Material properties

Alkanolamide as an accelerator, filler-dispersant and a plasticizer in silica-filled natural rubber compounds

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\textbf{A B S T R A C T}

A feasibility study was carried out on the utilization of Alkanolamide (ALK) on silica reinforcement of natural rubber (NR) by using a semi-efficient cure system. The ALK was incorporated into the NR compound at 1.0, 3.0, 5.0, 7.0 and 9.0 phr. An investigation was carried out to examine the effect of ALK on the cure characteristics and properties of NR compounds. It was found that ALK gave shorter scorch and cure times for silica-filled NR compounds. ALK also exhibited higher torque differences, tensile modulus, tensile strength, hardness and crosslink density of up to 5.0 phr of ALK loading, and then decreased with further increases of ALK loading. The resilience increased with increased ALK loading. Scanning electron microscopy (SEM) micrographs proved that 5.0 phr of ALK in the silica-filled NR compound exhibited the greatest matrix tearing line and surface roughness due to higher reinforcement level of the silica, as well as better dispersion and cure enhancement.

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1. Introduction

Carbon blacks and silicas of varying forms and particle-sizes are very popular and have been widely utilized as reinforcing fillers in the rubber industry. In general, the properties of silica-reinforced rubber vulcanisates are usually inferior to those of carbon blacks, even when they are of comparable size [1]. This is attributed to the apparent dissimilarity in the surface chemistry of the two materials. Carbon black will react with sulphur during vulcanization and form sulphur bonds that link the rubber chains, and also tie the carbon black to the rubber. This is known as filler-rubber crosslinking, another type of crosslink to the rubber system, and is defined as coupling bonds [2,3]. In marked contrast to the hydrocarbon functionality of carbon black, silica does not react with sulphur, and coupling bonds will not be formed due to its hydrophilic silanol groups that are relatively incompatible with hydrocarbon rubbers, such as natural rubber [2].

For many applications, silicas are not satisfactory alternatives to carbon blacks because of their relatively lower reinforcement levels. The fundamental problem is their surface chemistry, which is more polar and hydrated than carbon blacks, and which cause them to be difficult to wet, disperse and interact with hydrocarbon rubbers. Numerous methods have been carried out to improve the reactivity of silicas with the rubber phases. One such method is the utilization of a silane-coupling agent to modify the surface of the silica. The modified silica provide chemically active surfaces that can participate in vulcanization, providing coupling bonds between the silane and both the silica and the rubber phases [4,5]. Those products show significant improvement in performance compared to their base materials.
Another alternative method to overcome the deficiency of silica is the utilization of Alkanolamide. The ingredient is synthesized from Refined Bleached Deodorized Palm Stearin (RBDPS), a product of crude palm oil fractionation. This article describes the preparation and utilization of the new rubber ingredient as an additive in order to improve the reinforcement level of silica to natural rubber.

2. Experimental

2.1. Laboratory preparation of Alkanolamide

The reaction was carried out at atmospheric pressure in a 1000 ml reaction vessel fitted with a stirrer. Typically, 1.0 mol of RBDPS and 3.0 mol each of diethanolamine, sodium methoxide and methanol (as catalysts) were placed in the reaction flask. The specifications of the RBDPS are given in Table 1. The mixture was stirred and heated; the reaction temperature was kept constant at 70 °C for about five hours. The resultant mixture was extracted with diethyl ether, and washed with saturated sodium chloride solution. The crude Alkanolamide was purified with anhydrous sodium sulphate, and then concentrated by a rotary evaporator. The final product, a cream-coloured, waxy, solid material, namely Alkanolamide, was used as an additive in silica-filled natural rubber compounds. The reaction procedure was as shown in Fig. 1.

2.2. Materials

Natural rubber grade SMR L obtained from Guthrie (M) Sdn. Bhd., Seremban, Malaysia was used. Other compounding ingredients such as sulphur, zinc oxide, stearic acid, N-isopropyl-N′-phenyl-p-phenylenediamine (IPPD), benzothiazolyl disulphide (MBTS), and precipitated silica (grade Vulcasil – S) were supplied by Bayer Co. (M) Sdn. Bhd., Petaling Jaya, Selangor, Malaysia.

