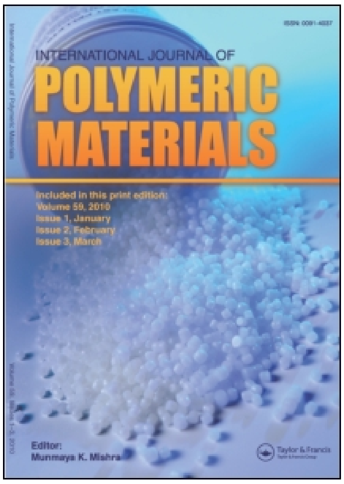

International Journal of Polymeric Materials
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713647664

The Comparison Effects of Palm Oil Fatty Acid and Stearic Acid on Dynamic Properties, Curing Characteristics and Mechanical Properties of Carbon Black Filled Epoxidized Natural Rubber Compounds

Hanafiismail[a]; S. K. Kamal[a]; S. E. Mark[a]
[a] School of Industrial Technology, Universiti Sains Malaysia, Minden, Penang, Malaysia

Intern. J. Polymeric Mater., 2001, Vol. 49, pp. 191–204
Reprints available directly from the publisher
Photocopying permitted by license only

The Comparison Effects of Palm Oil Fatty Acid and Stearic Acid on Dynamic Properties, Curing Characteristics and Mechanical Properties of Carbon Black Filled Epoxidized Natural Rubber Compounds

HANAFI ISMAIL*, S. K. KAMAL and S. E. MARK

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

(Received 16 January 2000; In final form 22 January 2000)

The effects of palm oil fatty acid and stearic acid on dynamic properties, curing characteristics and mechanical properties of carbon black filled epoxidized natural rubber compounds were studied. It was found that the scorch time, t_2 and cure time, t_{90} increased with increasing both acids concentrations. For dynamic properties, the maximum elastic torque, increased with acids concentration, whereas the minimum elastic torque shows a decreasing trend. Results also indicate that the decrease in viscous torque and tan δ is significant with increasing acids concentration. For tensile modulus, hardness, maximum torque–minimum torque and swelling index, results indicate that both acids have some effects on crosslink density. However tensile and tear strength pass through a maximum as the concentration of both acid increased.

Keywords: Palm oil fatty acid; Stearic acid; Dynamic properties; Curing characteristics; Mechanical properties

*Corresponding author. Fax: 04-6573678, e-mail: ihanafi@usm.my

1. INTRODUCTION

In our previous work [1], we have reported that effect of palm oil fatty acid on curing characteristics and mechanical properties of $CaCO_3$ filled natural rubber compounds. Results show that the scorch time and t_{90} increase with increasing concentration of palm oil fatty acid while the $M_{HR}-M_L$ (maximum torque–minimum torque) and mechanical properties passes through a maximum value and decreases with acid concentration. Reversion studies indicate that incorporation of palm oil fatty acid improves the resistance to reversion of the natural rubber compounds. In this study, the comparison effects of the similar palm oil fatty acid and a commercial stearic acid in carbon black filled epoxidized natural rubber compounds were carried out. The effects of concentration of palm oil fatty acid and commercial stearic acid on dynamic properties, curing characteristics and mechanical properties were investigated.

2. EXPERIMENTAL

2.1. Materials and Chemicals

Table I shows the materials, their manufacturers, and concentrations used in this study. All materials were freshly supplied and used without further purification. Palm oil fatty acid (Palmac 770) was obtained from Acidchem (Malaysia) Ltd., Penang, Malaysia and its specification is shown in Table II [1].

TABLE I Different concentrations of palm oil fatty acid and stearic acid in carbon black filled epoxidized natural rubber compounds

Materials	*Supplier*	*(phr)*	*(phr)*
ENR50	RRIM[a]	100	100
Carbon black (N330)	Malayan Carbon (M) Ltd.	40	40
Zinc oxide	Bayer (M) Ltd	5	5
Flectol H[b]	Bayer (M) Ltd	1	1
CBS[c]	Bayer (M) Ltd	0.5	0.5
Sulphur	Bayer (M) Ltd	2.5	2.5
Palm oil fatty acid	Acidchem (M) Ltd	1, 3, 5, 7	–
Stearic acid	Bayer (M) Ltd	–	1, 3, 5, 7

[a] Rubber Research Institute of Malaysia.
[b] Poly-1,2-dihydro-2,2,4-trimethylquinoline.
[c] N-cyclohexyl-benzothiazole-2-sulphenamide.

