

Journal of Elastomers and Plastics

<http://jep.sagepub.com>

Linseed Oil as a Multipurpose Ingredient in NBR Vulcanizate

V. Nandanam, Rani Joseph and D. J. Francis

Journal of Elastomers and Plastics 1996; 28; 326

DOI: 10.1177/009524439602800404

The online version of this article can be found at:
<http://jep.sagepub.com/cgi/content/abstract/28/4/326>

Published by:

 SAGE Publications

<http://www.sagepublications.com>

Additional services and information for *Journal of Elastomers and Plastics* can be found at:

Email Alerts: <http://jep.sagepub.com/cgi/alerts>

Subscriptions: <http://jep.sagepub.com/subscriptions>

Reprints: <http://www.sagepub.com/journalsReprints.nav>

Permissions: <http://www.sagepub.com/journalsPermissions.nav>

Linseed Oil as a Multipurpose Ingredient in NBR Vulcanizate

V. NANDANAN, RANI JOSEPH AND D. J. FRANCIS*

*Department of Polymer Science and Rubber Technology
Cochin University of Science and Technology
Kochi 682 022, India*

ABSTRACT: Linseed oil is used in the dual roles of the plasticizer and the fatty acid component of the activator in nitrile rubber vulcanizates. The study shows that the substitution can substantially improve the mechanical properties in addition to other advantages such as increased cure rate, reduced leachability and reduced compound cost.

KEY WORDS: linseed oil, nitrile rubber, plasticizer, fatty acid component, activator.

INTRODUCTION

VEGETABLE OILS, ESPECIALLY drying oils, and their derivatives have been occasionally used as ingredients in plastics and elastomers. Vulcanized vegetable oil (Factice) is used in elastomers for low temperature flexibility and low hardness [1]. Epoxidised linseed oil is widely used in plastics and rubbers as plasticizer [2,3] and heat stabilizer [4]. It is also used as vulcanizing agent in carboxylated nitrile rubber-ionomer blends [5]. Other derivatives such as linseed oil methyl ester diacetal [6] and linseed oil acetoxy methyl derivative [7] are also used as plasticizers. Linseed oil, as such, is used as plasticizer in PVC [8], in heat resistant neoprene rubbers [9], and in some elastomer blends for cold resistance [10].

*Author to whom correspondence should be addressed.

326 JOURNAL OF **ELASTOMERS AND PLASTICS** Vol. 28 - October 1996

0095-2443/96/04 0326-09 \$10.00 0

© 1996 Technomic Publishing Co., Inc.

This paper reports the use of linseed oil as the plasticizer and also as the fatty acid component of the activator in nitrile rubber vulcanizates.

EXPERIMENTAL

Materials Used

Nitrile rubber: Aparene-NBR-553 (Apar India Ltd.), Linseed oil: Refined linseed oil, marketed by Camlin Ink, India Ltd. The other compounding ingredients used were commercial grade.

Nitrile rubber was compounded on a two roll mixing mill (6" x 12") as per ASTM D 15-627 according to the formulation given in Table 1. In formulations where linseed oil is present, it was used as a substitute for DOP and stearic acid. The optimum cure time t_{90} (time to reach 90% of the maximum torque) and scorch time t_{10} (time to reach 10% of the maximum torque) were determined using a Goettfert Elastograph model 67.85. The compounds were vulcanized up to the optimum cure time in an electrically heated hydraulic press. Test specimens were punched out from the compression moulded sheets, and the tensile properties were determined as per ASTM D 412-80 using a Zwick universal testing machine model 1445. The results reported are the average value of at least five specimens. The tear strength was determined using angle test specimens. The ageing resistance of the sample was determined by determining the retention in tensile properties after ageing the sample at 100°C for 72 hrs. The crosslink density was determined by equilibrium swelling in chloroform for 48 hrs at room temperature, and calculated using the Florey Rehner equation. The oil resistance and leachability of the sample were studied by keeping the sample in castrol engine oil at 70°C for 72 hrs. The processability of the compounds was studied using a Brabender plasticorder.

Table 1. Formulation.