2.3. Compounding

A semi-efficient vulcanization system was used for the compounding. The recipes for the preparation of the natural rubber compounds are given in Table 2. The compounding was done on a two-roll mill (Model XK-160).

Table 3 shows the designation and composition of the NR-based recipes used in this study.

2.4. Cure characteristics

The cure characteristics of the silica-filled NR compounds were obtained using a Monsanto Moving Die Rheometer (MDR 2000), which was used to determine the scorch time (ts2), cure time (t90), and torque difference (MH1–MH2) according to ISO 3417. Samples of the respective compounds were tested at 150 °C. The compounds were subsequently compression moulded using a stainless steel mould at 150 °C with a pressure of 10 MPa using a laboratory hot-press based on the respective curing times.

2.5. Tensile, hardness and resilience properties

Dumbbell-shaped samples were cut from the moulded sheets. Tensile tests were performed at a cross-head speed of 500 mm/min using an Instron 3366 universal tensile machine according to ISO 37. The tensile strength and stress at 100% elongation (M100), 300% elongation (M300), and elongation at break were investigated. The hardness measurements of the samples were performed according to ISO 7691-1 using a Shore A type manual durometer. The resilience was studied using a Wallace Dunlop Tripsometer according to BS 903 Part A8. The rebound resilience was calculated according to the following equation:

\[
\% \text{ Resilience} = \left[ \frac{(1 - \cos \theta_2)}{(1 - \cos \theta_1)} \right] \times 100 \quad (1)
\]

where, \(\theta_1\) is the initial angle of displacement (45°), and \(\theta_2\) is the maximum rebound angle.

2.6. Scanning electron microscopy (SEM)

The tensile fractured surfaces of the compounds were examined by using a Zeiss Supra-35VP scanning electron microscope (SEM) to obtain information regarding the filler dispersion and to detect the possible presence of micro-defects. The fractured pieces were coated with a layer of gold to eliminate electrostatic charge build-up during examination.

2.7. Fourier transform infra-red (FTIR) spectroscopy

The FTIR spectra were obtained using a Perkin-Elmer 2000 series instrument. The spectrum resolution was 4 cm\(^{-1}\) and the scanning range was from 550 to 4000 cm\(^{-1}\).

2.8. Measurement of crosslink density

Swelling tests on the rubber vulcanisates were performed in toluene in accordance with ISO 1817. The cured test pieces (30 mm \(\times\) 5 mm \(\times\) 2 mm) were weighed using an electrical balance and swollen in toluene until equilibrium, which took 72 h at room temperature. The samples were taken out of the liquid, the toluene was removed from the sample surfaces and the weight was determined. The samples were then dried in the oven at 60 °C until constant weight was obtained. The swelling results were used to

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Table 1: Specification of RBDPS.

| Acid value | 0.30 mg KOH |
| Free fatty acid | 0.14% |
| Iodine value | 35.3 |
| Moisture | 0.01% |
| Colour | 2.8 red/20 yellow |

| Saturated |  |
| C12:0 (lauric acid) | 0.1% |
| C14:0 (myristic acid) | 1.2% |
| C16:0 (palmitic acid) | 59.1% |
| C18:0 (stearic acid) | 4.6% |
| C20:0 (arachidic acid) | 0.4% |

| Unsaturated |  |
| C18:1 (oleic acid) | 28.2% |
| C18:2 (linoleic acid) | 6.3% |
| Unknown | 0.1% |
calculate the molecular weight between two crosslinks ($M_c$) by applying the Flory-Rehner Equation [6].

$$M_c = \frac{-\rho V_s V_r^{1/3}}{\ln(1 - V_r) + V_r + \chi V_r^2} \quad (2)$$

$$V_r = \frac{1}{1 + Q_m} \quad (3)$$

where $\rho$ is the rubber density ($\rho$ of NR = 0.92 g/cm$^3$), $V_s$ is the molar volume of the toluene ($V_s = 106.4$ cm$^3$/mol), $V_r$ is the volume fraction of the polymer in the swollen specimen, $Q_m$ is the weight increase of the blends in toluene and $\chi$ is the interaction parameter of the rubber network-solvent ($\chi$ of NR = 0.393). The degree of the crosslink density is given by;

