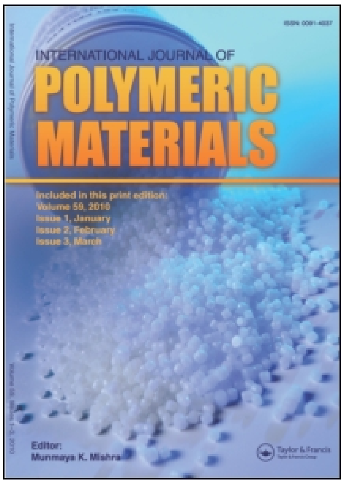

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Influence of Blending Sequence on the Technical Properties and Energy Consumption in Polymer Blending

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INTRODUCTION

Processing is in large part simply flow and forming of compounds and thus the frequent breakdown in production line can be traced to causes like high viscosity or elastic memory of the compounds. Efficient and quality production require reproducibility in the processing stages and it is realised that the rheological or stress deformation properties constitute the most important single factor. Moreover distribution of fillers in the polyblend system exert a great influence on the rheological properties.[1]

The most useful idea for the industrial polymer rheologist in designing the processes and equipments is the dimensional analysis. In this engineering analysis the conservation laws are placed in dimensionless form and the dimensionless group that arise are interpreted. The most important dimensionless parameters are Reynolds number and Power number. Metzner and co-workers[2] have studied the Reynolds number for various pseudoplastic non-newtonian fluids considering the shear rate at the impeller tip. On the basis of viscosity evaluated at the shear rate at the impeller tip an useful generalised correlations[3-6] for power input has been developed by calculating Reynolds number. The total power requirement of a mixer can be calculated from its geometry, dimensions, mode of operation and rheological behaviour of the materials to be mixed.[7-9] Theoretical consideration generally require power increase in proportion to chamber volume.[10-11]

Generally rheological properties are considered sufficient to specify processibility of polymers. Carbon black and other fillers have a great influence on the rheological properties of elastomers.[12-14] For the polyblend systems both the flow and physical properties are dependent on the phase distribution. The presence of fillers in the blends of more than one polymer melt makes the situation further complex because of the relative affinity of the filler towards a particular phase.[1,15] Present study consider the four blend systems based on bromobutyl and EPDM rubber varying in their order of mixing and addition of carbon black. Rheological parameters were studied and energy requirement for blending were considered taking into account the geometry of 11D banbury mixer. Physico-chemical characteristics of the blends were also studied by adding the curatives with special reference to the blending sequence.

EXPERIMENTALS

The four sets of blends, varying in blending sequence, in presence of HAF are given below. The Bromobutyl : EPDM ratio was chosen as 50 : 50 and HAF used was 40 phr.

Blend A: Bromobutyl and EPDM were preblended and kept for 24 hrs at ambient to equilibrate. This preblend is loaded with entire HAF black.

Blend B: The entire black was added to the Bromobutyl phase only and the master batch was allowed to equilibrate at ambient for 24 hrs. Then this black masterbatch was blended with fresh EPDM.

Blend C: The entire black was added to the EPDM phase only and the master batch was allowed to equilibrate at ambient for 24 hrs. This black master batch was blended with fresh Bromobutyl.

Blend D: Half of the black was added to the Bromobutyl phase and half of the black was added to EPDM phase. Both the black master batch was kept at ambient for 24 hrs to equilibriate and then they are blended.

The mixing was done in an open two roll mixing mill (13″ × 6″) identical condition of nip gap (0.075 cm), friction ratio (1 : 1.25) and temperature (50°–60°C). MCR-3210 capillary rheometer was used along with Instron 1195 machine for rheological measurements at four shear rates (30.585 s^{-1}, 305.85 s^{-1}, 611.70 s^{-1} and 1223.40 s^{-1}) at 120°C as described earlier.[1,17] n and K values were determined from the shear stress and shear rate data and apparent viscosities were calculated following the power law equation as

$$\eta = K\gamma^{n-1} \tag{1}$$

considering the system as 11D banbury mixer[18] the Reynolds number N_{Re} can be calculated from the equation[2–4] as

$$N_{\mathrm{Re}} = \frac{d^2 N \rho}{\eta} \tag{2}$$

where d is the diameter of the rotor (0.56 m), N is the speed of the rotor (40 rpm) and ρ is the density of the compound.

For non-newtonian system the shear rate (γ) at the rotor tip can be considered as[2]

$$\gamma = 10N \tag{3}$$

Combining the above equations we got the Reynolds number as

$$N_{\mathrm{Re}} = \frac{d^2 N^{2-n} \rho}{10^{n-1} \times K}$$

Power number (N_P) was calculated from the graphical correlations of N_{Re} and N_P as developed earlier.[2–6] The power input (P) can be calculated from the equation as[4–5]

$$N_P = \frac{P}{\rho N^3 d^5}$$

where N and d are the geometry of 11D boundary as shown above.

The blends are then compounded with the curatives as per formulation ZnO—3.0 phr, MgO—1.0 phr, stearic acid—1.0 phr, sulfur—1.0 phr, MBTS—1.0 phr and TMTD—1.0 phr.

The cure characteristics of the blends were determined in Monsanto rheometer at 150°C. Physical properties were determined on the cured sheet vulcanised upto optimum cure time at 150°C. Abrasion resistance was measured with the help of Du-pont abrader and heat build up was measured with the help of Goodrich flexometer. Resilence was measured with Dunlop tripsometer.