NBR	100 phr
HAF	50 phr
Zinc oxide	5 phr
Stearic acid	2 phr
MBTS	1 phr
TMTD	0.2 phr
Sulphur	2 phr
Antioxidants	1.5 phr
DOP	6 phr

RESULTS AND DISCUSSION

Figure 1 shows the flow curves of the compounds obtained from the Brabender plasticorder as (torque/rpm) representing viscosity and rpm representing shear rate, at different temperatures. All the compounds are highly pseudo plastic. The flow behaviour of the compound containing 5 phr linseed oil closely resembles that of the compound containing 6 phr DOP. It may be concluded that the processability of the compounds in which linseed oil replaces stearic acid and DOP is not adversely affected. In fact, viscosity is marginally lower showing the higher plasticizing action of linseed oil.

Figure 2 shows the cure curves of the compounds. The curing behaviour of the compound containing 5 phr linseed oil and the compound containing 6 phr DOP are very similar, as in the case of their flow curves. The curing becomes progressively faster with increase in the amount of the linseed oil. The cure time is decreased by 15% when

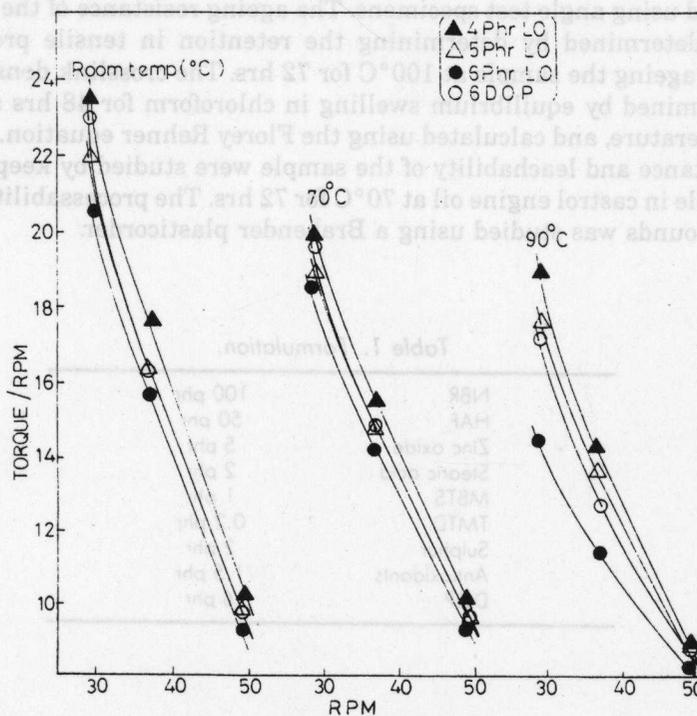


FIGURE 1. Flow curves of the compounds with different levels of linseed oil.

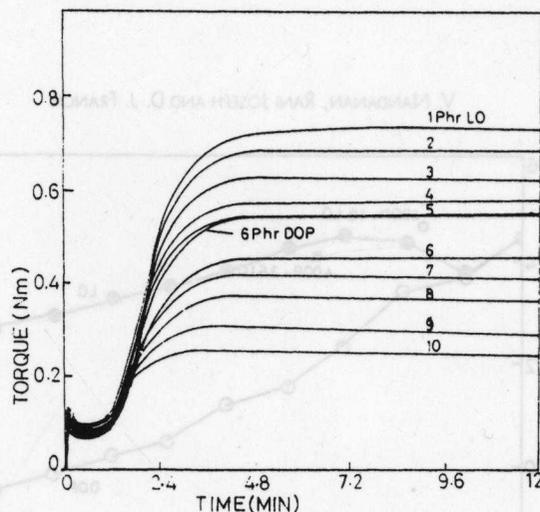


FIGURE 2. Cure curves of compounds with different levels of linseed oil.

DOP is substituted with the same loading of linseed oil. This may be due to the higher activity of unsaturated fatty acids compared to stearic acid. It may be noted that the increase in cure rate does not affect the scorch safety (Table 2).

Figure 3 shows the variation of tensile strength with DOP and linseed oil concentrations. It is interesting to compare the plasticizing

Table 2. Cure characteristics of the compounds.