TABLE II Specification for palm oil fatty acid (Palmac 770 – oleic acid 80% min.) [1]

Titre (°C)	9 max
Iodine value	94 – 102
Acid value	194 – 204
Colour Lovibond 5.25″ cell max.	10 yellow 1.5 red
Typical chain length	Composition (%)
Saturated	
$C_{10:0}$ (caprylic acid)	
$C_{12:0}$ (lauric acid)	
$C_{14:0}$ (myristic acid)	6 max.
$C_{16:0}$ (palmitic acid)	
$C_{18:0}$ (stearic acid)	
Unsaturated	
$C_{18:1}$ (oleic acid)	80 min.
$C_{18:2}$ (linoleic acid)	14 max.

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 equipment is replacing the oscillating disk rheometer (ODR) as described in ASTM Standard Test Method D 2084. The dynamic properties of the blends before, during and after cure were studied at 150°C. A unique signal processing system and Fourier transform software separates the complex torque into elastic torque (S') and viscous torque (S''). The tan δ 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. The compounds were then compression moulded at 150°C by using the respective cure times, t_{90}.

2.3. Measurement of the Mechanical Properties

The tensile properties and crescent tear strength of the vulcanizates were determined by using a Monsanto Tensometer, T10 according to BS903: Part A2 and BS 903: Part A3, respectively at 500 mm/min crosshead speed. The test for hardness (IRHD) was measured by using a Wallace dead load tester according to BS 903: Part A26. All tests were conducted at room temperature (25°C).

2.4. Swelling Index

Cured test samples of dimensions 30 mm × 5 mm × 2 mm were weighed using an electrical balance (W_o) and each sample was immersed in a glass vessel containing toluene (30 ml) at 25°C for 24 h and the swollen sample (W_t) was determined and the swelling index was calculated as follows:

$$\text{Swelling index} = \frac{\text{Swollen weight}(W_t)}{\text{Original weight}(W_o)}$$

3. RESULTS AND DISCUSSION

Figure 1 shows the comparison effect of palm oil fatty acid and stearic acid on the minimum elastic torque (S' @ ML) of carbon black filled ENR compounds. According to Sezna *et al.* [2], S' @ ML is commonly considered as representative of the uncured stock's elastic modulus and also provide valuable information about a compounds processability. It can be seen in Figure 1 that the minimum elastic torque decreased with increasing amount of both acids. This result indicates that the use of both acids improved the processability of the rubber compounds. However at similar acid concentration, stearic acid gives lower S' @ ML than palm oil fatty acid.

Figure 2 shows the comparison effect of palm oil fatty acid and stearic acid on the maximum elastic torque (S' @ MH) of carbon black filled ENR compounds. It can be seen that the maximum elastic torque increased slightly with increasing concentration of both acids. S' @ MH generally correlates with durometer hardness and/or modulus [2]. From Figure 2 it can be seen that at similar acid concentration, stearic acid gives higher S' @ MH then palm oil fatty acid.

The comparison effect of palm oil fatty acid and stearic acid on the torque difference, S' @ MH-S' @ ML (max. elastic torque − min. elastic torque) is shown in Figure 3. It can be seen that the torque difference increased with increasing concentration of both acid.

