Comparison of Intermeshing Rotor and Traditional Rotors of Internal Mixers in Dispersing Silica and Other Fillers

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ABSTRACT: An experimental study of mixing silica, carbon black, and talc into elastomers in an internal mixer with intermeshing and traditional separated/tangential double-flighted rotors is described. The dispersion in the compound was characterized by measuring the agglomerate size of filler at various mixing times by a scanning electron microscope and image analysis. The distributive mixing was investigated by measuring the incorporation time by a flow visualization technique. The viscosities of compounds were measured in a pressurized rotational viscometer. The intermeshing rotors were found to provide more effective dispersive mixing and their compounds possessed lower viscosities. © 2000 John Wiley & Sons, Inc. J Appl Polym Sci 78: 1551–1554, 2000

Key words: intermeshing rotor; internal mixer; flow visualization; filler agglomerate; silica

INTRODUCTION

The problem of mixing silica into elastomers has attracted considerable attention in the tire industry.[1–5] These studies have involved comparisons of types of particles and surface treatment of silicas. Generally, silica particles are small and possess very strong particle–particle interactions. Silica is much more difficult to disperse than carbon black of equivalent particle size or BET surface area.

There would seem various mechanical methods to overcome the problem of strong particle–particle interaction. This would involve more severe methods of dispersing filler into the polymer. This is the approach of the present paper. The traditional method of incorporating filler into rubber is by using an internal mixer designed by F. H. Banbury,[6–8] which involve two separated (tangential) counterrotating rotors. During the 1930s, R. A. Cooke[9] developed an intermeshing rotor internal mixer where intense deformations and stresses are applied to rubber compounds between the rotors as well as between the rotors and the chamber wall. This design of intermeshing rotor of internal mixer was initially commercialized by Francis Shaw and Company as the Shaw Intermix. The ability of this design to disperse agglomerates in compounds has been praised by the machines' manufacturer. P. S. Kim and White[10,11] have compared laboratory Cooke intermeshing and traditional F. H. Banbury design machines, and found the intermeshing design more effective.

In the present paper, we consider the relative abilities of the traditional nonintermeshing (tangential) rotor machines of F. H. Banbury design and intermeshing rotor machines of R. T. Cooke design to disperse silica in rubber. We also make comparison to mixing carbon black and talc.

EXPERIMENTAL

Material

A styrene–butadiene rubber, Duradene 706 supplied by Firestone, and seven different types of

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Table I Fillers Used in This Experiment

Material	Supplier	Tread Name	BET Surface Area	Code
Silica	PPG	Hisil 190	210	S 190
	PPG	Hisil 255LD	185	S 255
	PPG	Ciptane 255 LD	170	S 255T
	PPG	Silene 732	35	S 732
Carbon black	Cabot	Vulcan 9	143	N 110
	Cabot	Sterling NS1	29	N 762
Talc	Specialty Minerals	Ultratalc 609	16.5	Talc

filler including four silicas were used in the experiments. These are shown in Table I.

Mixing Experiments

The experiments were carried out in a modified Haake Rheocord. This is the same instrument used in earlier flow visualization studies.[10] The detailed design is shown in Figure 1. The intermeshing rotors are shown in Figure 2. The nonintermeshing (tangential) machine is essentially the Haake Rheocord described by Min and White.[12,13] The intermeshing machine contains new rotors and a larger mixing chamber, which have previously been described by P. S. Kim and White.[10,11] The addition of filler into the elastomer was observed and recorded. The incorporation time was determined by the time required to incorporate all the filler into the elastomer.

