

Properties of Silica/Clay Filled Heavy-Duty Truck Tire Thread Formulation

O. J. Ogbemor,¹ A. S. Farid,² U. N. Okwu¹

¹Rubber Research Institute of Nigeria, PMB 1049 Benin City, Nigeria

²School of Polymer Technology and Engineering, London Metropolitan University, 166–220 Holloway Road N7 8DB London, United Kingdom

Received 25 July 2003; accepted 24 February 2004

DOI 10.1002/app.20921

Published online in Wiley InterScience (www.interscience.wiley.com).

ABSTRACT: A tire thread formulation for heavy-duty trucks containing SBR/BR rubber blend and varying proportions of silica/clay fillers including a silane-coupling agent have been investigated. The various mixes were compounded in a Banbury 'O' mixer and vulcanized using the EV-system. Silica/clay (80/0) served as the control mix. The oscillating disc rheometer (ODR) was used in determination of cure characteristics. Substitution of silica (80 phr) with china clay up to 40 phr increased the cure rate of the rubber blend mixes as well as their maximum torque level (T_{\max}). T_{\max} was observed to be highest at a filler blend ratio of 40/40 phr. Synergism between silica and clay at this filler blend mixture is suggested to be responsible for the obser-

vation. The heat buildup was reduced from 43 to 20°C as the clay content increased. Results also showed that the rubber blend compound containing silica/clay (60/20) filler blend in the stated ratio exhibited the best balance of properties in the critical parameters such as the absolute torque level (69.5 dNm), heat buildup (39°C), and abrasion resistance (0.574 mg.loss/1,000 rev). The rate of depreciation of abrasion resistance of rubber blend compound as the clay content increased was found to be 0.035 mg loss/1,000 rev as silica is substituted with one part of china clay phr. © 2004 Wiley Periodicals, Inc. *J Appl Polym Sci* 94: 1024–1028, 2004

Key words: filler; tire; rubber; blends; mechanical properties

INTRODUCTION

In modern heavy-duty truck tires of radial construction, the majority of pre-cure thread formulations are based on SBR or SBR/BR blends as these have been found to exhibit wear performance under low severity (highway) conditions.¹

Increasing attention is given to the use of precipitated silica as a reinforcing filler.² However, its high cost in relation to other fillers is of concern to the industry. In this study, clay is used in a blend with silica both as a cheaper filler and as heat buildup depressant. Though the use of clays in the tire industry is relatively limited compared to carbon black, it has been reported^{3–5} to have been used in tire sealant and rubber lubricant formulations for curing tires.

The objective of this work, therefore, is to determine the influence of varying blend ratios of silica and clay fillers on the physical properties of tire thread compound.

EXPERIMENTAL

Mixing

Five mixes were prepared as shown in Table I. Mix 1 served as control. Mixing was carried out in a Banbury 'O' internal mixer (volume chamber 1,850 cc). Initial temperature of the mixer was set at 80°C.

The mixing involved a two-stage operation as follows:

1. An initial incorporation of SBR, BR, ½ silica/china clay, ½ silane, ZnO, stearic acid, oil, and wax for 1.5 mins, followed by the addition of ½ silica/clay ½ silane and 6 PPD for a further 2.5 min.
2. Final dump of mix onto two-roll mill. Mix temperature at dump was 160°C. This was allowed to cool to 80°C before addition of CBS, DPG, and sulfur on the two-roll mill for 2 min 20 s.

Cure behavior

The aptitude to vulcanization of raw stocks was determined on a Monsanto Rheometer (model R 100S) using a 3° rotor oscillating amplitude and frequency of 1.67 Hz. The cure rate index (CRI) and other parameters characteristic of the cure were estimated from rheographs of the respective compounds.

Correspondence to: O. J. Ogbemor (ojo0001@yahoo.com).