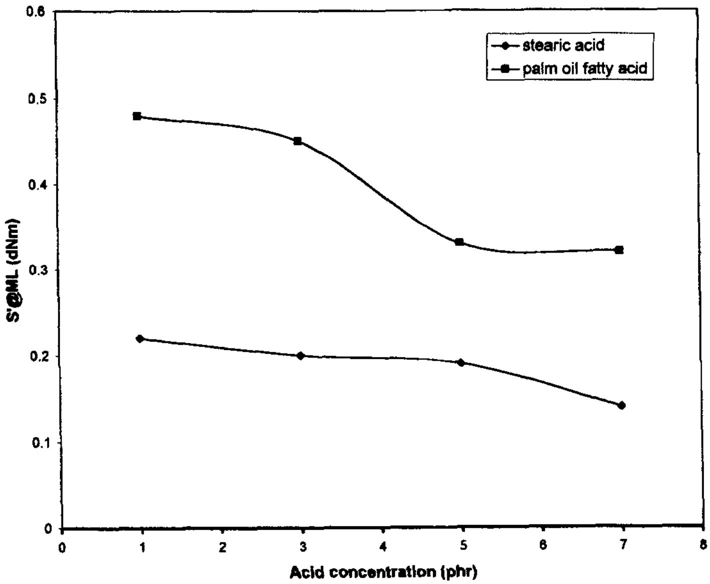

FIGURE 1 Effects of palm oil fatty acid and stearic acid on maximum elastic torque (S' @ ML) of carbon black filled ENR 50 compounds.

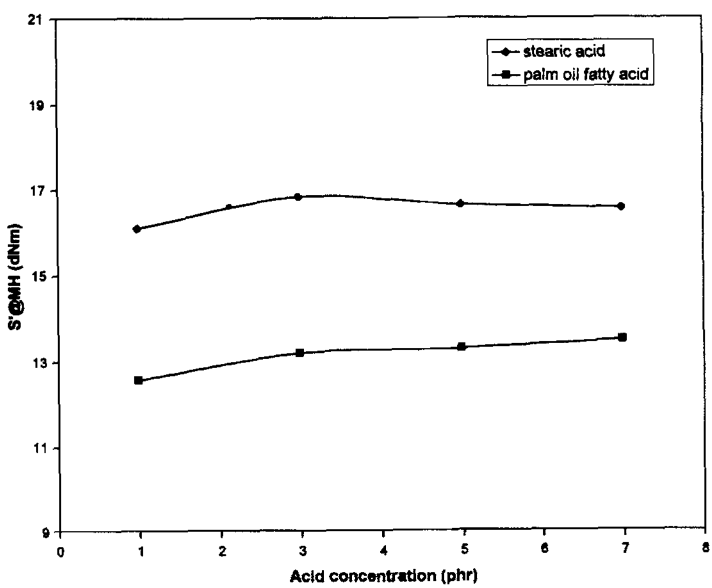

FIGURE 2 Relationship between S' @ MH and acid concentration (palm oil fatty acid and stearic acid) of carbon black filled ENR 50 compounds.

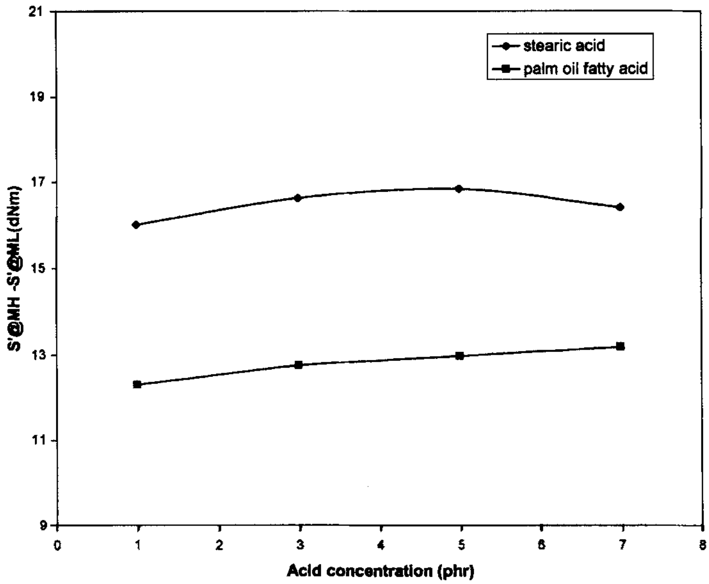

FIGURE 3 Relationship between torque difference and acid concentration (palm oil fatty acid and stearic acid) of carbon black filled ENR 50 compounds.

This result indicates that both acids have an activating effect to cause more efficient use of sulphur for higher degree of crosslinking. Previous works by other researchers [3,4] on MBT (2-Mercaptobenzothiazole) accelerated system reported there was an increment in crosslinking with increasing fatty acid on rubber compounds. However Adams and Johnson [5] reported that inclusion of MBT in a NR-sulphur system results in an increased rate of sulphur combination and crosslinking efficiency. For MBT to exercise the dual function of increasing both rate and efficiency, the additional presence of zinc oxide and a fatty acid on the corresponding zinc soap is required [4,5].