The compounds were mixed in a Haake Rheocord by intermeshing rotor and tangential rotor for 0.5, 1, 2, 4, 9, and 15 min at 100°C. A fill factor of 0.6 was used. The compound then was broken in the liquid nitrogen. A Hitachi S-2150 Scanning Electron Microscope (SEM) was used to observe the agglomerate size at the fractured surface then the image were taken. The SEM images of agglomerate particles were characterized using an image analyzer. The individual particle diameters were converted to mass average particle diameter. Specifically we have used the "mass" or "$z + 1$" average as shown in eq. (1).

$$D_m = \frac{\sum N_i D_i^3 \times D_i}{\sum N_i D_i^3} \tag{1}$$

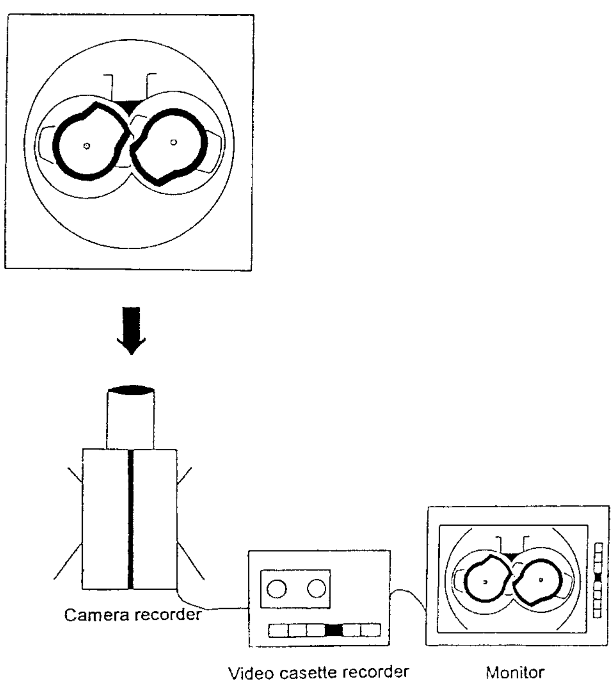

Figure 1 Flow visualization instrument.

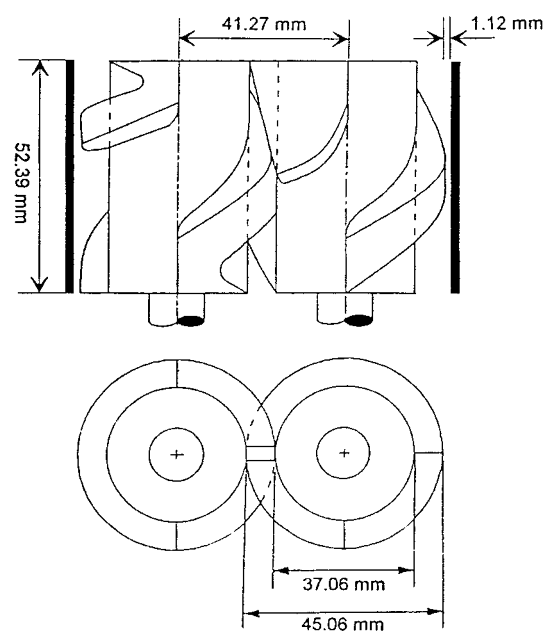

Figure 2 Cooke intermeshing rotor used in the experiments.

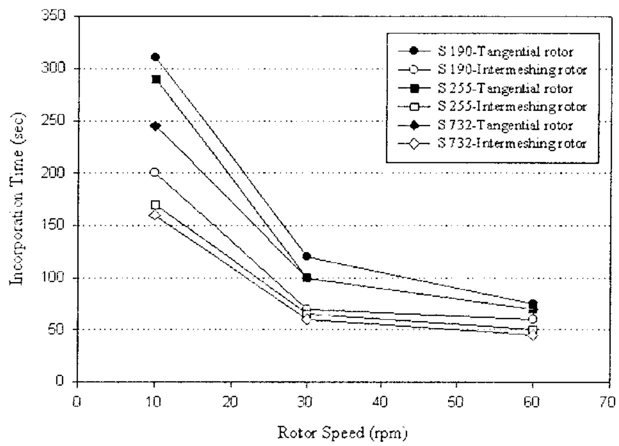

Figure 3 Incorporation time of various silicas in SBR mixed by tangential and intermeshing rotors.