Sample No.	Composition	Minimum Torque (Nm)	Maximum Torque (Nm)	Scorch Time (t ₁₀) (Min.)	Cure Time (t ₉₀) (Min.)
1	6 phr DOP	0.059	0.553	1.32	2.8
2	1 phr Linseed Oil	0.085	0.726	1.28	2.7
3	2 phr Linseed Oil	0.085	0.677	1.28	2.8
4	3 phr Linseed Oil	0.084	0.620	1.32	2.7
5	4 phr Linseed Oil	0.084	0.571	1.28	2.6
6	5 phr Linseed Oil	0.078	0.554	1.32	2.6
7	6 phr Linseed Oil	0.068	0.499	1.32	2.4
8	7 phr Linseed Oil	0.069	0.434	1.32	2.4
9	8 phr Linseed Oil	0.062	0.369	1.32	2.2
10	9 phr Linseed Oil	0.061	0.291	1.28	2.0
11	10 phr Linseed Oil	0.055	0.254	1.28	2.0

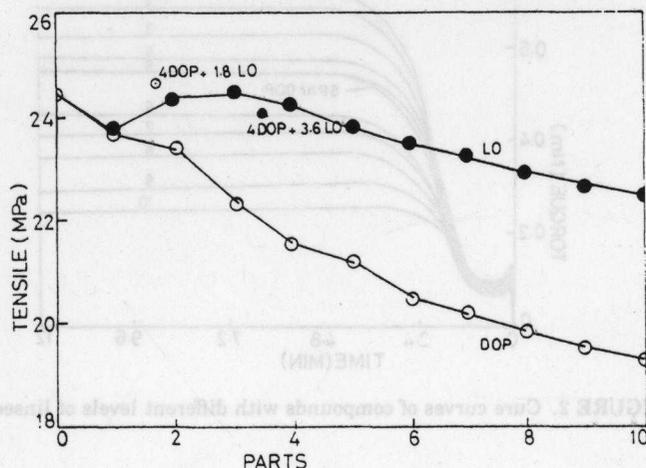


FIGURE 3. Variation of tensile strength with DOP and linseed oil content.

nature of DOP and linseed oil. The tensile strength decreases linearly up to 1 phr level, both for DOP and linseed oil, but thereafter the tensile strength shows a clear deviation. The vulcanizate with linseed oil shows a substantially higher value, up to 4 phr level, and then starts decreasing linearly with linseed oil content. But compounds with linseed oil always show a higher tensile strength than compounds with same loading of DOP. This shows that there is some phenomena other than mere plasticization, and it is likely that the linseed oil gets covulcanized along with the elastomer. The variation in tensile strength can also be related to crosslink density and additionally to better filler incorporation. But as the concentration of linseed oil increases, there is a possibility of the oil getting vulcanized to function as a factice, which will reduce the strength of the vulcanizate. The decrease in crosslink density supports this view (Figure 4) as part of sulphur is used for factice formation. Further, it may be noted that a low phr linseed oil combined with DOP gives the same increase in tensile strength by maintaining other properties.

Figure 5 shows the variation of modulus with linseed oil concentration which is directly proportional to the variation of crosslink density as expected while the elongation at break is inversely proportional to the variation of crosslink density (Figure 6). The variation of tear resistance is similar to tensile strength and can be explained on similar lines (Figure 7).

The air resistance and oil resistance of the vulcanizate containing

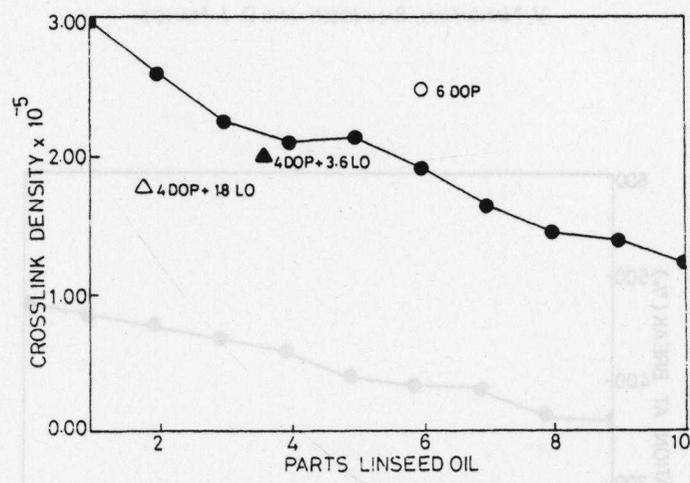


FIGURE 4. Variation of cross link density with linseed oil content.

