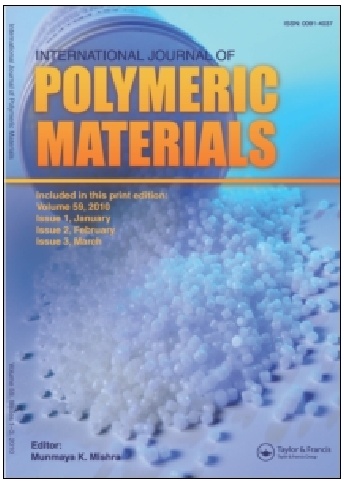

International Journal of Polymeric Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713647664

Palm Oil Fatty Acid Additive (POFA) Filled Natural Rubber Compounds - The Effects of Temperature on Curing Characteristics, Dynamic Properties and Reversion

Hanafi Ismail[a]; U. S. Ishiaku[a]
[a] School of Industrial Technology, Universiti Sains Malaysia, Penang, Malaysia

To cite this Article Ismail, Hanafi and Ishiaku, U. S.(2000) 'Palm Oil Fatty Acid Additive (POFA) Filled Natural Rubber Compounds - The Effects of Temperature on Curing Characteristics, Dynamic Properties and Reversion', International Journal of Polymeric Materials, 46: 1, 61 — 70

To link to this Article: DOI: 10.1080/00914030008054841

URL: http://dx.doi.org/10.1080/00914030008054841

Intern. J. Polymeric Mater., 2000, Vol. 46, pp. 61–70
Reprints available directly from the publisher
Photocopying permitted by license only

Palm Oil Fatty Acid Additive (POFA) Filled Natural Rubber Compounds – The Effects of Temperature on Curing Characteristics, Dynamic Properties and Reversion

HANAFI ISMAIL* and U. S. ISHIAKU

School of Industrial Technology, Universiti Sains Malaysia, 11800 Minden, Penang, Malaysia

(Received 19 August 1998)

The effects of temperature on curing characteristics, dynamic properties and reversion behaviour of palm oil fatty acid additive (POFA 2) filled natural rubber compounds were studied. The scorch time, t_2 and cure time, t_{90} were found decrease with increasing POFA 2 concentration. At similar POFA 2 concentration the t_2 and t_{90} also decreased with increasing cure temperature. For dynamic properties compared to control compound, the compounds with POFA 2 show higher maximum elastic torque but lower minimum elastic torque. At similar POFA 2 concentration, the maximum and minimum elastic torque decrease with increasing cure temperature. Results also indicate that the incorporation of POFA 2 and increment in cure temperature decrease the loss tan delta. Reversion decreased with increasing POFA 2 concentration whereas increasing the cure temperatures exhibits opposite trend.

Keywords: Palm oil fatty acid additive; curing characteristics; dynamic properties; reversion; natural rubber compounds

1. INTRODUCTION

In our previous report [1], the comparison effects of palm oil fatty acid additive (POFA) and commercial processing aids on the properties of

*Corresponding author. Tel.: 04-6577888, ext 2214; Fax: 04-6573678(e-mail: ihanafi@usm.my).

carbon black filled natural rubber compounds were evaluated. Incorporation of POFA reduces the scorch time while commercial processing aids show retardation effect. All POFA and commercial processing aids improve the processability of the rubber compounds. At similar concentration POFA was found to give better mechanical properties compared to commercial processing aids.

In this work, the similar POFA called POFA 2 was used in carbon black filled natural rubber compounds to study the effect of temperature on curing characteristics, dynamic properties and reversion behaviour.

2. EXPERIMENTAL

2.1. Materials, Formulation and Mixing Procedure

Table I shows the formulation used in this work. SMR L was supplied by Rubber Research Institute of Malaysia (RRIM). Other chemicals such as sulphur, zinc oxide, stearic acid, 2,2,4-trimethyl-1,2-dihydroquinoline (Flectol H) and 2-mercaptobenzothiazole (MBT) were purchased from Bayer (M) Ltd. while carbon black, N330 was obtained from Malayan Carbon (M) Ltd. All materials were used as supplied and a conventional sulphur vulcanization system (CV) was employed. POFA 2 was synthesised in our laboratory as previously reported [2]. Mixing was carried out on a laboratory size (160 × 320 mm) two roll mixing mill (Model XK – 160) in accordance to the method described by the American Society for Testing and Materials (ASTM) designated D 3184.