TABLE I
Mix Formulation for the Heavy-Duty Truck Tire Thread

Ingredients	Mix No.				
	1	2	3	4	5
SBR/BR ^a	75/25	75/25	75/25	75/25	75/25
Silica/clay ^{b,c}	80/0	60/20	40/40	20/60	0/80
Silane	7.0	7.0	7.0	7.0	7.0
Aromatic oil	32.5	32.5	32.5	32.5	32.5
Stearic acid	1.0	1.0	1.0	1.0	1.0
Zinc oxide	2.5	2.5	2.5	2.5	2.5
Wax	1.5	1.5	1.5	1.5	1.5
6PPD ^d	2.0	2.0	2.0	2.0	2.0
Sulphur	1.4	1.4	1.4	1.4	1.4
CBS ^e	1.9	1.9	1.9	1.9	1.9
DPG ^f	2.0	2.0	2.0	2.0	2.0

^a SBR (solution SBR), BAYER AG, BR (KUMHO, equivalent of Takzene 1220).

^b Silica, Zeosil 1165 MP from BAYER.

^c Clay, China clay - supreme.

^d 6PPD©, C6 *p*-phenylene diamine.

^e CBS, cyclohexyl benzothiozole sulfenamide.

^f DPG diphenyl guanidine. (All chemicals were supplied by EEC International (Sales) Ltd., UK.

Vulcanization procedure

Compounded slabs were compression molded at 150°C for 40 min using a steam heated hydraulically operated press.

Characterization

The cure properties⁶ as well as mechanophysical characteristics⁷⁻¹⁰ were determined and evaluated in accordance with British Standard Methods of Testing Vulcanized Rubbers.

RESULTS AND DISCUSSION

The properties of the heavy-duty tire thread containing varying filler blend composition (Table I) are shown in Table II.

Cure curves obtained using the oscillating disc rheometer (ODR)⁶ are shown in Figure 1. Wolf¹¹ has stated that ODR is a convenient technique for the evaluation of fillers for their reinforcing potentials. The all-silica filled compound (mix 1 and control sample) had the highest minimum torque ($T_{min} = 27$ dNm). T_{min} for compounds loaded with silica/clay

TABLE II
Properties of Heavy-Duty Truck Tire Formulations with Varying Filler (Silica/Clay) Blend Composition^a

	Mix number				
	1	2	3	4	5
Silica/clay	80/0	60/20	40/40	20/60	0/80
Rheometer at 150°C (arc + 3)					
Maximum torque, T_{max} (dNm)	75.0	75.5	78.5	63.9	52.0
Minimum torque, T_{min} (dNm)	27.0	15.1	9.2	7.0	6.0
ΔT	48.0	60.4	69.3	56.9	46.0
Optimum cure, t_{90} (0.9 ΔT)	43.2	54.4	62.4	51.2	41.4
Absolute torque level ($T_{90} + T_{min}$)	70.2	69.5	71.6	58.2	47.4
Cure time (min)	13.5	9.3	9.8	9.3	9.1
Scorch time, t_{s1} (min)	3.8	3.6	3.7	2.1	2.4
Cure Rate Index, CRI	10.31	17.54	16.39	13.89	14.93
Vulcanizate properties: cured at 40 min/150°C					
Hardness, IRHD	70.6	66.0	64.3	54.3	48.7
M100 (Mpa)	3.2	3.0	3.0	2.4	2.2
M200 (Mpa)	6.5	6.2	6.0	5.4	3.8
TS (Mpa)	11.84	11.80	10.09	9.75	8.2
Elongation at break (%)	320	360	410	400	310
* Temp. rise °C	43	39	31	23	20
Akron abrasion mg. loss/1,000 rev.	0.0083	0.574	1.352	2.128	2.776

^a **Tread mix:** Rubber [SBR/BR (75/25) blend] 100 : silane 7.0; aromatic oil 32.5.

* Heat (Goodrich flexometer) buildup.