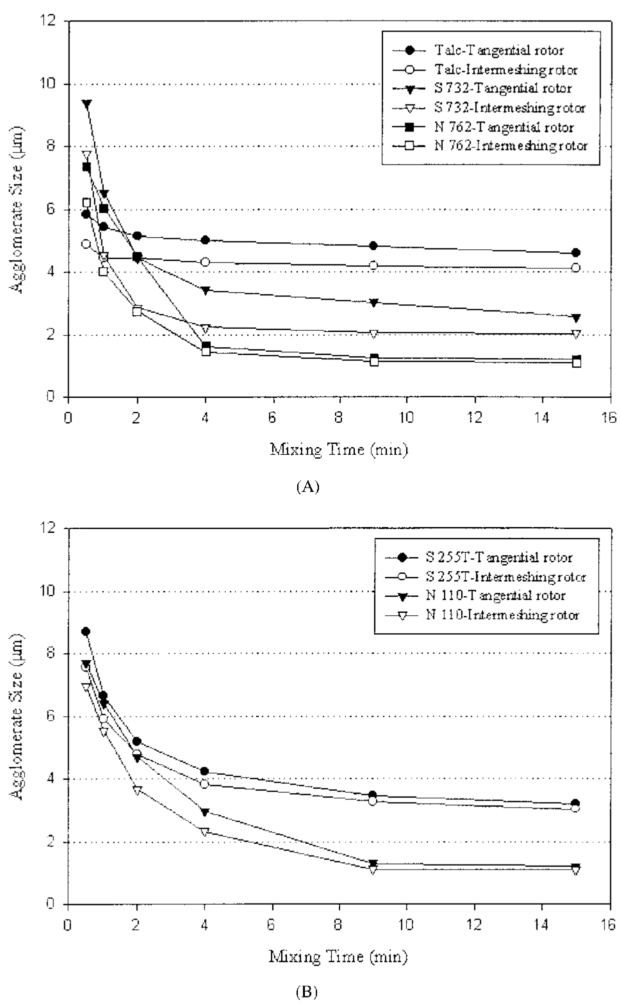

Figure 4 (A) Agglomerate sizes of low BET surface area talc, silica, and carbon black in SBR mixed by tangential and intermeshing rotors. (B) Agglomerate sizes of high BET surface area silica and carbon black in SBR mixed by tangential and intermeshing rotors.