Figures 4 and 5 show the comparison effect of palm oil fatty acid and stearic acid on the viscous torque (S'' @ MH) and tan δ @ MH. Viscous torque relates to the damping characteristics of a rubber compound [2,6] whereas for tan δ the lower the tan δ for a cured compound, the greater its resiliency [7]. From Figures 4 and 5, it can be seen that the decrease in viscous torque or loss modulus and in tan δ values are significant with increasing both acids concentration.

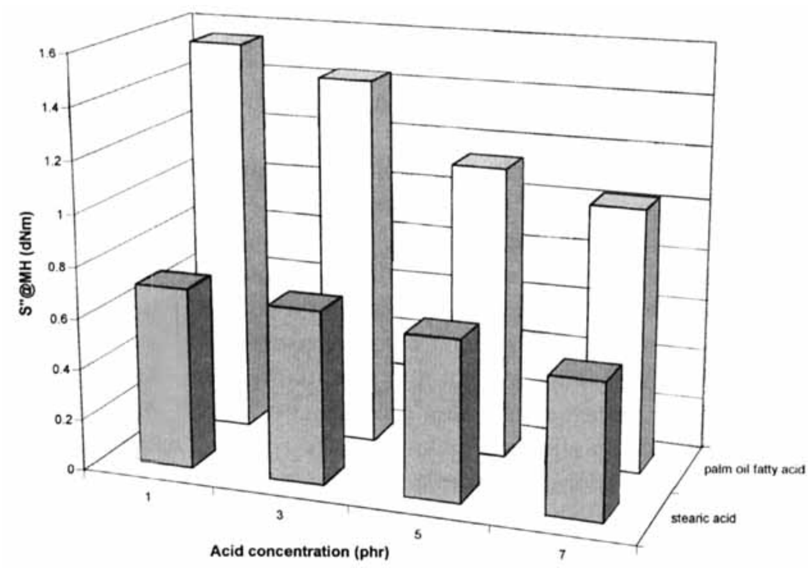

FIGURE 4 Variation of viscous torque (S'' @ MH) and acid concentration (palm oil fatty acid and stearic acid) of carbon black filled ENR 50 compounds.

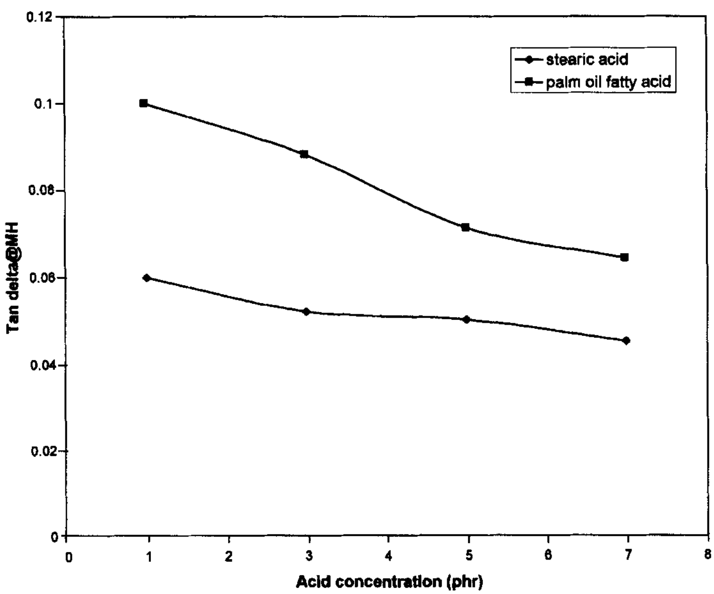

FIGURE 5 Effect of acid concentration (palm oil fatty acid and stearic acid) on tan δ @ MH of carbon black filled ENR 50 compounds.

However at similar acid concentration, stearic acid gives lower viscous torque and tan δ @ MH.

