



Synthesis of 2-Alkylbenzimidazole Moiety as a Novel Antioxidant and Its Effect on Physico-mechanical and Electrical Properties of Acrylonitrile Butadiene Rubber



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IN the present work, 2-propyl-, and 2-heptyl-, *1H*-benzo[*d*]imidazole were prepared by condensation reaction of *o*-phenylenediamine with *n*-butanoic acid and *n*-octanoic acid, respectively. The prepared products were characterized by FT-IR, ¹H-NMR spectroscopy and melting point. These products were incorporated into acrylonitrile butadiene rubber (NBR) composites with two different fillers (Silica and High Abrasion Furnace carbon black HAF) as an antioxidant additive with different concentrations from 1up to 2 phr as a comparison with 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ) as a traditional antioxidant. Their effects on the rheometric, physico-mechanical and electrical properties of NBR composites were evaluated. Thermo-oxidative aging was carried out for NBR composites and distribution of the prepared products observed by Scanning Electron Microscope (SEM).The results showed that the prepared products can act as highly efficient antioxidants in acrylonitrile butadiene rubber vulcanizates comparing with commercial antioxidant TMQ and revealed that there was enhancement in mechanical properties of NBR composites that containing the prepared products, as well. The results also illustrated that the optimum ratio from 2-alkylbenzimidazole incorporated into acrylonitrile butadiene rubber vulcanizates is 1.5 phr if compared with the same ratio from traditional antioxidant (TMQ).

Keywords: 2-Alkyl-*1H*-benzo[*d*]imidazole, 2,2,4-trimethyl-1,2-dihydroquinoline, Antioxidant, Physico-mechanical and electrical properties.

Introduction

Acrylonitrile butadiene rubber (NBR) has been widely used all over the world due to its coveted chemical and physical properties such as good processability, mechanical properties, gas impermeability, resistance to oil, fuel and greases due to the polar character of NBR by presence of the polar nitrile group and moderate cost. NBR is a copolymer composed of butadiene and acrylonitrile, also used as a strategic material in automotive rubber products industries and incorporated in manufacturing of seals, oil resisting rubber hose [1-4]. Rubber products undergo deterioration which is generally occasioned by oxygen, ozone, heat and dynamics

stress. Accordingly, there is a great effort to promote stability of rubber during processing, when exposed to the vulcanization thermal conditions and during the life time when exposed to the external environment.

A suitable technique was used to enhance the aging properties of rubbers by addition of antioxidants [5, 6]. Antioxidants protect the rubber materials from attack by air, heat, light and even ozone in the atmosphere. Herein, the commonly utilized antioxidants in polymers can be classified into two main classes, namely, hindered phenols and aromatic amines, depending on the nature of the matrix [7, 8]. Amine antioxidants are more common which perform their role by termination

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1622 (C=N). ¹H-NMR (DMSO- *d*₆) δ (ppm): 12.23 (s, 1H, NH, imidazole, exch. with D₂O), 7.09–7.47 (m, 4H, H_{ar}), 2.78–2.81 (t, 2H, -CH₂-(CH₂)₃), 1.25–1.78 (m, 10H, (CH₂)₃), 0.83–0.86 (t, 3H, CH₃).

Preparation and characterization of NBR composites

NBR composites with the formulations were prepared using a laboratory two roll mill. The rubber compounds were left overnight before vulcanization. All compounds were compressed and molded using a hydraulic hot press according to their respective cure time ($T_{c_{90}}$) that was determined through TA instruments, MDR one [Moving Die Rheometer], USA.

Mechanical properties of vulcanizates were determined according to the standard methods using an electronic Zwick tensile testing machine (model Z010, Germ-any), in accordance with ASTM D412 standard.

Thermal oxidative ageing was carried out at 90±1°C in an air circulating oven for different time periods according to ASTM: D 572-04, 2010. The reported results were averaged from a minimum five specimens.

The morphology of NBR composites were investigated by using scan electron microscope (SEM) Quanta instrument (model FEG250, FEI, Hillsboro, Oregon, USA). Samples were gold coated, and the electron microscope was operated at 10 kV of excitation potential. SEM was performed by mounting the polymer blends sample on standard specimens tube and then created with very thin layer of gold by deposition.

Dielectric Measurements

The permittivity ϵ' and the dielectric loss ϵ'' in addition to the electrical conductivity σ measurements were carried out using Novocontrol Alpha Analyzer (GmbH concept 40, $\tan \delta > 10^{-4}$), over a frequency range from 10⁻¹ Hz to 10⁷ Hz. The samples were placed between two gold plated electrodes (the upper electrode's diameter is 10 mm) of a parallel plate capacitor. This technique among others has the ability to probe molecular fluctuations and charge transport in a broad frequency range.