TABLE I Different concentration of POFA 2 in carbon black filled natural rubber compounds

Materials	*phr*
Natural rubber (SMR L)	100.0
Carbon black (N 330)	50.0
Zinc oxide	5.0
Stearic acid	2.0
Sulphur	2.5
MBT	1.0
Flectol H	1.0
POFA 2	0, 1, 2, 3, 4, 5

2.2. Measurement of Curing Characteristics and Dynamic Properties

The MDR 2000 moving die rheometer (MDR), a rotorless curemeter, has gained much acceptance by the rubber industry since its introduction in 1988. In many cases, this instrument is replacing the oscillating disk rheometer (ODR) as described in ASTM standard Test Method D2084. The dynamic properties before, during and after cure were studied at 140°C, 150°C and 160°C. A unique signal processing system and Fourier transform software separates the complex torque into elastic torque (S′) and viscous torque (S″). The tan delta is derived by dividing S″ by S′. In addition to the dynamic properties, the MDR gives digital outputs of curing characteristics such as scorch times, cure times, cure rates and torque values.

2.3. Measurement of Reversion

A Monsanto rheometer, model MDR 2000 was also used to test the rubber compound since at 150°C the rheometer torque is found to be suitable as an indicator of reversion behaviour [3].

Percentage reversion (R) is defined as:

$$R = (T_{\text{max}} - T_t)100/(T_{\text{max}} - T_{\text{min}})$$

where T_{max} = maximum torque, T_t = torque at t minutes and T_{min} = minimum torque on the rheograph.

3. RESULTS AND DISCUSSION

3.1. Effect on Dynamic Properties

The effect of POFA 2 on the maximum elastic torque (S′@MH) and minimum elastic torque (S′@ML) obtained from the MDR 2000 at 140, 150 and 160°C are shown in Figures 1 and 2. It can be seen in Figure 1 that compared to control compound (without POFA 2), compounds with POFA 2 show higher maximum elastic torque. S′@MH generally correlates with durometer hardness and/or modulus. Our previous works [1, 2] indicate that POFA 2 filled rubber have better

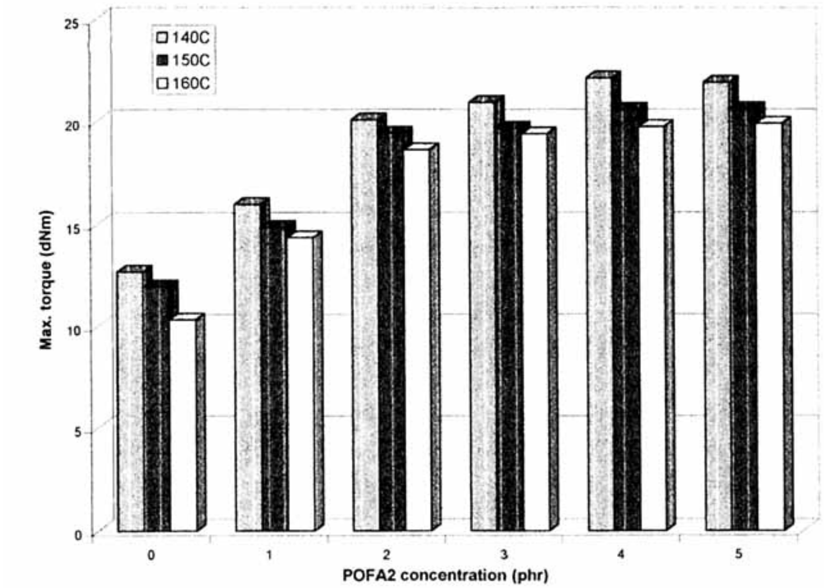

FIGURE 1 The effect of POFA 2 concentration on maximum elastic torque at three different temperatures.