$$V_c = \frac{1}{2M_c a} \quad (4)$$

3. Results and discussion

3.1. Characterization of Alkanolamide

Fig. 2 shows the typical infrared spectrum of the Alkanolamide (ALK) obtained, and Table 4 shows the wavenumber of the functional groups in the ALK molecule. The strong broadband at 3370 cm$^{-1}$ is an O–H stretch of the ethanol. The C–H stretches above and below 3000 cm$^{-1}$ indicate saturated and unsaturated portions exist in the molecule [7]. The umbrella mode at 1364 cm$^{-1}$ confirms the presence of a methyl group (CH$_3$) which is attached to the carbon atom. The (CH$_2$) rocking band at 719 cm$^{-1}$ means that there are more than 4 methylene atoms in a row in the molecule. The C=O stretch is at 1615 cm$^{-1}$, and the amide C–N stretch is at 1248 cm$^{-1}$. The spectrum clearly shows the presence of wavenumbers of the functional groups of ALK.

3.2. Cure characteristics and crosslink density

The effects of ALK on the cure characteristics of the silica-filled NR compound can be seen in Fig. 3 and Table 5. Fig. 3 shows that the scorch and the cure times decreased with increased ALK loading. It was observed that there was an enhancement in the cure characteristics, indicating that ALK could act as a co-curing agent or secondary accelerator in the curing process of the silica-filled NR compounds. The cure enhancement can be attributed to the amine content of the ALK. Amine is an alkaline substance which will increase the pH of the rubber compound and, in most instances, enhance the cure rate. Any material that makes the rubber compound more basic will enhance the cure rate since acidic materials tend to retard the effect of the accelerator [8]. Increasing the ALK loading has the same effect as increasing the amount of amine in the silica-filled NR compound. Hence, it can be said that the higher the ALK loading, the greater the amount of amine and the shorter the cure rate.

According to Bateman [9], and Ismail & Ng [10], a certain organic substance containing nitrogen atoms promotes the curing process of olefinic rubber, comprising sulphur and primary accelerators, through the formation of complexes which are responsible for the fission of the sulphur molecules to form crosslinks between the linear rubber chains. It is expected that the amine-content of the ALK would accelerate the cure and is responsible for enhancing the cure rate and the cure state of the silica-filled NR compound.

As presented in Table 5, the incorporation of up to 5.0 phr of ALK into the silica-filled rubber compound increased the torque difference ($M_{H}-M_{L}$), i.e. the maximum torque minus the minimum torque. Further
increases in the ALK loading decreased the torque difference. It is also evident that the minimum torque (M_L) decreased with increases in the ALK loading. It has been reported that M_L corresponds to the filler-filler interagglomeration [11] and is a measure of the stock viscosity [12]. The incorporation of ALK into the silica-filled NR compound reduced the viscosity of the compound and facilitated a relatively strong interaction between the ALK and the silica filler, while the silica particles had a weaker tendency to interact with each other and form a filler-filler agglomeration. Consequently, the processability of the silica dispersion in the rubber phase became easier and increased the degree of silica dispersion.

Theoretically, the torque difference (M_H - M_L) represents the shear dynamic modulus, which is indirectly related to the total crosslink density of a rubber compound [10,13–15]. The total crosslink density is contributed by the sulphide crosslinks and physical crosslinks [16,17]. The addition of up to 5.0 phr of ALK into the silica-filled NR compounds increased the torque difference (M_H - M_L) of the silica-filled NR vulcanisates. This is clearly attributed to the action of the ALK, not only as a secondary accelerator, but also as filler dispersant. The ALK accelerated the sulphur reaction and enhanced the state of the sulphide crosslink. The ALK increased the degree of silica dispersion and formed the additional physical crosslinks by silica–NR attachments (which will be seen in FTIR result).

The reduction of the torque difference (M_H - M_L) after the 5.0 phr loading, i.e. the E/7.0 NR compound, is most probably due to the softening or lubricating effect of the excessive ALK, which caused a lower crosslink density. This can be attributed to the phenomenon of dissolving a part of the elemental sulphur and silica particles into the excessive ALK and, as a consequence, less sulphur and silica were attached to the NR chains. The ALK is a unique molecule which is structured by a non-polar hydrocarbon chain and polar terminal groups. The non-polar hydrocarbon chain dissolved in the non-polar NR, forming a monolayer and lubricating the NR; whereas the polar terminal groups together with other curatives and silica formed intermediate complexes that remained insoluble. The excessive amount of ALK caused a more pronounced lubricating effect, and produced boundary layers which coated and absorbed not only the curatives but also the silica filler inside the layers. Through this mechanism, the ability of sulphur to form a sulphide crosslink and silica to form a physical crosslink would be less. This explanation could be supported by a morphological study of the tensile fractured surface of the E/7.0 NR compound. The fractured surface seemed smoother than that of the D/5.0 NR compound due to excessive ALK loading. ALK was synthesized from the

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**Table 4**
The Wavenumbers of Functional Groups of Alkanolamide Molecule.

