Preparation of Multifunctional Additive [MFA] from Castor Oil (CO) and Study of its Effects on the Physical Properties of Natural Rubber Vulcanizates

Kingsley Kema Ajekwene, Johnson Oseghale Oboh, Ugonna Kingsley Ugo, Simon Ikechukwu Ichetaonye

Abstract – This experiment focuses on the synthesis of Multifunctional Additives (MFA) from Castor seed oil (CO) and pure Oleic acid (OA) respectively. These multifunctional additives (CO-MFA and OA-MFA) were investigated as potential substitutes for process oil, activator, co-activator, and accelerator in the formulation of a natural rubber vulcanizate. During processing, the castor seed oil was characterized for acid value (AV), free fatty acid (FFA) and saponification value (SV) and subsequently reacted with 1, 6-hexane diamine to obtain a mixture of salts termed castor oil-based multifunctional additives (CO-MFA), similarly, a pure oleic acid was also reacted with 1, 6-hexane diamine to obtain a pure salt termed oleic acid-based multifunctional additive (OA-MFA). These compounds were investigated as substitutes for process oil/stearic acid, process oil/stearic acid/zinc oxide, process oil/MBT, and process oil/stearic acid/zinc oxide/MBT in the formulation and compounding of natural rubber vulcanizate. The pre-mastication of the rubber and subsequent mixing with additives was carried out in a Banbury Internal Mixer at a rotor speed of 50 rpm for 7 mins while the vulcanization was achieved on a compression molding machine at a temperature of 140°C for 15 mins. The various vulcanizates samples were evaluated for tensile properties, compression set, abrasion resistance, and hardness. The use of castor seed oil-based multifunctional additives (CO-MFA) as a substitute for process oil/stearic acid/zinc oxide/MBT gave the highest value of tensile strength (12.24 MPa) as against the conventional rubber compound (4.21 MPa).

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I. INTRODUCTION

The unique viscoelastic characteristics, low specific weight and its window for vagaries of modification to meet broad ranges of application requirements of rubbers have made them indispensable choices as matrix materials for highperformance composites where energy dissipation is an essential component of end-use requirements [1-7]. However, raw natural rubber is subjugated by a double bond in its repeat unit thereby limiting the durability and strength properties of rubber end use applications [8-11]. Therefore, the compounding of rubber involves the addition of numerous additives for specific processing and finished product requirements [12, 13]. In a typical rubber compounding line, there is the need for accurate metering of all the additives to the base polymer to obtain a uniform and homogenous mix of desired properties [7, 8]. Despite the unique properties of pristine rubbers such as elasticity, high rebound energy, and excellent damping characteristics amongst others, they are required to be compounded with other materials before meeting desired requirements due to their susceptibility to thermal and oxidative degradation [14-16]. Compounding involves the incorporation into the rubber matrix of some ingredients known as additives to achieve desired properties such as processability, stiffness, weatherability, and bondability amongst others. An elaborate list of compounding ingredients are Base polymer, Vulcanizing agent, Accelerator for cross-linking reaction, Accelerator modifiers (activators, retarders, inhibitors), Processing aids [plasticizers, peptizers, factice, dispersing aids, tackifiers, process oil, lubricants, softeners, and extenders], Fillers, Antistatic agents, Anti-degradants (antioxidant, antiozonant, stabilizers), Special purpose additives (colourants and pigments, flame retarders, deodorants, blowing agents, stiffening agents, abrasives, diluents, integral bonding additives, and wetting agents). It