The effects of both acids on scorch time, t_2 and cure time, t_{90} are shown in Figures 6 and 7. It can be seen that t_2 and t_{90} increased with increasing both acids concentration. These results exhibit that both acids retard the onset of vulcanization. According to Coran [8, 9] the specific rate of vulcanization is decreased if the concentration of stearic acid is increased. This is attributed to the complex formation of chelates between zinc ions (brought into solution by stearic acid) and the accelerator, intermediate reaction products, or crosslink precursors.

The comparison effects of palm oil fatty acid and stearic acid on various vulcanization properties are shown in Figures 8–13. Figures 8 and 9 show that the tensile modulus at 100% and 300% elongation increased with increasing both acids concentration. Results from hardness test in Figure 10 also show a similar trend. At similar acid concentration, stearic acid exhibits better tensile modulus and hardness than palm oil fatty acid.

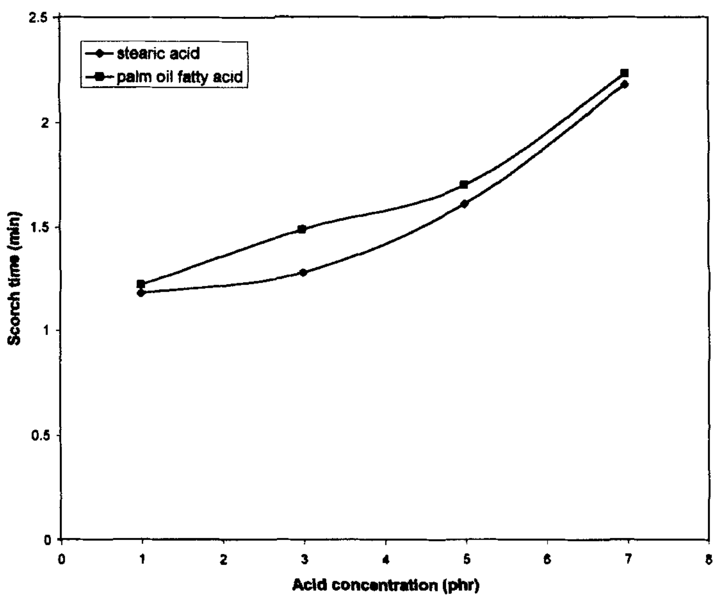

FIGURE 6 Effect of acid concentration (palm oil fatty acid and stearic acid) on scorch time, t_2 of carbon black filled ENR 50 compounds.

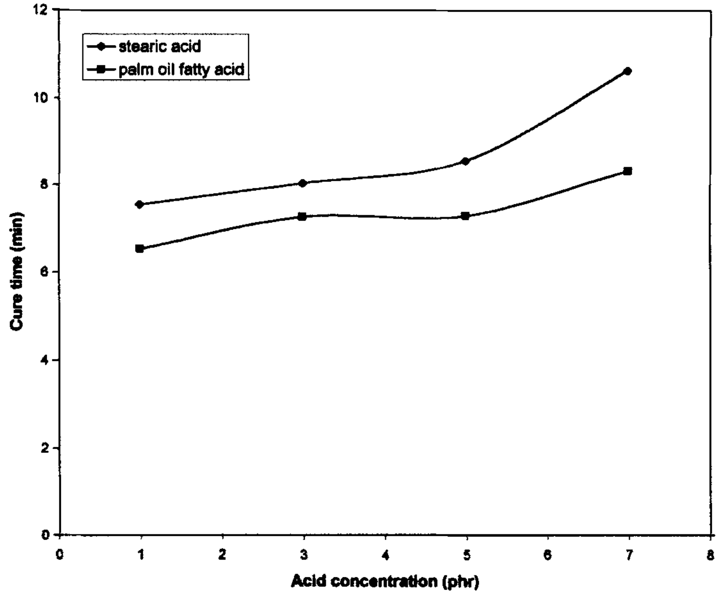

FIGURE 7 Effect of acid concentration (palm oil fatty acid and stearic acid) on cure time, t_{90} of carbon black filled ENR 50 compounds.