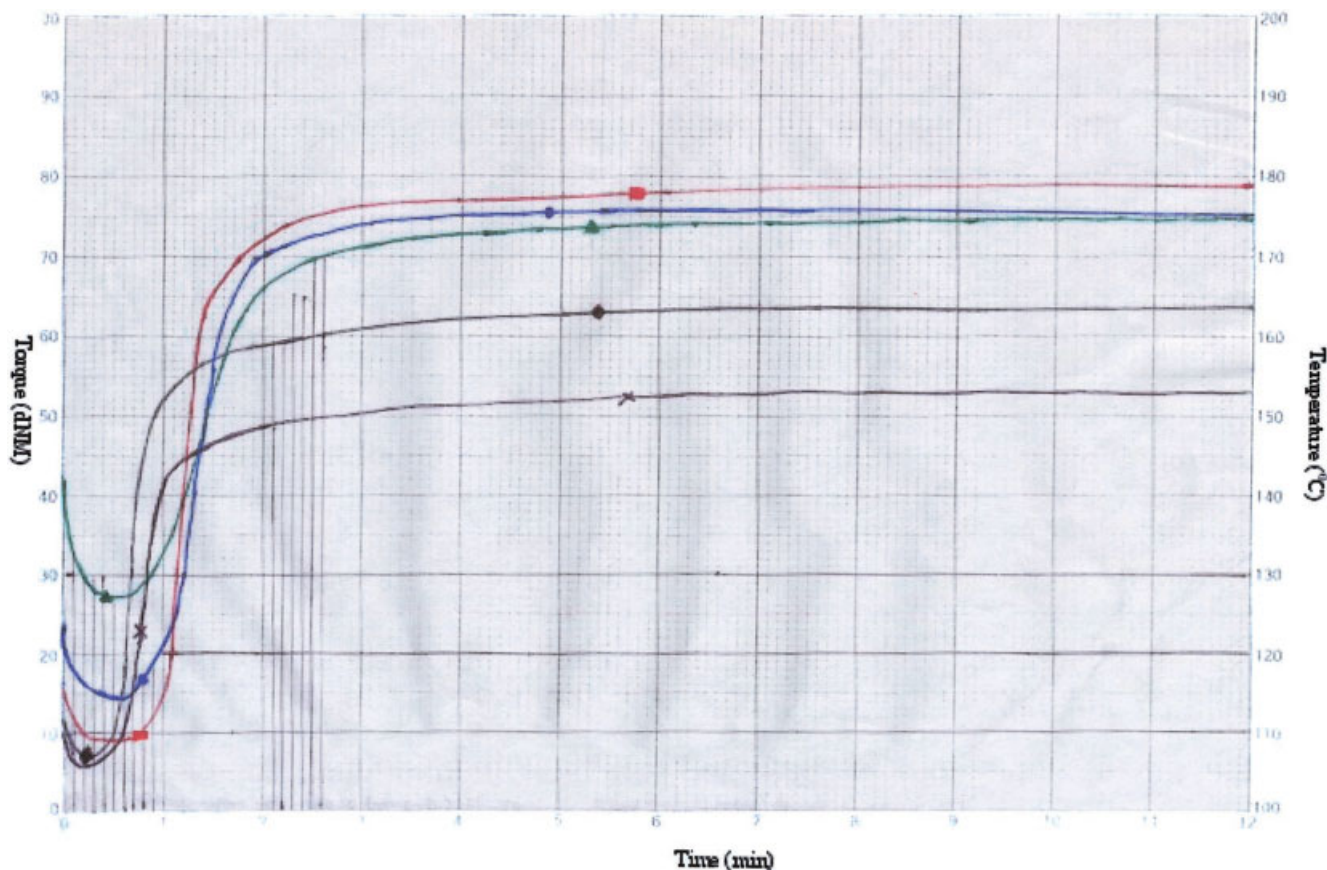


Figure 1 Rheographs at 150°C (arc 3) of varying filler blend compositions. ×, Silica/clay 0/80; ♦, silica/clay 20/60; ▲, silica/clay 80/0; ●, silica/clay 60/20; ■, silica/clay 40/40.

(60/20), silica/clay (40/40), and silica/clay (20/60) were 15.1, 9.2, and 7.0 dNm, respectively. Since T_{\min} is a measure of effective viscosity of unvulcanized mix, the results show that stiffness as well as the viscosity of the uncured compound is reduced as the clay component of the blend is increased.