can be seen that the overall total number of ingredients in a sophisticated rubber formulation can be very large [15-16]. Castor oil, known primarily for its medicinal use as a cathartic [to release stress and tension], is now used as an industrial raw material for the preparation of chemical derivatives used in coatings, urethane derivatives, surfactants and dispersants, cosmetics, lubricants and textiles [17]. Castor oil is a triglyceride of fatty acids and is sometimes described as a triglyceride of ricin-oleic acid being its principal component. It is one of the few naturally occurring glycerides that approaches being a pure compound. Ricinoleic acid (cis-1, 2-hydroxy octadec-9-enoic acid or 1, 2-hydroxyoleic acid or castor oil acid) with the formula: CH_3 [CH₂]₅CH [OH] CH₂CH = CH [CH₂]₇COOH is an 18carbon unsaturated fatty acid comprising over 80% of the fatty acid content of castor oil [17, 18]. Most accelerators used in Sulphur cured rubbers are amine-based obtained through the reaction of diamine and fatty acid [R-NH [CH₂] $NH_3]_2 + 2[C_{17}H_{35}COO]$ - However, the amine-containing part of the salt has varying [CH₂] content depending on the amine that is used. In this study, Hexane diamine is chosen to react with an agro-based mineral oil with the objective to substitute a petroleum-based processing additive with an eco-friendlier biobased alternative. In addition, using a single additive to replace multiple additives will reduce the number of storage tanks, automatic metering scales, other associated equipment, total homogenization time, and additives cost. Aminated epoxidized soybeans oil has also been reported to promote rubber curing and increased tensile strength by approximately 20 % [19]. Biobased MFA from other natural sources such as animal fat [tallow], rubber seed oil, and other seed oils with high AV and FFA have been investigated for rubber compounding [20-23].

II. MATERIALS AND METHODS

A. Materials

The major materials used in this experiment were castor seed oil, dry natural rubber, and commercial-grade rubber compounding ingredients. Others were analytical reagent grade 1, 6 diamine hexane, oleic acid, sodium hydroxide, potassium hydroxide, hydrochloric acid, and ethanol.

B. Methods

i. Characterization of Castor Seed Oil

The castor seed oil was characterized in terms of Acid Value (AV), Free Fatty Acid (FFA), and Saponification Value (SV). In the case of AV and FFA, 10.03 g of the oil was

weighed out and dissolved in 50 ml of 96 % ethanol. Two drops of the indicator were added to the solution in a flat bottom flask and titrated against 0.1 sodium hydroxide solution. The volume of NaOH required to completely neutralize the sample was obtained. An average volume was obtained from three tests using eqn. (1) and (2)

$$AV = \frac{56.1 NV}{W}$$
(1)
$$FFA = \frac{(VNM)}{W}$$
(2)

Where V = Volume of NaOH (ml), W = Mass of castor seed oil (g), N = Normality of NaOH, M = Molecular weight of fatty acid (castor oil is predominantly Ricinoleic acid and has a molecular weight of 298)

In the case of saponification value, 2.02 g of the oil was refluxed at 80 °C with 2.5 ml, 0.5 molar ethanoic KOH solution for 60 minutes. It was allowed to cool and the mixture was titrated against 0.5 M HCl acid solution using phenolphthalein as an indicator. A blank test, using the same volume of ethanoic KOH solution (without the oil mixed with it) was also carried out and the saponification value was calculated using eqn. (3)

$$SV = \frac{(Vo-Vi)*C*56.1}{W}$$
 (3)

Vo = Volume of HCL, Vi = Volume of castor seed oil (ml), C = Concentration of HCl (moles / liter), W = Mass of oil (g)

ii. Preparation of Multifunctional Additives(MFA)