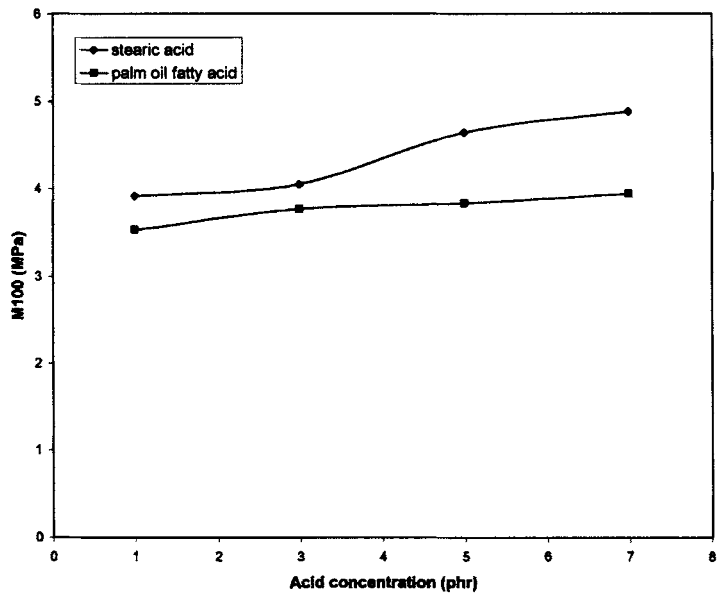

FIGURE 8 Relationship between M100 and acid concentration (palm oil fatty acid and stearic acid) of carbon black filled ENR 50 compounds.

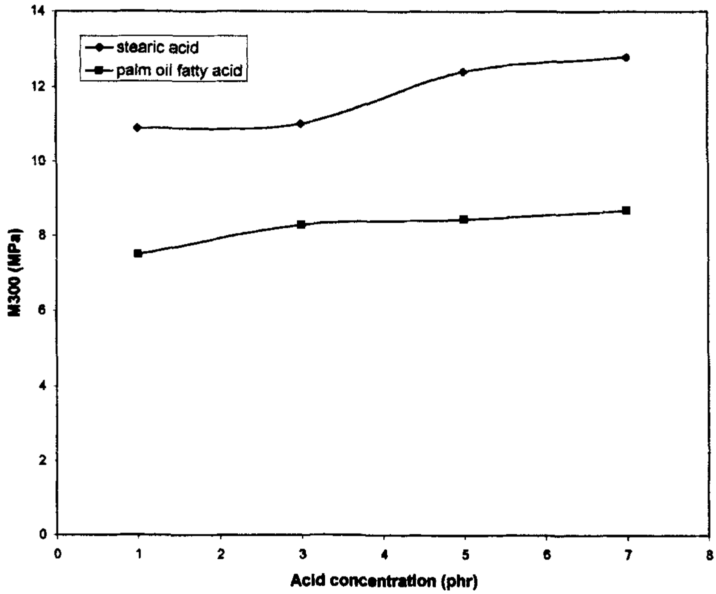

FIGURE 9 Relationship between M300 and acid concentration (palm oil fatty acid and stearic acid) of carbon black filled ENR 50 compounds.

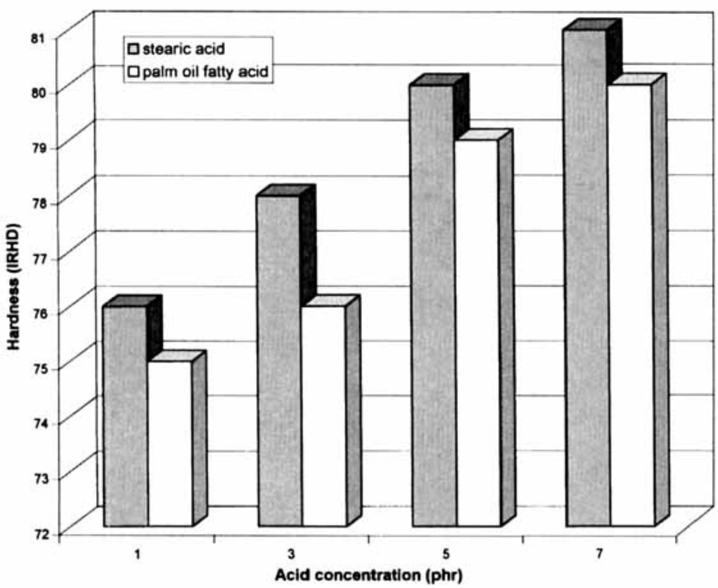

FIGURE 10 Relationship between hardness and acid concentration (palm oil fatty acid and stearic acid) of carbon black filled ENR 50 compounds.