RESULTS AND DISCUSSION

Processibility and cure characteristics

As observed from Table I that the viscosity, both at 100°C and 150°C, of the blend B is lower than the blend C and those for blends

TABLE I

Processing characteristics and Rheometric data (150°C) of the blends

Blend Nos.	A	B	C	D
Min. viscosity (in lb)	24.4	20.5	25.8	24.7
Induction time (ti) (mins)	4.2	4.2	4.7	4.5
Scorch time (t_2) (mins)	5.3	4.8	5.6	5.8
Max. cure (T_{max}) (in lb)	52.0	47.0	50.0	49.0
Optimum cure time (t_{90}) (mins)	36.8	24.7	31.0	38.3
Cure rate $[100/(t_{90} - t_2)]$	3.2	5.0	3.9	3.1
Mooney scorch at 120°C (min)	25.0	21.0	28.0	34.0
ML_{1+4} (100°C)	74.0	67.5	79.0	77.0

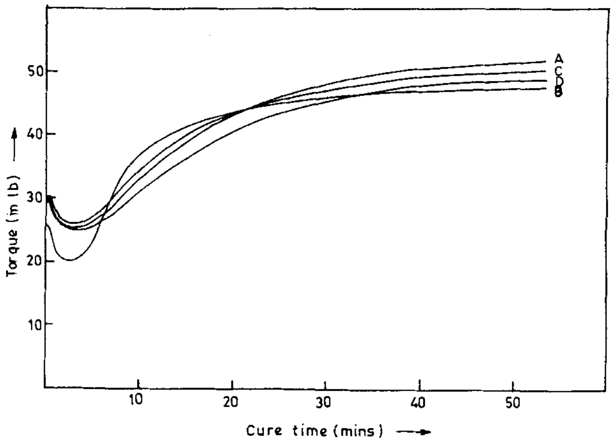

FIGURE 1 Cure characteristics of the blends at 150°C.

A and D lie in between with slight higher viscosity for blend D. The scorch time both at 120°C and 150°C show that the black preferentially in the EPDM phase (blend C) exhibit higher scorch time than the black preferentially in the Bromobutyl phase (blend B). Preblended compound (blend A) gives rise to scorchier compound than the proportionate master batch blending (blend D). Cure characteristics of the blends are represented as graphical plot of torque against curing time in Figure 1. When the entire black is in Bromobutyl phase the optimum cure time is less than when the black is in EPDM phase i.e., blend B is faster curing then the blend C as observed from the cure rate data in Table I. However the blends A and D are having equivalent cure rate.

Physical properties

The physical properties as determined were shown in the Table II. The Table II reveals that the preferential location of carbon black in the EPDM phase gives rise to better tensile, modulus and tear properties than when the carbon black locates itself in the Bromobutyl phase. Hardness is higher with low elongation at break for blend C than the blend B have been observed. The properties of the preblended system (blend A) and blending of 50% black master batches (blend D) lie in between the above two extremes. Abrasion

TABLE II

Physical properties of the blends cured at optimum cure time at 150°C

Blend Nos.	A	B	C	D
Hardness (IRHD)	57.0	54.0	60.0	55.0
Tensile strength (kg/cm^2)	165.0	125.0	176.0	170.0
Modulus (300%) (kg/cm^2)	42.0	22.0	50.0	38.0
Elongation at break (%)	650	750	600	675
Tear strength (kg/cm)	38.0	25.0	42.0	32.0
Abrasion loss (cc/1000 rev.)	0.48	0.54	0.41	0.45
Heat build-up (ΔT°C)	32.0	40.0	30.0	28.0
(%) Resilience)	54.0	52.0	55.0	58.0

loss is better for the blend C then that of blend B. Heat build up is less for the blends having carbon black preferentially in the EPDM phase. The resilience also is in conformity with the heat build observations.

Rheology and power requirement

Rheological parameters were shown in the Table III, along with power requirement. When the entire black is being added to the Bromobutyl phase the non-newtonian index is found to be lower than when the entire black is added to EPDM phase. Higher '*K*' value is observed for the blend C and lower '*K*' value is observed for the blend B. It seems probable that the location of black in the EPDM phase makes the system less pseudoplastic in nature and exhibit plastic like flow. Considering preferred affinity black towards EPDM phase and from the rheological data it may be logical to assume the higher proportion of black in the EPDM phase in the case of preblended system (blend A).

Power requirement have been calculated from the power number and Reynolds number considering the geometry of 11D banbury are shown in the Table III. It is seen in the table that the power

TABLE III

Rheological parameters and power requirement for the blends

Blend Nos.	A	B	C	D
Non-newtonian index (n) at 120°C	0.240	0.214	0.236	0.225
Consistency index ($K \times 10^{-5}$) at 120°C	1.02	0.79	1.23	1.09
Reynolds number (N_{Re} 10^3)	9.53	12.93	7.96	9.18
Power Number ($N_P \times 10^{-4}$)	2.1	1.7	2.6	2.3
Power required (P) *KW*	372.0	301.0	460.0	407.0

required is maximum for the blend C and minimum for the blend B. However those for blend A and blend D lie in between those two extreme with higher power for blend D than for blend A. The preferential location of black in the Bromobutyl phase in blending involve lower power consumption than when the black locates itself in EPDM phase. Whereas distribution of black in both the phases in the blend involve moderate power consumption.

CONCLUSION

The best balanced properties, including the processibility may be achieved for Bromobutyl/EPDM blend by blending the black master batches of the individual elastomers. Although the power consumption, calculated theoretically, is slightly higher for this process but this method probably gives rise to more intimate attachment of the filler particle with each individual elastomer matrix so as to enhance certain required properties of the composite blend.

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