In this experiment, two types of MFA were prepared, one from castor seed oil and the other from pure oleic acid. In the former, 4.6 g of hexane 1, 6-diamine was added dropwise to 28.2 g of castor oil in a 250 ml reaction vessel with constant stirring. The reaction was fully stirred for 60 minutes at a temperature of 65 °C in order to ensure that the diamine was

fully reacted. The reaction mixture was then cooled for 30 minutes in a cold-water bath and labeled as Castor oil-based multifunctional additive (*CO-MFA*). Similarly, the other one was prepared from pure oleic acid such that 4.6 g of hexane 1, 6-diamine was added dropwise to 28.2 g of oleic acid (pure fatty acid). This reaction was observed to be highly exothermic. After cooling, the resulting product (paste) was labeled oleic acid-based multifunctional additive (*OA-MFA*). Both compounds were separately investigated as substitutes for either process oil, Accelerator, co-activator (stearic acid

and zinc oxide), or accelerator and co-activator (stearic acid and zinc oxide)

iii. Formulation and compounding of MFA Rubber-Based Compound

The (*CO-MFA*) and (*OA-MFA*) were used in the formulation of natural rubber vulcanizates as shown in Table 1 and 2 respectively, as potential substitutes for either process oil, Accelerator, co-activator (stearic acid and zinc oxide) or accelerator and co-activator (stearic acid and zinc oxide). The compounding was carried out in a Banbury Internal Mixer at a rotor speed of 50 rpm for 7 minutes and vulcanized in a compression molding machine at 140 °C for 15 minutes.

	COMPOUND CODES				
Ingredients	Blank NR	NR/OA- MFA ₁	NR/OA- MFA ₂	NR/OA -MFA ₃	NR/OA- MFA ₄
Natural rubber	100	100	100	100	100
OA-MFA	-	2	2	2	2
Process oil	4	-	-	-	-
Zinc oxide	5	5	5	-	-
Stearic acid	2	-	2	-	-
MBT	1	1	-	1	-
TMQ	1	1	1	1	1
Sulphur	2.5	2.5	2.5	2.5	2.5
Carbon black	40	40	40	40	40

Table 1: Castor Oil-Based Multifunctional Additive (Mixed Salt)

	COMPOUND CODES				
Ingredients	Blank NR	NR/CO- MFA ₁	NR/CO- MFA ₂	NR/CO- MFA ₃	NR/ CO- MFA ₄
Natural rubber	100	100	100	100	100
CO-MFA	-	2	2	2	2
Process oil	4	-	-	-	-
Zinc oxide	5	5	5	-	-
Stearic acid	2	-	2	-	-
MBT	1	1	-	1	-
TMQ	1	1	1	1	1
Sulphur	2.5	2.5	2.5	2.5	2.5
Carbon black	40	40	40	40	40

 Table 2: Oleic Acid-Based Multifunctional Additive

 (Pure Salt)

iv. Evaluation of Physical Properties of MFA Rubber-Based Compound

a. Tensile Test (Tensile Strength, Modulus, and Elongation at break)

ASTM D412 method was adopted for this test with the aid of Universal Instron Machine Model 3366 with a load cell of 12 KN, a pre-molded dumbbell-shaped sample with a crosssection area of 8 x 4 mm² was used to perform the test at the strain rate of 80 mm/mm at room temperature. The tensile strength, % elongation at break, and moduli at 100 and 300 % strains were obtained from the stress-strain curve plotted by the Instron

b. Hardness

The hardness test was carried out with the aid of a Shore Wallace Hardness tester made in the United Kingdom. The test pieces were measured for the level of penetration by machine indenter using the International Rubber Hardness Degree Scale (IRHD).

Abrasion Resistance c.

The abrasion resistance test was carried out with the aid of Wallace Akron's abrasion tester. The angle between the test specimen and the abrasive wheel was adjusted to 15 degrees at 1000 revs. The volume of material loss as a result of the abrasive action of the rotating wheel is used to calculate the abrasive resistance index (%).

d. **Compression Set**

The constant load compression set test was used in this experiment with the aid of a Wallace compression set machine, such that the test specimen was compressed between parallel steel plates under a constant stress of 2.8 Mpa for 24 hours at 70 °C for 24 hours. The percentage change in thickness of the test specimen after the experiment is used to evaluate the compression set.