Silica is known to exhibit a cure retarding effect when incorporated as filler in mixes cured using the efficient vulcanization system.¹² The extent of vulcanization is measured by maximum torque (T_{\max}). Substitution of silica (80 phr) with china clay up to 40 phr increased the cure rate of the rubber blend mixes as well as their T_{\max} . T_{\max} was observed to be highest (78.5 dNm) at a blend ratio of 40/40 phr. Synergism between silica and clay at this filler blend mixture is suggested to be responsible for the observation (Fig. 2). Synergistic reactions are observed in combinations of antioxidants and in combination of carbon black filler with certain antioxidants. For example, mercapto-benzimidazole is of special interest as a synergist for other antioxidant types.¹³ It is also thought¹⁴ that some network bound antioxidants such as those containing sulfur-bearing phenols¹⁵ and prepared via free radical mechanism are autosynergistic.

The all-silica mix exhibited the characteristic reinforcing property of silica fillers (Table II). In general,

the tensile properties were found to decrease as silica is substituted with china clay.

Tires are continuously stressed and abraded when in contact with road surfaces in service life. During this cyclic process, part of the energy is dissipated as unwanted heat (heat buildup). Thus, they are compounded to resist wear and this property of tires, known as abrasion resistance,⁹ is of immense importance. Similarly, heat buildup¹⁰ must be kept as low as possible to guarantee good performance and appreciable longevity. The substitution of silica with china clay was observed to have improved heat buildup properties: the temperature rise in °C (measured using a Goodrich flexometer¹⁰) was reduced from 43°C for silica/clay (80/0) to 20°C for silica/clay (0/80). On the other hand, abrasion resistance deteriorated with an increase in the blend ratio of china clay in the aforementioned filler blend spectrum from 0.083 to 2.776 mg loss/rev. These results from the study clearly show that, while the substitution of silica with china clay improved the heat buildup in polymer compound, it had an adverse effect on the abrasion resistance. Thus, in trying to minimize excessive heat buildup through the substitution of silica with china clay, care should be taken to avoid sacrificing its abrasion resistance, which is a vital property of truck tires.