Results and Discussion

Effect of adding 2-Alkylbenzimidazole on physico-mechanical properties of NBR / Silica composites

2-propyl-, and 2-heptyl-, 1*H*-benzo[*d*]imidazole

were incorporated into NBR at different concentrations from 1 up to 2 phr. As well as comparing their effect with the commercial applied antioxidant in rubber products, as (2,2,4-trimethyl-1,2-dihydroquinoline [TMQ] shown in Table 1.

The mechanical properties of the polymeric composites are one of the most significant performance parameters in the practical application of rubber material. The variations in tensile strength and elongation at break were shown in Fig.1 and Fig. 2, respectively. From figure 1, it was obvious that the highest tensile strength was achieved for NBR vulcanizates containing 1.5 phr from 2-heptyl-1*H*-benzo[*d*]imidazole followed by 2-propyl-1*H*-benzo[*d*]imidazole and finally TMQ and this is due to increase of crosslinking density between NBR chains. In addition, it was clear that the values of tensile strength of the NBR vulcanizates increased slightly with incorporation of traditional and prepared antioxidant up to 1.5 phr and then decreased with increase of antioxidant content up to 2 phr. Figure 2 also illustrated that the increase in the concentration of antioxidants from 1 up to 1.5 phr led to slight increase in the elongation at break and then decrease at 2 phr.

Effect of adding 2-Alkylbenzimidazole as a novel antioxidant on thermal oxidative aging for NBR/Silica composites

Figure 3 shows the tensile strength of the vulcanizates before and after ageing. All the vulcanizates show fairly good resistance to aging at 90°C. The vulcanizates containing 2-propyl-, and 2-heptyl-, 1*H*-benzo[*d*]imidazole show good resistance when the aging time increased more than traditional antioxidant. Figure 4 shows the variation in elongation at break of the vulcanizates before and after ageing. The vulcanizates containing 2-heptyl-1*H*-benzo[*d*]imidazole show better retention in elongation at break after ageing while vulcanizates containing 2-propyl-1*H*-benzo[*d*]imidazole show slight less retention in elongation at break after ageing. In addition, the prepared antioxidants improve the aging resistance of NBR composites.

Effect of adding 2-Alkylbenzimidazole on physico-mechanical properties of NBR/HAF composites

Also, 2-propyl-, and 2-heptyl-, 1*H*-benzo[*d*]imidazole were incorporated into NBR containing HAF as black filler at different concentrations from

TABLE 1. Formulation and rheometer characteristic of NBR /silica vulcanizates.

Ingredients	TMQ			2-propylbenzimidazole			2-heptylbenzimidazole		
	1 Phr	1.5 Phr	2 Phr	1 Phr	1.5 Phr	2 Phr	1 Phr	1.5 Phr	2 Phr
NBR	100	100	100	100	100	100	100	100	100
Basic recipe: ZnO 4, Stearic acid 2, Silica 20, Sulfur 2, TMTD 1, CBS 0.8, DOP 3 Phr									
Rheometer characteristic									
ML (d.Nm)	0.64	0.60	0.61	0.64	0.64	0.61	0.6	0.59	0.59
MH (d.Nm)	11.56	10.55	11.22	11.83	11.61	11.29	11.08	11.18	11.06
ts ₂ (min)	1.56	1.41	1.31	1.38	1.39	1.37	1.43	1.42	1.42
Tc ₉₀ (min)	4.48	4.15	4.13	4.32	4.52	4.59	4.37	4.55	5.00

Notes: M_L: Minimum torque M_H: Maximum torque
 ts₂: Scorch time Tc₉₀: Optimum curing time