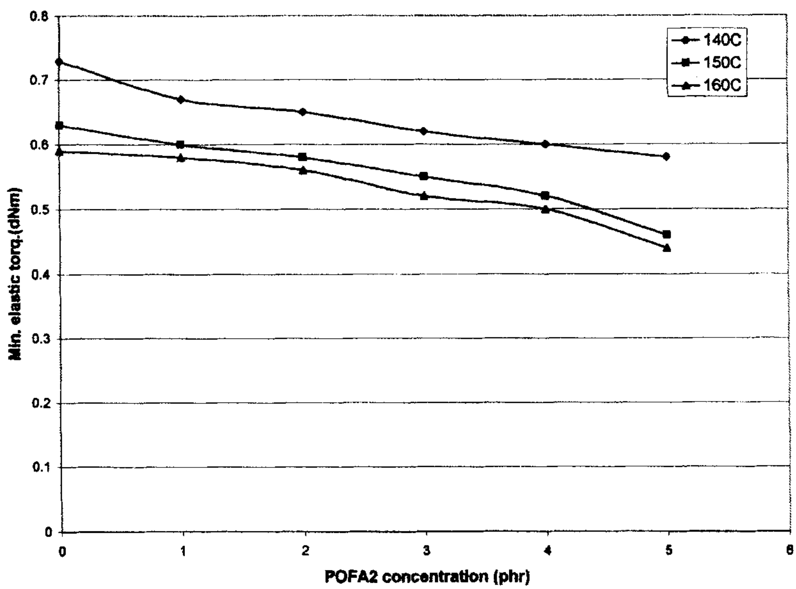

FIGURE 2 The effect of POFA 2 concentration on minimum elastic torque at three different temperatures.

hardness and tensile modulus than the control compound. At a similar POFA 2 concentration, the maximum elastic torque decreases with increasing temperature. This might be due to the softening effect of rubber as the temperature increases. Since in our studies the conventional vulcanization system was used, the reduction of S′@MH was also due to the reversion behaviour of the rubber compounds (see Section 3.3).

Figure 2 shows that the minimum elastic torque, S′@ML decreases with increasing POFA 2 concentration. S′@ML is commonly considered as representative of the uncured stock's elastic modulus and also provide valuable information about a compound's processability [4]. This result indicates that the use of POFA 2 improves the processability of the rubber compounds. As for S′@MH, the S′@ML also decreases with increasing curing temperature of similar POFA 2 concentration.

The torque difference, S′@MH − S′@ML (max. elastic torque-min elastic torque) *versus* POFA 2 concentration at different curing temperatures is shown in Figure 3. It can be seen that the torque difference

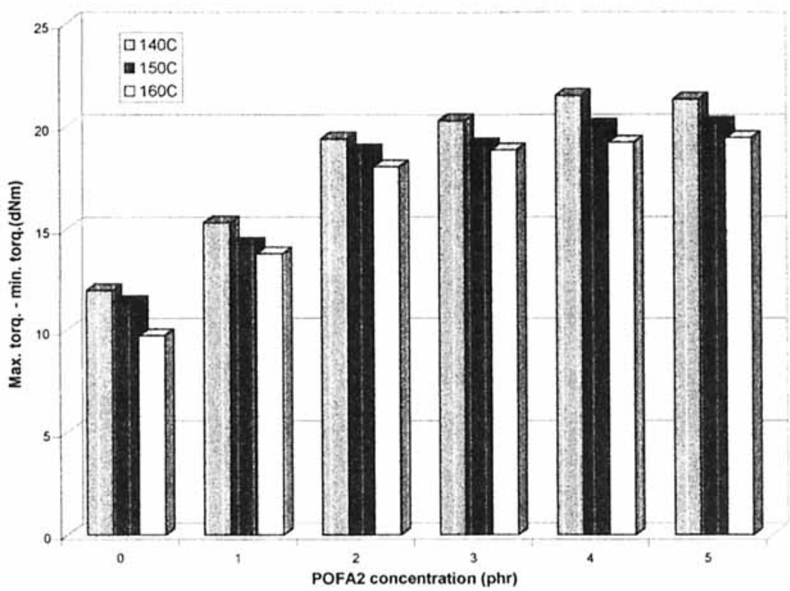

FIGURE 3 The effect of POFA 2 concentration on max. elastic torq. − min. elastic torque at three different temperatures.