III. **RESULT AND DISCUSSIONS Properties of Castor Seed Oil** a.

The characteristics of the castor seed oil used in this experiment are shown in Table 3

Table 3: Proximate Analysis of Castor Seed Oil				
Acid value of castor oil	3.03			
Free fatty, FFA [%]	1.61			
Saponification value	162			

b. Processibility

The function of processing process oil or any other processing aid used in rubber mixing is basically to ease the incorporation of dry additives into the rubber matrix during rubber mixing by providing better wetting and dispersion of the dry additives into the rubber matrix and subsequently reducing energy consumption required for mixing [19]. Figure 1 showed the mixing energy consumption by various samples, the blank NR samples have in their formulation (see Table 1 & 2) petroleum-based process oil while other samples contain only MFA as a processing aid. The mixing energy obtained directly from the Banbury internal as presented in the figure clearly showed that the blank NR sample consumed far more energy than other samples. Therefore, both CO-MFA and OA-MFA invariably enhance the carbon black filler wetting and dispersion.

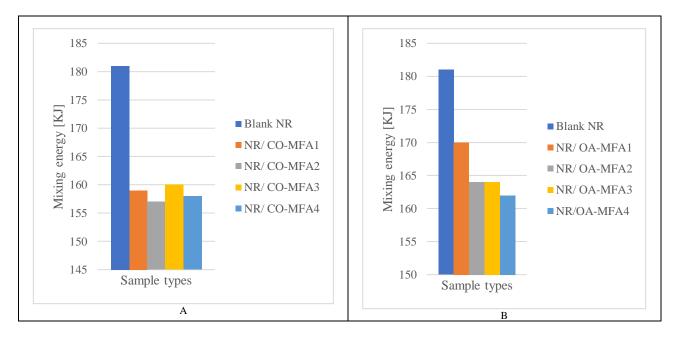


Fig. 1. Effect of MFA on Mixing energy [a] CO-MFA [b] OA-MFA

c. Tensile Properties

The effect of both MFA samples on Tensile strength, Modulus at 100 % elongation, Modulus at 300% elongation, and Elongation at break of vulcanizates samples are presented in Figures 2, 3,4, and 5 respectively. Simple deductions based primarily on a comparison of compounds can be made since the same base polymer (NSR 10) and cure conditions were used for all compounds. NR/CO-MFA3 and NR/OA-MFA₃ both without activator and process oil had similar values of tensile strength as the conventional formulation used as the control sample (Blank NR). The compounds of NR/CO-MFA2 and NR/OA-MFA2 (without accelerator and process oil) gave the lowest values of tensile strength. This possibly indicates the presence of free rubber molecular chains free from cross-links as their elongations at break were quite appreciable (see Figure 5). However, NR/CO-MFA1 and NR/OA-MFA1 both without process oil and stearic acid gave higher tensile strength values than all other compounds exempt from NR/CO-MFA4 and NR/OA-MFA₄. The samples of NR/CO-MFA₄ and NR/OA-MFA₄ showed a tensile behavior that is difficult to explain. The observation of compounds NR/CO-MFA2 and NR/OA-MFA₂ both without accelerator and process oil having lower tensile strength values of 2.99 and 2.68 MPa respectively than compounds of NRCO-MFA4 and NR/OA-MFA4 both without activators, accelerators, and process oil with respective values of 10 MPa and 12 MPa as against the conventional blank NR of only 4.21 MPa is striking. The only possible deduction could be that some sort of masking effect must have taken place such that the presence of the activator system of stearic acid and zinc oxide may have masked the accelerating effect of the multifunctional additives. Similarly, the moduli at 100 and 300 % were greatly enhanced for CO-MFA4 and OA-MFA4 as against the blank NR