<table>
<thead>
<tr>
<th>Vibration</th>
<th>Wavenumber (cm⁻¹)</th>
</tr>
</thead>
<tbody>
<tr>
<td>O–H stretch</td>
<td>3370</td>
</tr>
<tr>
<td>Unsaturated (C=C)</td>
<td>3006</td>
</tr>
<tr>
<td>Saturated (CH₃)</td>
<td>2852</td>
</tr>
<tr>
<td>C=O stretch</td>
<td>1615</td>
</tr>
<tr>
<td>CH₃ Umbrella mode</td>
<td>1364</td>
</tr>
<tr>
<td>C-N stretch</td>
<td>1248</td>
</tr>
<tr>
<td>CH₂ rocking</td>
<td>719</td>
</tr>
</tbody>
</table>

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**Fig. 2.** The infrared spectrum of Alkanolamide.

**Fig. 3.** Scorch time (t₂) and cure time (t₉₀) of the silica-filled NR compounds at various ALK loadings.
RBDPS, a palm oil fraction. Palm oil has the effect of plasticizing or lubricating rubbers [18].

The crosslink density of the silica-filled NR vulcanizates was determined by the Flory—Rehner approach [Eq. (2)]. Fig. 4 shows the crosslink density of the vulcanizates at room temperature. The addition of up to 5.0 phr of ALK into the silica-filled rubber compound increased the crosslink density, and further increases in the ALK loading decreased the crosslink density. This observation is in line with that of the torque difference \((\text{M}_{\text{H}} - \text{M}_{\text{L}})\), which is an indirect measure of the crosslink density of the rubber vulcanizates.

### 3.3. Silica dispersion

The degree of silica dispersion in the NR phase can be determined quantitatively by equation [19,20],

\[
L = \eta_r - m_r
\]

in which: \(\eta_r = [M_{\text{L}}/M_{\text{H}}]\), \(m_r = [M_{\text{H}}/M_{\text{L}}]\), where \(M_{\text{L}}\) and \(M_{\text{H}}\) are the minimum and the maximum torques of the filled vulcanizates; \(M_{\text{L}}\) and \(M_{\text{H}}\) are the minimum and the maximum torques of the gum NR vulcanizates. Based on the previous study [21] (for NR gum compound vulcanized with the same semi-efficient formulation), \(M_{\text{L}} = 0.04\); \(M_{\text{H}} = 4.81\). The lower the value of \(L\) at a particular silica loading, the better is the silica dispersion in the rubber phase. The value of \(L\) for the silica dispersion in the NR phase is presented in Table 6.

Table 6

<table>
<thead>
<tr>
<th>Torque Property</th>
<th>NR-based compound</th>
</tr>
</thead>
<tbody>
<tr>
<td>Minimum torque, ((\text{M}_{\text{L}}), \text{dN.m})</td>
<td>A/0.0, 1.23</td>
</tr>
<tr>
<td>Maximum torque, ((\text{M}_{\text{H}}), \text{dN.m})</td>
<td>1.23</td>
</tr>
<tr>
<td>Torque diff., ((\text{M}<em>{\text{H}} - \text{M}</em>{\text{L}}), \text{dN.m})</td>
<td>9.11</td>
</tr>
</tbody>
</table>

From the data shown in Table 6, it can be seen that ALK caused a lowering in the value of \(L\). The higher the ALK loading, the lower is the value of \(L\). The lower value of \(L\) indicates better silica dispersion, which provides a greater available surface area for the silica-NR interaction. The greater the available surface area for the interaction, the higher is the total crosslink density [16,17]. However, as presented in Fig. 4, the total crosslink densities of the E/7.0 and F/9.0 silica-filled NR vulcanizates were lower than those of the B/1.0, C/3.0 and D/5.0 silica-filled NR vulcanizates, and even the former vulcanizates had lower values of \(L\). This phenomenon can be attributed to the dominant plasticizing effect of ALK. As discussed before, the excessive amount of ALK trapped the silica inside its layers, thus causing less formation of physical crosslinks.