increase with increasing POFA 2 concentration. The increment in the torque difference indicates that POFA 2 has an activating effect to cause more efficient use of sulphur for higher degree of crosslinking. As explained before the reduction of torque difference at high temperature was due to softening effect and reversion behaviour (Section 3.3) of the rubber compounds.

Figure 4 shows the effect of POFA 2 concentration on the tan delta @MH of the natural rubber compounds at different curing temperatures. Tan delta relates to the damping characteristics of a rubber compound [5–6]. The lower the tan delta for a cured compound, the greater its resiliency [4]. From Figure 4. It can be seen that compared to the control compound, the incorporation of POFA 2 decreases the tan delta which give beneficial effect to the natural rubber compounds by reducing the degree of damping. Figure 4 also indicates that higher curing temperature resulted in a lower than delta.

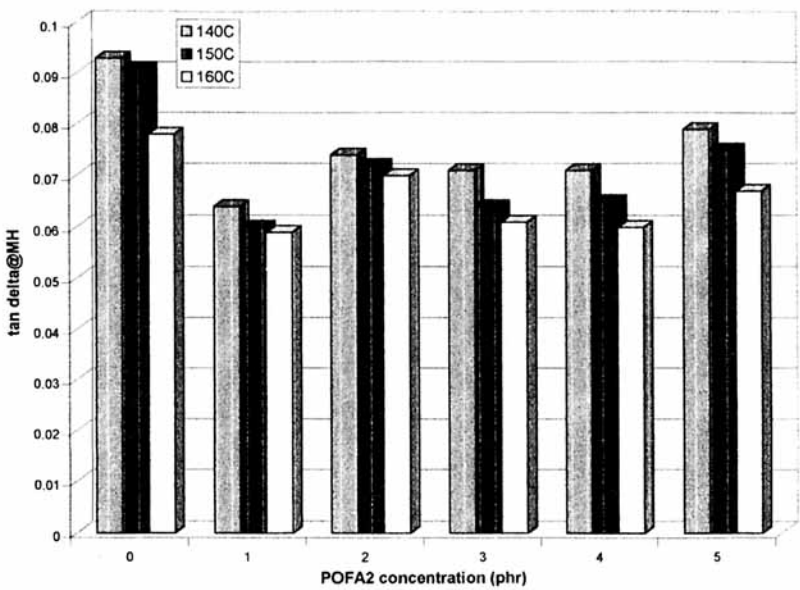

FIGURE 4 Relationship between tan delta and POFA 2 concentration at three different temperatures.

3.2. Effect on Curing Characteristics

Figures 5 and 6 show the effects of POFA 2 concentration on scorch time, t_2 and cure time, t_{90} at different curing temperatures. It can be seen that the t_2 and t_{90} decreased with increasing POFA 2 concentration. As reported before [1 – 2] POFA 2 has nitrogen atoms in the form of diamine. The diamine group has ability to promote the vulcanization of olefinic rubber comprising sulphur and primary accelerators through the formation of complexes which are responsible for the fission of sulphur molecules to form crosslinks between the linear rubber chains.

Figures 5 and 6 also show that at similar POFA 2 concentration, t_2 and t_{90} decreased with increasing temperature. As temperature of curing is increased, there is sufficient thermal energy to cause faster curing. Also, the mobility of rubber chain is increased, and this would mean that the probability for crosslinking is increased. Since the rate of reaction is exponentially dependent on temperature as given by the