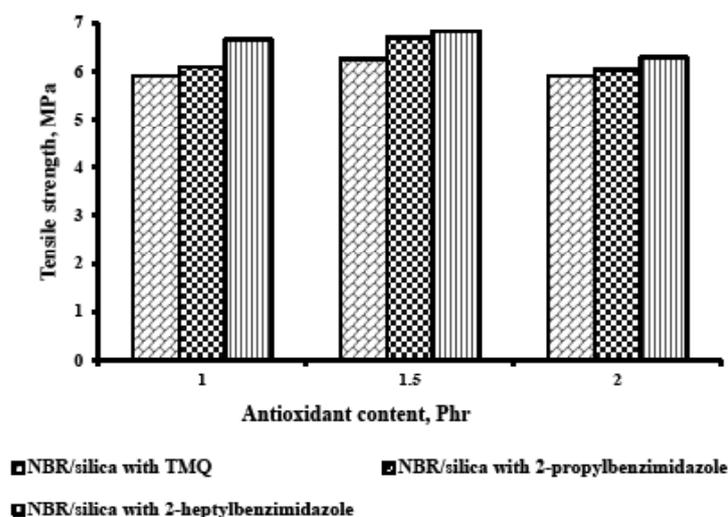


Fig. 1. Tensile strength for NBR/ Silica composites

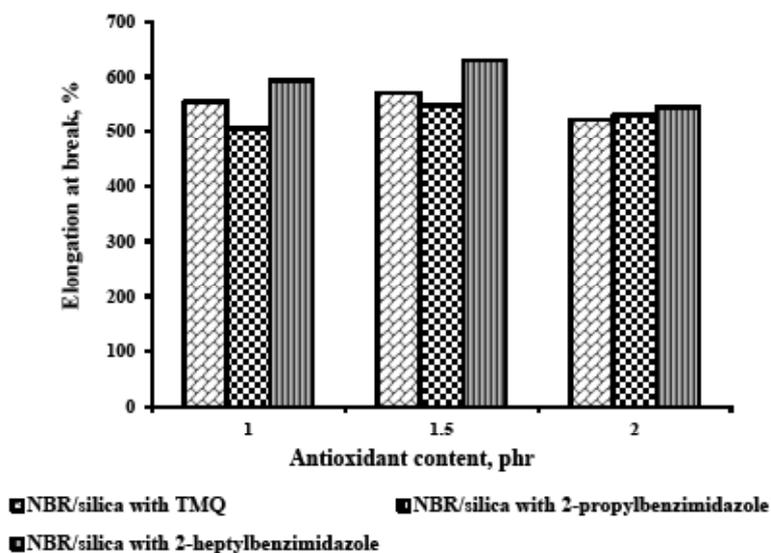


Fig. 2. Elongation at break for NBR/ Silica composites.

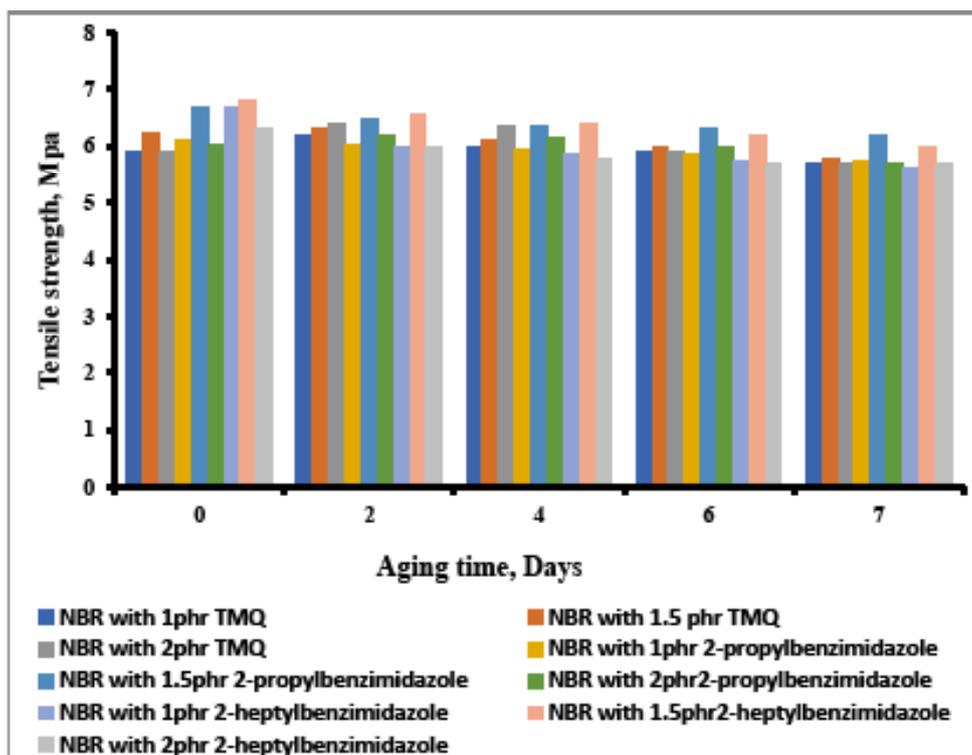


Fig. 3. Variation in tensile strength for NBR/Silica composites before and after thermal aging.