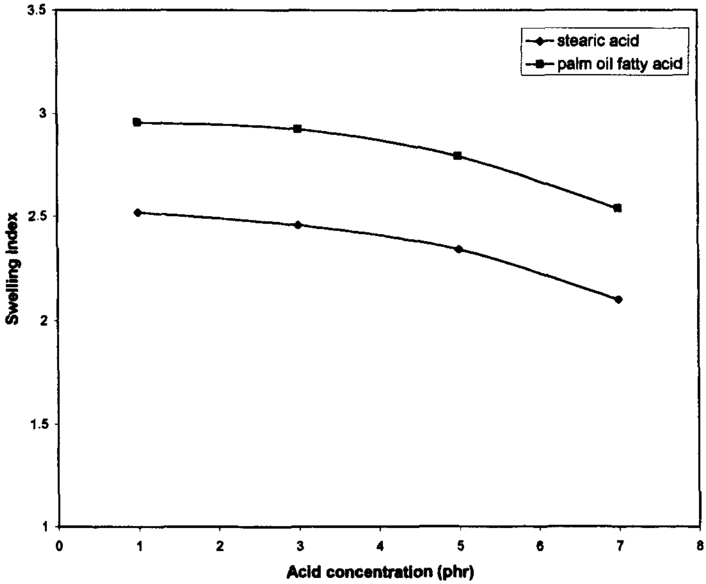

FIGURE 11 Relationship between swelling index and acid concentration (palm oil fatty acid and stearic acid) of carbon black filled ENR 50 compounds.

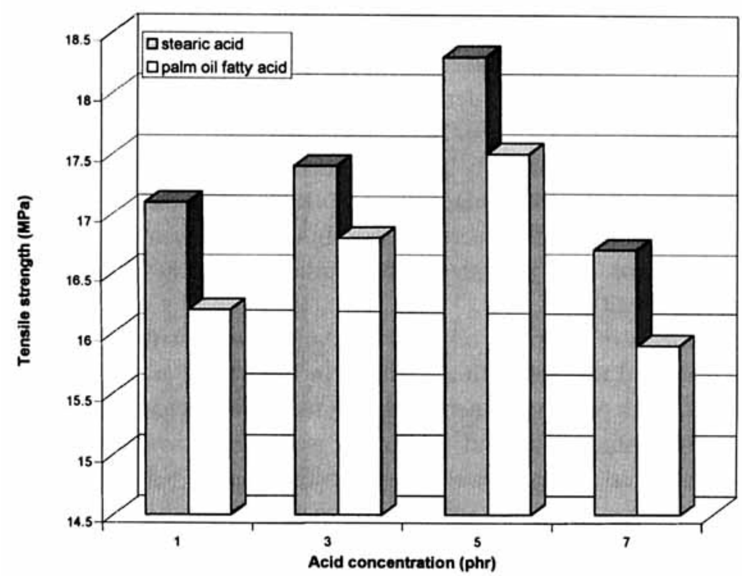

FIGURE 12 Effect of acid concentration (palm oil fatty acid and stearic acid) on tensile strength of carbon black filled ENR 50 compounds.