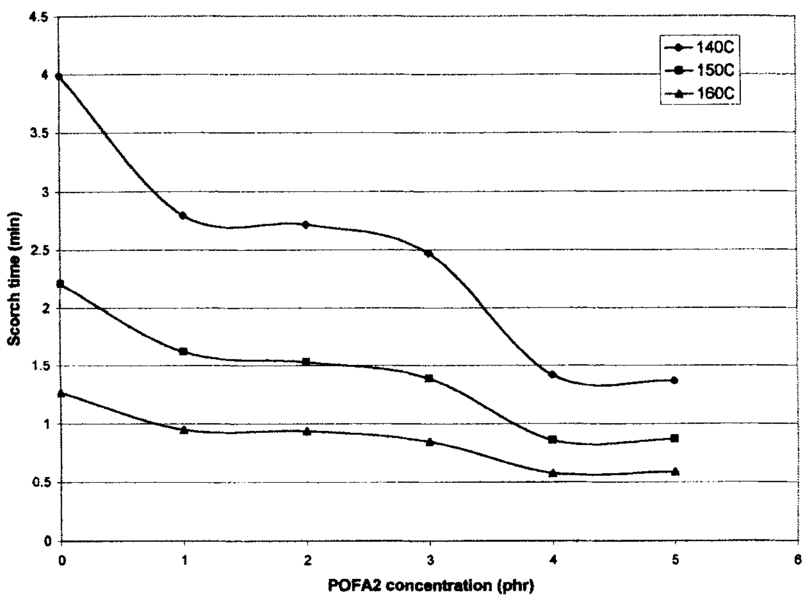

FIGURE 5 Variation of scorch time with POFA 2 concentration at three different temperatures.

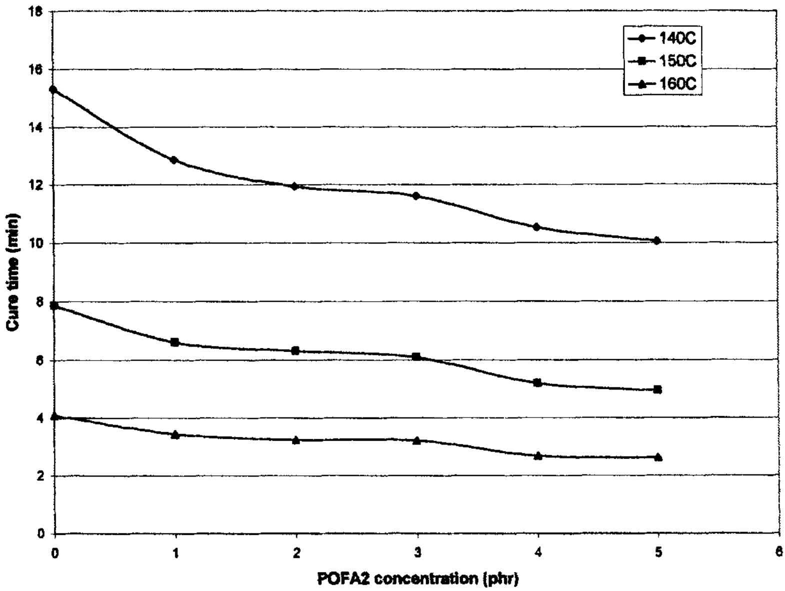

FIGURE 6 Relationship between cure time and POFA 2 concentration at three different temperatures.

Arrhenius equation,

$$K = A_o e^{-E/RT}$$

We would thus expect an exponential dependence of t_2 and t_{90} with curing temperature.

3.3. Effect on Reversion

Chen *et al.* [7-8] reported that a reversion process in an accelerated sulphur vulcanization is associated with the formation of a trans-methine structure, which is generated by the main-chain modification through the desulphuration process. Figure 7 shows the effect of POFA 2 concentration on the reversion behaviour of carbon black filled natural rubber compounds at different curing temperatures. It can be seen that, the reversion decreases with an increase in POFA 2 concentration indicating the formation of more stable crosslinks. In