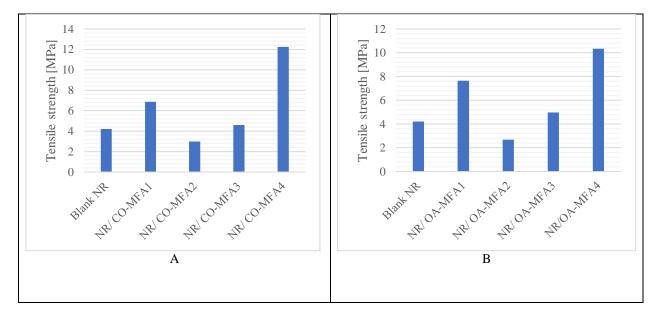
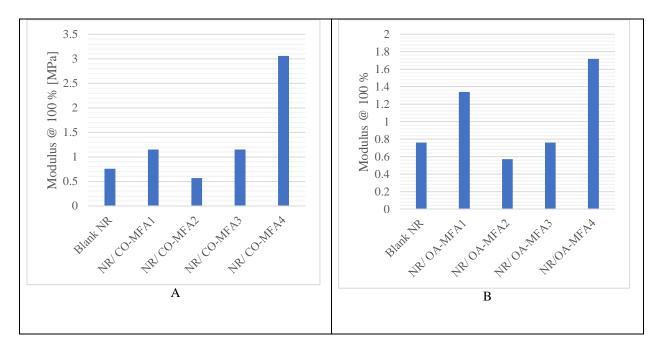


Fig. 2. Effect of MFA on tensile strength of vulcanizates (a) CO-MFA (b) OA-MFA



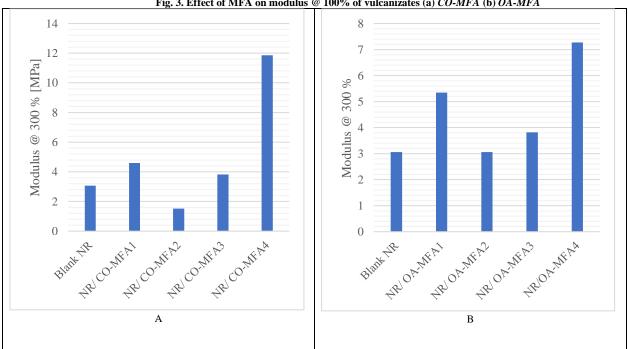


Fig. 3. Effect of MFA on modulus @ 100% of vulcanizates (a) CO-MFA (b) OA-MFA

Fig. 4. Effect of MFA on modulus @ 300% of vulcanized (a) CO-MFA (b) OA-MFA

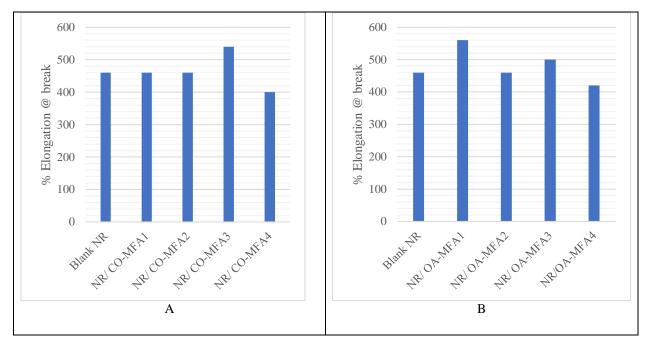


Fig. 5. Effect of MFA on % elongation of vulcanizates (a) CO-MFA (b) OA-MFA

d. Hardness

The hardness of vulcanizate samples measured on the international rubber hardness degree scale (IRHD) is shown in Figure 6. The use of castor oil as a multifunctional additive did not significantly improve the hardness of the rubber vulcanizate, this could be due to its substantial lubrication on the rubber chain leading to chain flexibility.