### 3.4. Mechanical properties

Figs. 5–8 show the effect of ALK on the tensile, hardness and resilience of the silica-filled NR vulcanizates. It can be...
seen that M100 and M300 increased up to the maximum level (5.0 phr) and then decreased with further increases in the ALK loading. The results of the tensile strength and hardness also exhibited a similar trend. According to Hertz [22], Ismail & Chia [23], the modulus or stiffness/hardness and tensile properties are dependent only on the degree of crosslinking. According to Ignatz-Hoover & To [24], as the crosslink density of a rubber vulcanisates increases, the elastic properties, such as tensile modulus, hardness and tensile strength increase, whereas viscous loss properties, such as hysteresis, decrease. Further increases in the crosslink density will produce a vulcanisate that tends towards brittle behaviour. Thus, at a higher crosslink density, such elastic properties as mentioned earlier begin to decrease.

The enhancement in tensile modulus, tensile strength and hardness with up to 5.0 phr ALK loading is attributed to a higher reinforcement level of silica to natural rubber due to a better degree of silica dispersion or wetting in the NR phase, as well as the cure rate and cure state enhancements phenomenon of the silica-filled NR compound. This explanation is in line with the data in Table 6, and the SEM micrographs of the silica-filled NR vulcanisates. The micrograph of D/5.0 exhibits a homogeneous micro dispersion of silica as a result of the incorporation of ALK. According to Hamed [25], in order for a filler to give significant reinforcement, the size of its particles must be small (<1 μm) and well dispersed in the rubber matrix. The deterioration of those properties after 5.0 phr can be attributed to the lower degree of crosslink density and the more pronounced softening effect of the excessive ALK.

As presented in Fig. 7, the addition of 1.0 phr ALK into the silica-filled NR compound resulted in the vulcanisated B/1.0 NR compound having a higher elongation at break. Increasing the ALK loading caused a further increase in the percentage of elongation at break. This can be attributed to the function of the ALK as an internal plasticizer to the silica-filled NR system. According to Moneypenny et al. [24], a plasticizer is a compounding material that is used not only to improve the rubber compound processing but also to modify the physical properties such as hardness, flexibility or distensibility of the rubber vulcanisates, and one of the sources from which it is made is natural oil/fat [18]. However, ALK is a waxy, solid material, and is derived from RBDPS-Crude Palm Oil, a type of natural oil. Although ALK has a smaller molecular size compared to NR, that additive could provide a monolayer in the rubber compound which provides free volume, thus allowing more mobility/flexibility for the rubber chains. Increasing the ALK loading has the same effect as an increase in free volume that will enhance the flexibility or the extensibility of the silica-filled NR vulcanisates.

Fig. 9 shows the effect of ALK on the resilience of the silica-filled NR vulcanisates. Resilience is the ratio of energy released by the recovery from deformation to that required to produce the deformation [26]. According to Hofmann [27], and Ignatz-Hoover & To [24], the rebound resilience is enhanced to some extent as the crosslink density rises, and the resilience is related to the flexibility of the molecular chains; the more flexible the molecular chains, the better the resilience. The incorporation of ALK into the silica-filled NR compound enhanced the resilience of the silica-filled NR vulcanisates. Again, this can be attributed to the functions of the ALK as an accelerator and filler dispersant which enhances the total crosslink density, and as a plasticizer which improves the flexibility of the filled rubber chains.

3.5. Scanning electron microscopy (SEM) study

Scanning electron microscopy (SEM) was used to determine the silica dispersion in the NR matrix. Fig. 10 provides the SEM micrographs of the tensile fractured surfaces of the silica-filled NR vulcanisates with and without the addition of ALK: (A) A/0.0; (B) C/3.0; (C) D/5.0; and (D) E/7.0 at 300x magnification.