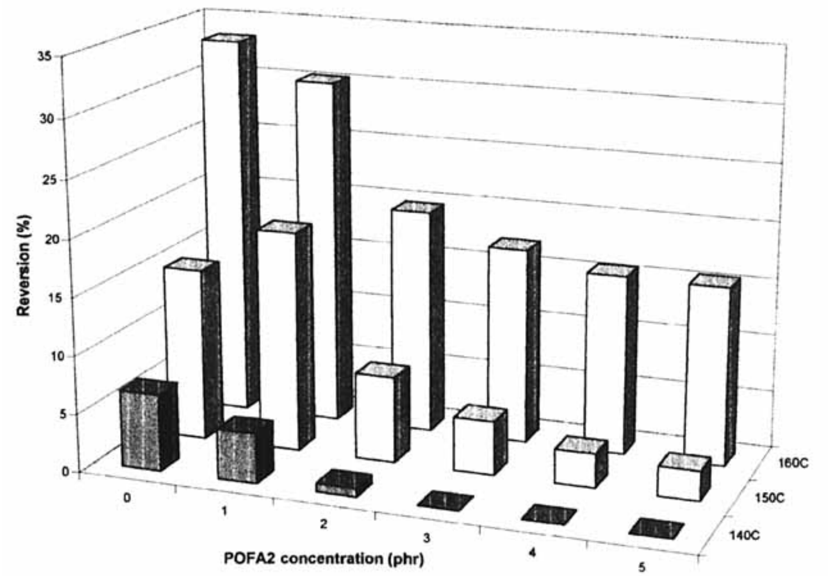

FIGURE 7 Variation of percentage reversion with POFA 2 concentration at three different temperatures.

our previous works [1–2], we have reported that the excessive lauric acid may have remained after the preparation of POFA 2. According to Bristow [9] the higher fatty acid concentrations were effective in increasing reversion resistance. It can be seen also from Figure 7 that at a similar POFA 2 concentration, the reversion increases with increasing curing temperature. Poh *et al.* [10] reported that reversion increases with increasing temperatures of vulcanization. For temperature less than 160°C, reversion increased steadily with increasing cure time and this observation is associated with the gradual thermal decomposition of di and polysulphidic crosslinks.

4. CONCLUSIONS

Based on this study, the following conclusions can be drawn:

1. Compared to the control compound, the incorporation of POFA 2 increases the maximum elastic torque but decreases minimum

elastic torque. At similar POFA 2 concentration, increment in cure temperature reduces both maximum and minimum elastic torques.
2. Incorporation of POFA 2 and higher cure temperature resulted in a lower damping properties of the natural rubber compounds.
3. Incorporation of POFA 2 and higher cure temperature decreases the scorch time t_2 and cure time t_{90}.
4. Reversion decreases with increasing POFA 2 concentration but increases with increasing cure temperature.

Acknowledgements

The supply of palm oil fatty acid, PALMAC 98 – 12 by Acidchem (M) Ltd. is gratefully acknowledge. MPKSN (Ministry of Science, Technology and Environment, Government of Malaysia) and Universiti Sains Malaysia (USM) is acknowledge for the IRPA financial grant.

References

[1] Hanafi Ismail and Ng, C. T. (1998). *Intern. J. Polymeric Mater*, **40**, 105.
[2] Hanafi, Ismail and Ng, C. T., *J. Elastomer and Plast.* (in press).
[3] Kok, C. M. (1987). *Eur. Polym. J.*, **23**, 611.
[4] Sezna, J. A., Pawlowski, H. A. and De Coninck, D. (1989). *136 th Meeting of the ACS Rubber Division*, Fall, p. 9.
[5] Mauro, P. J. D., de Rudder, J. and Etienne, J. P. (1990). *Rubber World.*
[6] Sezna, J. A., Dimauro, P. J. and Pawlowski, H. A. (1988). *Rubber and Plastics News.*
[7] Chen, C. H., Koerrig, J. L., Shelton, J. R. and Collins, E. A. (1981). *Rubb. Chem. Technol.*, **54**, 734.
[8] Chen, C. H., Koenig, J. L., Shelton, J. R. and Collins, E. A. (1982). *Rubb. Chem. Technol.*, **103**, 1221.
[9] Bristow, G. M. (1986). *NR Technology*, **17**, 7.
[10] Poh, B. T., Kwok, C. P. and Lim, G. H. (1995). *Eur. Polym. J.*, **31**, 223.