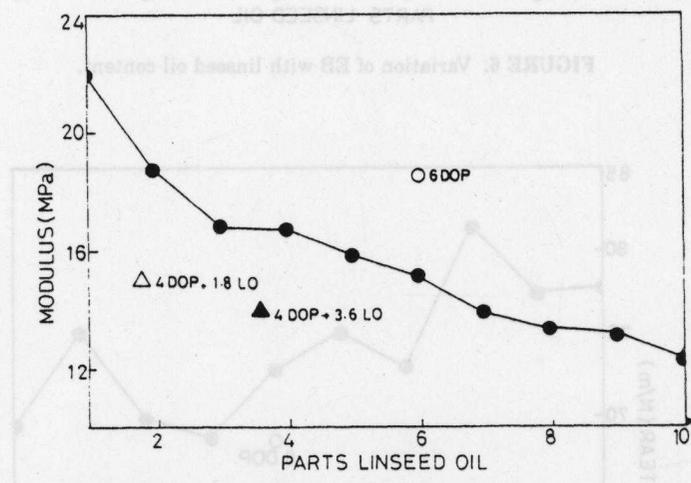


FIGURE 5. Variation of modulus with linseed oil content.

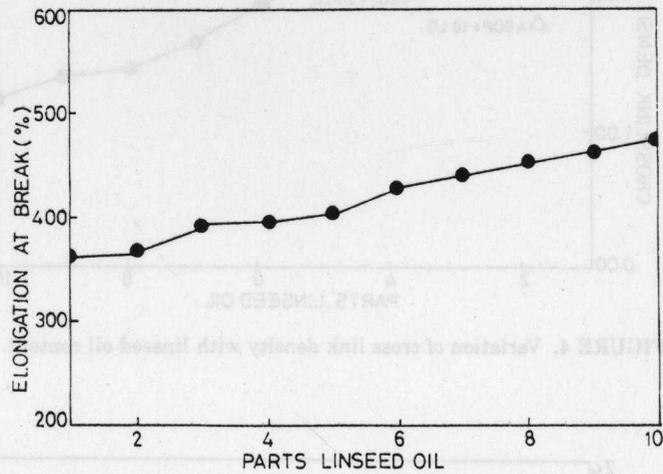


FIGURE 6. Variation of EB with linseed oil content.

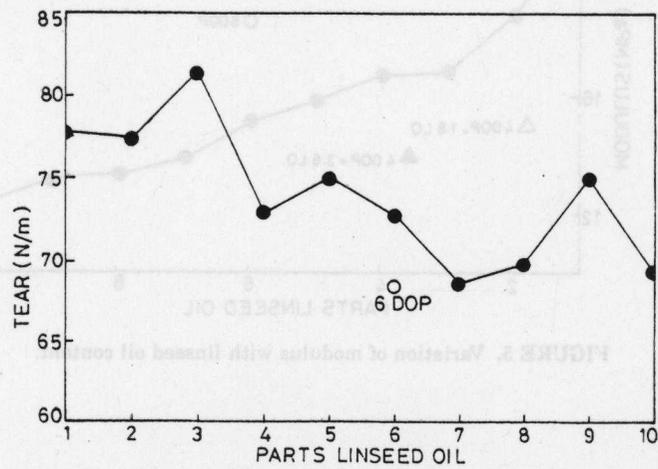


FIGURE 7. Variation of tear resistance with linseed oil content.

Table 3. Tensile properties of the aged and unaged compounds.