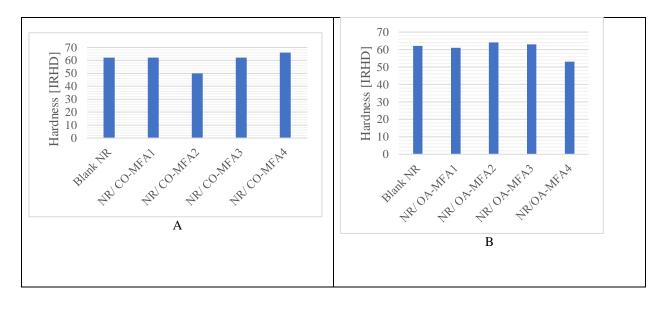


Fig. 6. Effect of MFA on the hardness of vulcanizates (a) CO-MFA (b) OA-MFA

e. Abrasion Resistance

The results for abrasion resistance are presented in Figure 7 such that abrasion resistance slightly improved for NR/CO-MFA₂, NR/CO-MFA₄, and NR/OA-MFA₁ over the control

sample and other samples. The micro-tearing associated with the mechanical erosion of vulcanized rubber surfaces is usually influenced by surface texture, elastic behavior, and chemical composition [24].

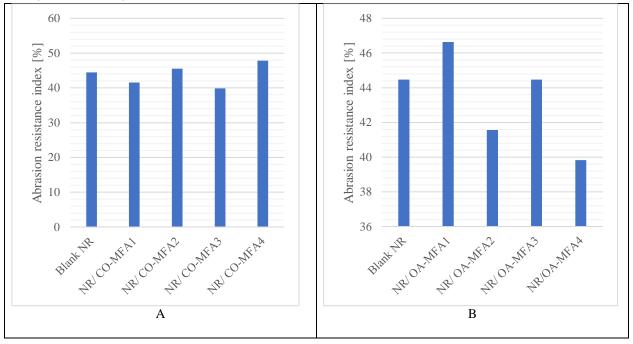


Fig. 7. Effect of MFA on abrasion resistance of vulcanizates (a) CO-MFA (b) OA-MFA

f. Compression Set

Compression set results as shown in Figure 8 reveal an improvement of vulcanizate samples with the exemption of NR/CO-MFA₄ over the control sample and OA-MFA₂ to a

level largely comparable to the blank NR vulcanizates. CO-MFA₃ has the highest percentage compression set. The cross-links formed in this compound become liable under compressive load for 24 hours at 70 °C thereby resulting in the permanent deformation of samples.

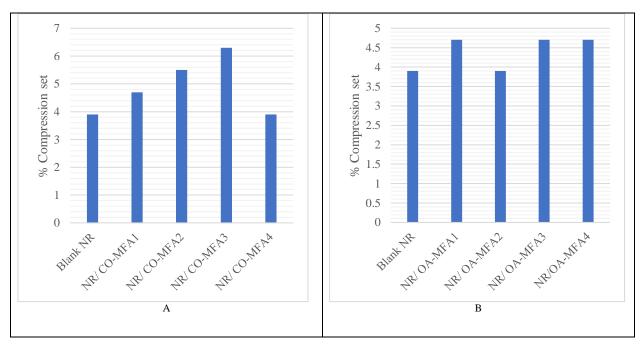


Fig. 8. Effect of MFA on compression set of vulcanizates (a) CO-MFA (b) OA-MFA

IV. CONCLUSION

Multifunctional additives have been synthesized from castor seed oil and oleic acid as a possible substitute for processing aid/ accelerator/ activator in the formulation and compounding of carbon black-filled natural rubber The findings revealed that the use of the vulcanizate. synthesized multifunctional additives as a substitute for process oil/stearic acid/zinc oxide/MBT resulted in to increase in tensile strength and moduli at 100 and 300% elongation in the vulcanized compounds, however, there were no signs in the change in Hardness, compression set, and abrasion resistance index. Therefore, castor seed oilbased multifunctional additives have tremendous potential as multipurpose processing additives in the processing of rubber products.

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Conflict of Interest/Competing Interest

The authors declare that they have no conflict of interest.

Authors Contributions

All authors contributed to the study conception and design.

All authors read and approved the final manuscript.

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