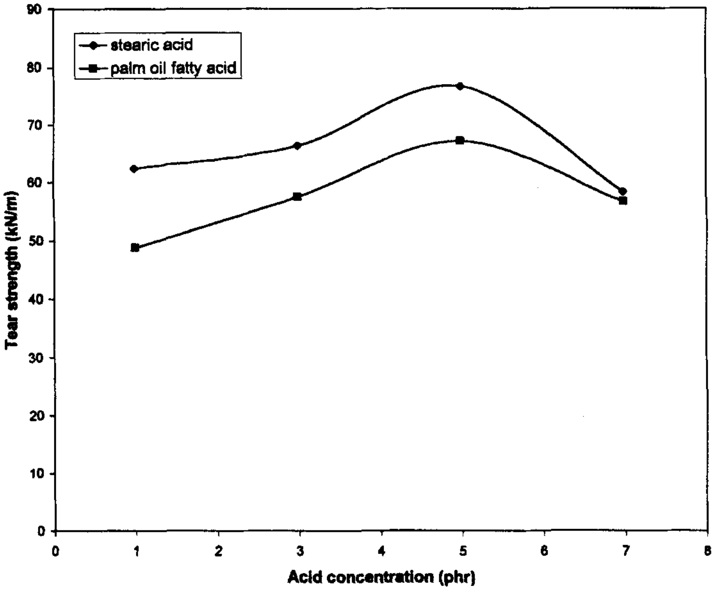

FIGURE 13 Effect of acid concentration (palm oil fatty acid and stearic acid) on tear strength of carbon black filled ENR 50 compounds.

Gent [10] reported that modulus and hardness increase monotonically with crosslink density, and at the same time, networks become more elastic or less hysteretic. Figure 11 shows the relationship between palm oil fatty acid and stearic acid concentrations with swelling index of carbon black filled ENR compounds. It can be seen that the swelling index decreases continuously with increasing acids concentrations. This provides a good indication of the increase in the crosslink density.

The effects of the palm oil fatty acid and stearic acid concentration on tensile and tear strength are shown in Figures 12 and 13. It can be seen that both properties increased with increasing acids concentration up to maximum value and then decreased. Both crosslink type and degree of crosslinking have a significant effect on the tensile strength of natural rubber. Normally the tensile and tear strength pass through a maximum with increasing degree of crosslinking [11, 12]. As previous properties, at similar acid concentration natural rubber compounds with stearic acid show higher tensile and tear strengths than palm oil fatty acid.

From Figures 1 – 13 for various properties, although the palm oil fatty acid shows a similar trend as the stearic acid, at similar acid concentration the properties for carbon black filled ENR compounds with palm oil fatty acid differ slightly compared to compounds with stearic acid. As indicated in Table II the palm oil fatty acid used in this study contains various acids. This explains why the properties of carbon black filled ENR compounds with both acids differ slightly. However from the overall observations, it can be concluded that the palm oil fatty acid can be used in the rubber industry in the same roll as a commercial stearic acid.

4. CONCLUSION

From this study, the following important conclusions can be drawn:

1. The incorporation of palm oil fatty acid and stearic acid increases the scorch time, t_2, cure time, t_{90} and maximum torque – minimum torque of the carbon black filled ENR compounds.
2. The viscous torque and tan δ decrease with increasing both acids concentration. However at similar acid concentration, stearic acid gives lower viscous torque and tan δ.
3. Tensile modulus and hardness increased with increasing both acids concentration whereas tensile and tear strengths passed through a maximum. At similar acid concentrations, stearic acid gives higher mechanical properties than palm oil fatty acid.

References

[1] Hanafi, Ismail and Ruhaizat, T. A. (1997). *Iranian Polym. J.*, **6**, 97.
[2] Sezna, J. A., Dimauro, P. J. and Pawlowski, H. A., *Rubber Plastics News*, 18 April, 1988.
[3] Moore, C. C. and Porter, M. (1967). *J. Appl. Polym. Sci.*, **11**, 2227.
[4] Barton, B. C. and Hart, E. J. (1953). *Rubb. Chem. Technol.*, **26**, 510.
[5] Adams, H. E. and Johnson, B. L., *Rubb. Chem. Technol.*, **26**, 741.
[6] Sezna, J. A., Pawloski, H. A. and De Coninck, D. (1989). 136th Meeting of the ACS Rubber Division, Fall, p. 9.

[7] Mauro, P. J., deRudder, J. and Etienne, J. P., *Rubber World*, January, 1990.
[8] Coran, A. Y. (1964). *Rubb. Chem. Technol.*, **37**, 679.
[9] Coran, A. Y. (1965). *Rubb. Chem. Technol.*, **38**, 1.
[10] Gent, A. N. (1992). *Engineering with Rubber: How To Design Rubber Components*, Hanser, p. 21.
[11] Bristow, G. M. and Tiller, R. F. (1970). Kautsch. *Gummi Kunst.*, **23**, 55.
[12] Morrison, N. J. and Porter, M. (1984). *Rubb. Chem. Technol.*, **57**, 63.