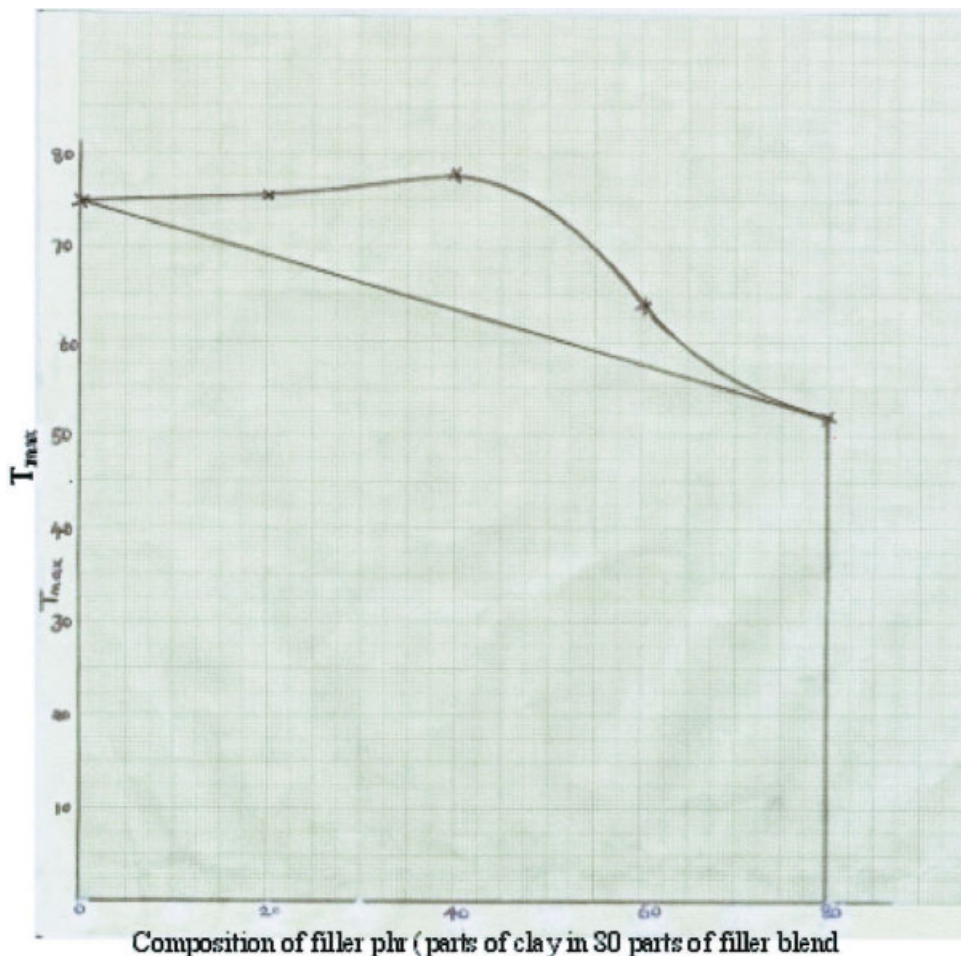


Figure 2 Synergistic effect of silica/clay filler blend on T_{max} .

The use of precipitated silica in truck tires has been reported¹⁶ to improve abrasion resistance on bad roads. The particle size of china clay is larger than that of silica; hence the decrease in abrasion resistance as the blend ratio of china clay is increased. As the particle size of fillers in a polymer compound is decreased, its resilience is decreased while the abrasion resistance and heat buildup are increased.¹⁷ Heat buildup depends on hysteresis and resilience, but in a complex way, since these properties determine the heat-generation behavior.¹⁸ In general, fillers, which increase the hysteresis energy of rubber compounds, such as silica, increase heat buildup. By inference and based on results of this study, increased in the filler blend ratio of china clay could be said to have lowered the hysteresis energy of polymer compound, hence the reduction in heat buildup.

We estimated the rate of depreciation of abrasion resistance of the polymer blend compounds as the level of incorporation of china clay was increased. This was achieved by plotting a graph of abrasion loss in the various mixes versus filler loading (Fig. 3). The resulting slope (0.035), obtained from the line of best

fit, was a measure of the rate of abrasion loss (in mg/1,000 revolutions) as silica is substituted with one part of china clay phr.

CONCLUSION

Of the five filler blend mixes that were incorporated in the polymer blend and studied, the silica/clay (60/20)-loaded compound exhibited the best balance of properties in the critical parameters such as the absolute torque level (69.5 dNm), heat buildup (39°C), and abrasion resistance (0.574 mg loss/1,000rev). China clay has acted as both abrasion resistance and heat buildup depressant in the silica/clay filled polymer composite.

The financial support of the Common Funds for Commodities (CFC), Amsterdam through the Professional Association of Natural Rubber in Africa (ANRA) in Abidjan, Cote d'Ivoire and the late Dr. A. B. Fasina are gratefully acknowledged as well as the technical assistance of the technical staff of the Polymer Science and Engineering Department of the London Metropolitan University.