From the micrographs, it can clearly be observed that the presence of ALK improved the silica dispersion. The
dispersion of the filler is the least homogeneous in A/0.0, where large silica agglomerates can be observed. The SEM micrograph of A/0.0 also seemed relatively smooth compared to those of the others, which indicates that A/0.0 is less ductile than the others. However, the SEM micrograph for D/5.0 exhibits the greatest matrix tearing line and surface roughness. Better silica dispersion in the D/5.0 altered the crack path, which led to increased resistance to crack propagation, and thus caused an increase in elastic properties, such as tensile modulus, tensile strength, and hardness. The micrographs of the tensile fractured surfaces were in good agreement with the results obtained by Ismail and Mathialagan [28] as well as Nabil et al. [29] which reported that an increase in energy was responsible for the
roughness and the matrix tearing line of the fractured surface. However, the matrix tearing line and surface roughness of E/7.0 was smoother than those of D/5.0, which indicated the more pronounced plasticizing or lubricating effect of the excessive ALK.

3.6. Infrared spectroscopic study

Fig. 11 shows the results of the FTIR spectroscopic study on the vulcanisates of A/0.0 and D/5.0, the control and the control with 5.0 phr. of ALK. All the figures show that the C–H stretches above and below 3000 cm\(^{-1}\), indicating that the vulcanisates has saturated and unsaturated portions [7]. The strong bands occurred around 2954–2956 cm\(^{-1}\), 2916 cm\(^{-1}\) and 2848 cm\(^{-1}\), which are attributed to the symmetric and asymmetric stretching vibration of the C–H bonds based on CH\(_2\) and CH\(_3\). Also, the bands at 1538 cm\(^{-1}\) and 1456 cm\(^{-1}\) are related to the bending vibration of the methylene (CH\(_2\)) and methyl (CH\(_3\)) groups, respectively. The spectra clearly show the characteristic wavenumbers of the NR vulcanisates. These characteristic wavenumbers of the NR were also reported by Wang et al. [30] for starch/NR composites, and Ooi et al. [31] for oil palm ash filled NR vulcanisates.

Fig. 11(a) shows that the presence of the O–H group peak at 3380 cm\(^{-1}\) is related to the presence of silanol Si–OH. In addition, two peaks are observed, i.e. at 890 cm\(^{-1}\) and 1096 cm\(^{-1}\), which can be related to the Si–O–Si symmetric stretch, and Si–O–Si asymmetric stretch, respectively. The spectrum clearly shows the presence of silica in the NR vulcanisates.

The IR spectrum of D/5.0 (Fig. 11b) showed similar characteristic bands to that of A/0.0. In addition, new bands are observed, i.e. at 3830 cm\(^{-1}\) (O–H stretch), 1398 cm\(^{-1}\) (NO\(_2\) symmetric stretch), 1279 cm\(^{-1}\) (C–N stretch), and 666 cm\(^{-1}\) (NO\(_2\) bend), the occurrence of which are due to the effect of ALK on the silica filled-NR vulcanisates. It is also observed that the Si–O–Si asymmetric stretching peak has shifted towards a lower frequency (1095 cm\(^{-1}\)). This spectral feature indicates that there are strong interactions, not only between ALK and silica, but also between ALK and NR.

According to Limper [32], due to its low boiling point, alcohol, which is attached to the TESPT coupling agent, can be classified as a Volatile Organic Compound (VOC). During curing, the ethanol in the ALK is released, and the amine group becomes reactive and forms coupling bonds with the silanol Si–O group of silicas, instead of the non-polar hydrocarbon of ALK interacting physically through moment dipole interaction with the long chains of NR. By this mechanism, the silica-ALK-NR bonding/interaction is formed, and the probable mechanism for such an interaction is presented in Fig. 12.

4. Conclusions

Alkanolamide was synthesized from RBDPS-palm oil and diethanolamine. Alkanolamide can be utilized as a new additive in silica-filled natural rubber compounds. The incorporation of Alkanolamide enhanced the cure rate, and reduced the scorch time of silica-filled natural rubber compounds. The results also indicated that Alkanolamide can be utilized as a plasticizer to increase the elongation at break and resilience. The incorporation of Alkanolamide also improved the reinforcement level of silica to natural rubber compounds. The tensile modulus, hardness, tensile strength and crosslink density of the silica-filled natural rubber vulcanisates were enhanced, especially up to 5.0 phr loading.

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