No.	Sample Composition	Tensile Strength (MPa)		
		Unaged	Oil Aged	Air Aged
1	6 phr DOP	20.45	17.65	14.56
2	1 phr Linseed Oil	23.78	21.13	17.87
3	2 phr Linseed Oil	24.41	21.56	17.92
4	3 phr Linseed Oil	24.40	21.54	16.54
5	4 phr Linseed Oil	24.15	20.44	16.32
6	5 phr Linseed Oil	23.56	20.37	15.95
7	6 phr Linseed Oil	23.36	21.22	16.09
8	7 phr Linseed Oil	22.97	19.21	17.86
9	8 phr Linseed Oil	22.68	20.70	16.94
10	9 phr Linseed Oil	22.49	18.93	15.06
11	10 phr Linseed Oil	22.17	18.29	15.04

DOP and linseed oil do not show much variation. The standard and reference samples were aged in engine oil at 70°C for 72 hrs and air at 100°C for 72 hrs. The properties of the aged samples were compared with those of the fresh sample (Table 3).

Leachability is considerably reduced by substituting DOP with linseed oil. The percentage loss in weight of oil aged standard and reference samples are given in Table 4. The sample containing 6 phr linseed oil shows only 40% leachability of the one containing 6 phr DOP.

Sulphur blooming is a general phenomena in sulphur vulcanized

Table 4. Leachability of compounds.

No.	Sample Composition	Weight before Oil Ageing (gm)	Weight after Oil Ageing (gm)	% Loss in Wt.
1	6 phr DOP	2.279	2.244	1.535
2	1 phr Linseed Oil	2.573	2.561	0.466
3	2 phr Linseed Oil	2.764	2.759	0.18
4	3 phr Linseed Oil	2.717	2.707	0.368
5	4 phr Linseed Oil	2.676	2.66	0.59
6	5 phr Linseed Oil	2.666	2.648	0.67
7	6 phr Linseed Oil	2.355	2.341	0.59
8	7 phr Linseed Oil	2.311	2.293	0.77
9	8 phr Linseed Oil	2.385	2.367	0.75
10	9 phr Linseed Oil	2.624	2.607	0.64
11	10 phr Linseed Oil	2.467	2.447	0.8

materials. But incorporation of linseed oil shows very little blooming compared to samples containing DOP.

CONCLUSION

Use of linseed oil gives appreciable increase in properties like tensile strength, tear resistance etc. in nitrile rubber vulcanizates. The viscosity of the compound is marginally lower than that of the compound with DOP. Cure rate is increased and leachability is reduced. The optimum concentration of linseed oil seems to be in the range 2-5 phr and this can advantageously replace about 6 phr DOP and 2 phr stearic acid in conventional NBR vulcanizates. Further, the cost of the compound can also be reduced by substituting linseed oil for DOP and stearic acid.

REFERENCES

1. Barlow, F. W. 1993. *Rubber Compounding*, Marcel Dekker Inc.
2. Boccaico, G. 1981. "Collog. Lipochim Ind. Son Avenir Cote.," *Petrochim (Actes)*, pp. 52-60.
3. Abdul B., E. M., B. M. Badran, Khalifa and A. A. Yahia. 1978. *Elastomerics*, 110(11):38-42.
4. Aoki, A., T. Ibaragi, T. Yamada and M. Honda. 7762363 (cl. C08 L53/02), 23 May 1977. Appl. 75/137, 749, (18th Nov. 75):p. 8.
5. Clark, L. 1986. Ger Offen DE 3,738,335 (cl.C08 L 13/00) 26 May 1988, US Appl. 931, 389 (17 Nov. 1986):p. 13.
6. Richard, A. and H. P. Everett. (US Dept. of Agrl.), U.S. Publ. Pat. Appl. B. 485,060. (C.A. Vol. 84, 137575).
7. Frankel E. N. and H. P. Everett. 1976. U.S. Patent, Appl. 609,920, 25 June, 1976, 28 pp. Avail NTIS.
8. Lima, D. A. and J. P. Hamilton. (FMC Corp.), U.S. Patent, 3,481,894 (Cl 260-235 C08 gf). Dec. 2, 1969. Appl. 27 Sept. 1966, p. 3.
9. Kaneshige, Y., K. Sanuki and Y. Nakamoto. 1975. *Toyo Soda Kemkya Hokoku*, 19(1):13-31.
10. Velchova, I., D. Beshdarov and E. Ozgharova. 1989. *Plaste Kautsch.* 36(4):123-126.