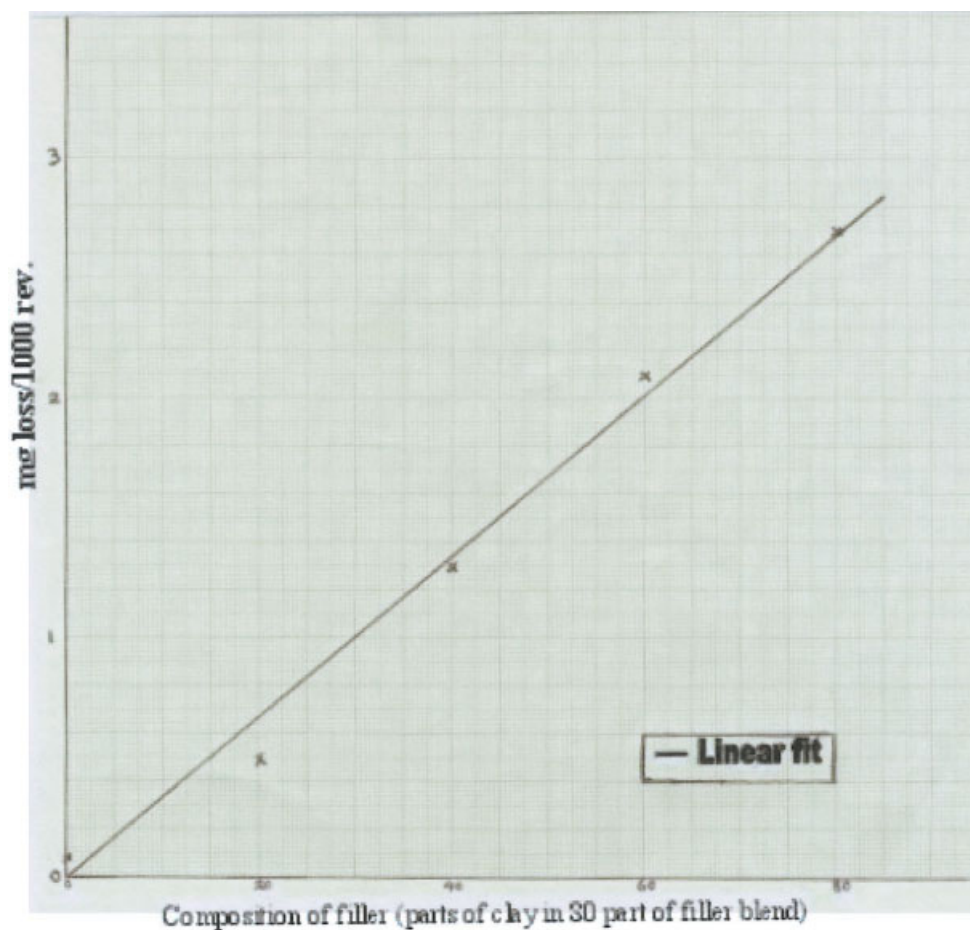


Figure 3 Effect of silica/clay filler blend on abrasion properties of neoprene vulcanisate.

References

- Baker, C. S. L.; Wallace, I. R. *J Nat Rubb Res Malaysia* 1986, 1, 270.
- Hewitt, N. *Compounding with Non-black Fillers*; Educational Symposium 4; Presented at the Rubber Conference, ACS, Cleveland, Ohio, 1979.
- Ilenbeck (to Goodrich). Patent EP 037946 A2 (10/21/81).
- Kihn, J.; Dobson, R. L. (to Goodyear Tire and Rubber Co.), EP 161201 A (1985).
- Gonzalez-Hernandez, L.; Santiago, V. Patent ES 2009241 A6 (9/16/89).
- ISO 3417 (1991 -E) Rubber -Measurement of Vulcanisation Characteristics with Oscillating Disc Curemeter.
- British Standards Institute. *British Standard Methods of Testing Vulcanised Rubber BS 903; Part A2: Determination of Tensile Stress-Strain Properties*, 1989.
- British Standards Institute. *British Standard Methods of Testing Vulcanised Rubber BS 903; Part A57: Determination of Hardness*, 1995 (ISO 7619).
- British Standards Institute. *British Standard Methods of Testing Vulcanised Rubber BS 903; Part A9 Determination of Abrasion Resistance*, 1988.
- British Standards Institute. *British Standard Methods of Testing Vulcanised Rubber BS 903; Part A 50: Determination of Heat Build-up*, 1984; (ISO 4666/3 -1982).
- Wolf, S. *Kautschuck GummiKunststoffe* 1969, 22, 367.
- Wagner, M. P. *Rubber Chem Technol* 1976, 49, 741.
- Blow, C. M. *Rubber Technology and Manufacture*; Butterworths: London, 1971; p. 214.
- Brydson, J. A. *Rubber Chemistry*; Applied Science: London, 1978.
- Scott, G. Paper presented to the International Rubber Conference; Brighton, England, 1997.
- Davies, K. L.; Lionnet, R. *Rubbercon* 1981, 2, G41.
- Blow, C. W. *Rubber Technology and Manufacture*; Butterworths: London, 1971; p. 186.
- Southern, E. In *Development in Rubber Technology -1: Principles of Product Design*; Whelan, A.; Lee, K. S., Eds.; Applied Science Publishers Ltd.: London, 1979; Chap 8.