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NOTES

The Flammability of Vulcanized Butadiene–Acrylonitrile Elastomer

The conventional method for mixing elastomers involves the use of solid bulk material and heavy-duty shearing mixers such as the Banbury or Intermix. These mixers rupture the polymer, and a good deal of heat is generated that can cause thermal degradation. The recent introduction of powdered elastomers allows one to mix by a dry blending process that avoids this degradation and also permits more efficient blending of vulcanizing and reinforcing fillers. As part of a program of comparing the properties of elastomers produced by conventional techniques and by dry blending, it was decided to test the flammability of vulcanized nitrile rubber.

EXPERIMENTAL

BREON 452 (a blend of 27% acrylonitrile and 73% styrene butadiene rubbers) was mixed with standard vulcanizing and reinforcing agents either by conventional shear techniques or by powder blending. The powder was converted into sheet on an open mill. Sheet material was obtained from the bulk-mixed polymer by the same technique. All specimens were vulcanized at 155°C for 25 min. Critical oxygen index (*C.O.I.*)[1] was measured on a Stanton Redcroft FTA using specimens 75 mm × 6.5 mm × 0.5 mm, a gas flow of 4 cm/sec, and butane ignition (15 sec, 15-mm flame). *C.O.I.* results were obtained on specimens after exposure to the mineral oil (ASTM No. 3 D471.72) and phosphate esters normally encountered in service.

CRITICAL OXYGEN INDEX

Although the critical oxygen index is primarily a measure of flammability, there are grounds for using it more widely for quality control. The test is rapidly carried out and is fairly simple, straightforward, and reproducible. Since the characteristic measured (flammability) is of vital importance in the application of many rubbers, the critical oxygen index should be considered when one is looking for a laboratory quality control technique. As with all such techniques, it is important that standard conditions are used, for example, a value for the *C.O.I.* of 23.2 obtained when the apparatus was in a fume cupboard rose to 23.7 when the extractor was not in use. Another error that may be found when reinforced vulcanizates are used is that, at the standard gas flow rate of 4 cm/sec, carbon black particles are not fully removed from the chimney but float in the buoyant area at the side of the burning sample and do not disperse through the chimney throughout the duration of each test. Further work on this point is in hand.

RESULTS

The relative amounts of hydraulic fluid absorbed are shown in Table I, along with critical oxygen index values. The dry-blended butadiene–acrylonitrile elastomer (B) absorbs less in each case than the conventional material (A). After contact with mineral oil at 70°C, the powder blended elastomer (B) has in all cases a significantly higher ($P < 0.005$) critical oxygen index, i.e., a lower flammability than the bulk elastomer (A). After immersion in phosphate ester, the dry-blended vulcanizate again has lower flammability than the bulk material. After prolonged immersion in the phosphate ester, both specimens contained high proportions of the ester, and the difference is not so marked. If all the phosphate results are taken together, the higher value of *C.O.I.* for the powdered polymer is significant at $P \doteqdot 0.1$. It is evident that, after removal of surplus fluid, the *C.O.I.* is not significantly changed on standing in air. The dry-blended vulcanizate has a significantly ($P < 0.005$) lower flammability than the bulk material before any absorption occurs.

TABLE I
Critical Oxygen Index Values for Breon Nitrile Rubber 452 After Immersion in Hydraulic Fluids

			ASTM Oil No. 3 168 hr 70°C[b]		Phosphate ester 168 hr 22°C		Phosphate ester 168 hr 70°C		Not immersed	
					Fluid Absorbed, %					
		A	7.6	S.D. 0.32	85.0		96.9			
		B	7.5	S.D. 0.12	64.0		84.1			
					Critical Oxygen Index (C.O.I.)					
Hours in air at 22°C	2	A	21.21	S.D. 0.08	22.93	S.D. 0.82	23.09	S.D. 0.09	21.7	S.D. 0.15
after immersion:		B	21.98	0.11	23.48	0.42	23.15	0.05	23.45	0.11
	336	A	21.33	0.11	22.93	0.82	23.18	0.09		
		B	22.16	0.06	23.46	0.66	23.26	0.08	—	
	840	A	21.38	0.11	23.01	0.13	23.19	0.1	—	
		B	22.33	0.11	23.5	0.87	23.33	0.04	—	

[a] A = Conventional mixing; B = dry blending; S.D. = standard deviation.
[b] Mean of 12 results.
[c] *C.O.I.* are means of four experiments.

DISCUSSION

It is clear that the use of dry-blended powdered butadiene–acrylonitrile rubber gives a material with lower flammability, both alone and after immersion in various hydraulic fluids, than the conventional product. This improvement is attributed to the fact that the dry blending does not impose the same mechanical shear nor internal build-up of heat that shearing mixing imposes, whether this is done on open mills, Banbury, or Intermix equipment. This extra shearing action will reduce the molecular weight of the elastomer; molecular weight is clearly inversely correlated with flammability. The dry-blending process gives a more uniform product, resulting in more efficient vulcanization which leads to a lower absorption of the hydraulic fluid. It is interesting to note that the nonflammable phosphate ester gives little reduction in the flammability of the vulcanizate. Further work on this point is in progress.

Conclusions

Dry blended butadiene acrylonitrile rubber is less flammable, and absorbs less hydraulic fluid, than the conventionally compounded polymer. The critical oxygen index is a useful quality control technique, when used with the appropriate safeguards, for the quality control of vulcanizates.

References

1. ASTM D2863-70, American Society for Testing and Materials, Philadelphia, Pa., 1973

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