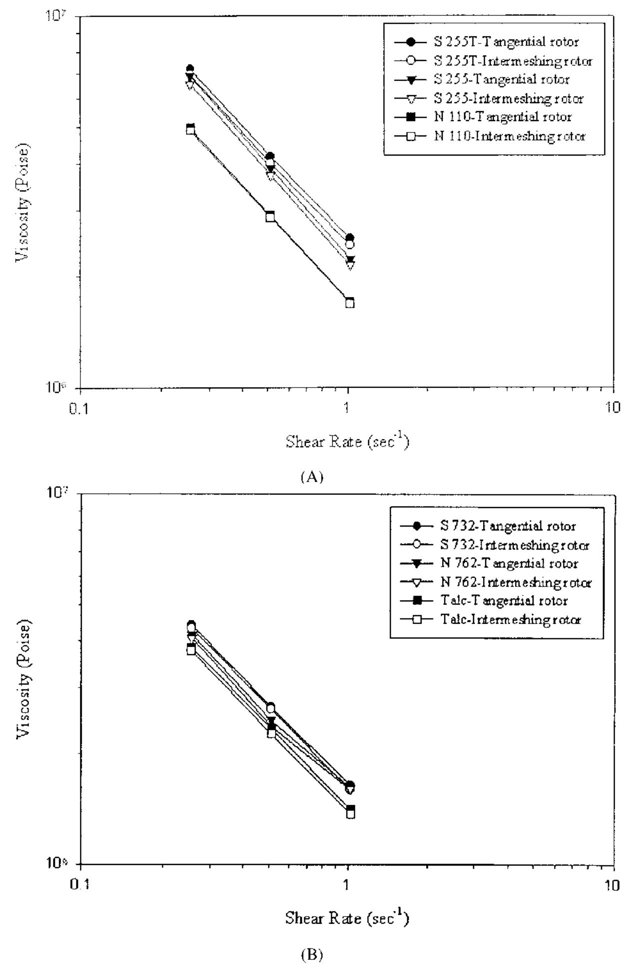

Figure 5 (A) Viscosity of silica and carbon black of SBR compounds mixed by tangential and intermeshing rotors. (B) Viscosity of silica, carbon black, and talc of SBR compounds mixed by tangential and intermeshing rotors.

Rheological Measurement

The shear viscosity was measured in a pressurized variable speed rotational rheometer with a biconical rotor. This instrument has been widely used by many researchers in our laboratory.[14,15] The experiment was run at shear rates of 0.25, 0.5, and 1 s^{-1}.

RESULTS

Mixing

The incorporation times of 10% by volume of filler as a function of rotor speed are shown in Figure 3. The incorporation time is shorter for the intermeshing rotor than for the tangential rotor. Generally, the small particles, high BET surface area, required more time to be incorporated into the elastomer.

Table II Viscosity of SBR Compounds Mixed by Tangential and Intermeshing Rotors

	Viscosity * 10^6											
	S 255T		S 255		S 732		N 110		N 762		Talc	
Sher Rate (s^{-1})	T	I	T	I	T	I	T	I	T	I	T	I
0.25	7.2	6.96	6.88	6.55	4.42	4.34	4.99	4.91	4.18	4.09	3.85	3.77
0.5	4.18	4.01	3.89	3.75	2.66	2.62	2.91	2.87	2.46	2.36	2.33	2.25
1	2.52	2.42	2.21	2.13	1.64	1.6	1.7	1.68	1.62	1.6	1.41	1.37

The influence of mixing time on agglomerate size of talc, silica and carbon black of equivalent BET surface area in SBR is shown in Figure 4(A,B). Low and high BET surface area particles are shown in Figure 4(A) and 4(B), respectively. The agglomerates break up faster in the intermeshing rotor mixer than in the tangential rotor machine, particularly in the beginning of mixing. However, after a period of 4 min, there was a little additional agglomerate breakup. The intermeshing rotor mixer produces slightly smaller agglomerates.

We now compare the different particles. First for the larger particles, talc possesses a larger agglomerate size compared to silica and carbon black. The carbon black has the smallest agglomerate size.

For the small particles, silica had larger agglomerate size than carbon black. Clearly, the agglomerates of silica were more difficult to break than those of carbon black.

Compound Viscosity

The effects of rotor design on viscosity of the compounds having various fillers are shown in Figure 5(A,B) and Table II. It can be seen that the viscosity of compounds mixed by intermeshing rotor is slightly lower than that mixed by tangential rotor.

For both the carbon black and the silica, the viscosity of the compound increases with decrease of filler particle size. At equivalent BET surface area, the silica compounds exhibit higher viscosity than carbon black and talc compound, respectively.

DISCUSSION AND CONCLUSIONS

The intermeshing rotors of R. T. Cooke design are more effective in distributive and dispersive mixing than the tangential rotors of F. H. Banbury design. It can be seen by the flow visualization that the intermesh design circulates and mixes the material at a higher rate because of the kneading action between the rotors, while material is stagnant between the rotors of tangential design. From the analysis of SEM photomicrographs, the agglomerates of compounds mixed by intermeshing rotors were more rapidly dispersed than those mixed by tangential rotors. Among the particles studied, high surface area silica was the most difficult to disperse and incorporate.

The compounds produced by the intermeshing mixer possess lower viscosity than those produced by the tangential mixer. Viscosity levels were higher for the silica than for carbon black at the same particle size. The compound viscosity increased with decreasing particle size.

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