of PP/PC/SEBS ternary polymer blends. To obtain this aim, L9 orthogonal array of Taguchi's method is used to analyze the influence of these variables on performance of the PP/PC/SEBS compounds with same composition. The responding variables are impact energy and tensile properties (Young's modulus and yield stress), which are influenced by the morphology of ternary blends. It is shown, based on Taguchi analysis, that the morphology and the mechanical properties are greatly dominated by the processing parameters. It was also found that the high temperature and screw speed was favorable for the improvement of mechanical properties of pure PP. The role of blending sequence on the mechanical properties is associated with their influence on the morphology evolution. The results indicated that the M3 and M1 blending sequence have significant effect on the mechanical properties. Finally, the results based on Taguchi approach confirmed that the optimum processing conditions to achieve simultaneous high impact strength, Young's modulus, and yield strength were as follows: $T = 255^{\circ}\text{C}$, $N = 130$ rpm, blending sequence = M3.

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