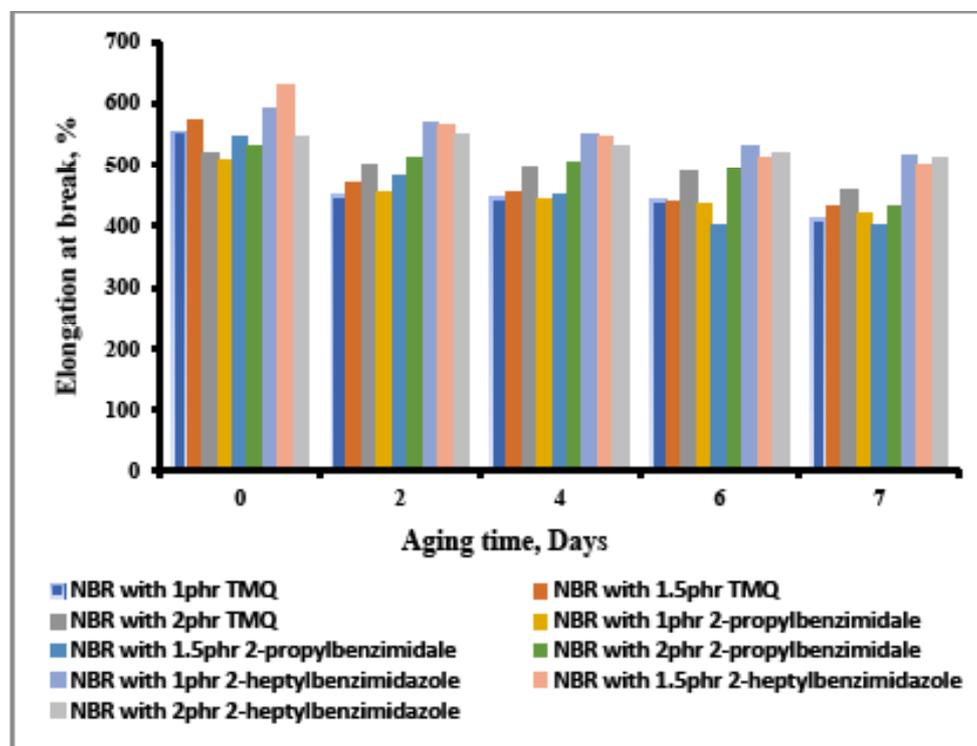


Fig. 4. Variation in elongation at break for NBR/Silica composites before and after thermal aging.

TABLE 2. Formulation and rheometer characteristic of NBR /HAF vulcanizates.

Ingredients	TMQ			2-propylbenzimidazole			2-heptylbenzimidazole		
	1 Phr	1.5 Phr	2 Phr	1 Phr	1.5 Phr	2 Phr	1 Phr	1.5 Phr	2 Phr
NBR	100	100	100	100	100	100	100	100	100
Basic recipe: ZnO 4, Stearic acid 2, HAF 30, Sulfur 2, TMTD 1, CBS 0.8, DOP 3 Phr									
Rheometer characteristic									
ML (d.Nm)	1.08	1.21	1.19	1.15	1.10	1.08	1.10	1.07	1.02
MH (d.Nm)	20.32	18.79	18.46	17.53	17.43	17.11	16.52	16.98	16.80
ts ₂ (min)	1.18	1.17	1.17	1.28	1.29	1.29	1.34	1.33	1.36
Tc ₉₀ (min)	6.30	4.49	5.8	5.0	5.40	6.13	5.14	5.55	6.51

Notes: M_L: Minimum torque M_H: Maximum torque
 ts₂: Scorch time Tc₉₀: Optimum curing time

1 up to 2 phr. As well as, comparison of their effect to the commercial applied antioxidant in rubber products, (2,2,4-trimethyl-1,2-dihydroquinoline [TMQ] shown in Table 2.

Similarly, the mechanical properties of NBR / HAF composites with different concentrations of different antioxidants displayed a slight increase in the tensile strength and elongation at break before thermal aging time were shown in Fig. 5 and Fig. 6, respectively. Also, increase the concentration of antioxidants from 1 up to 1.5 phr leads to slight increase in the tensile strength and elongation at break and then decrease at 2 phr, however, a slight increase in 2-propyl-1*H*-benzo[*d*]imidazole was shown than in 2-heptyl-1*H*-benzo[*d*]imidazole.

Effect of adding 2-Alkylbenzimidazole as a novel antioxidant on thermal oxidative aging for NBR/HAF composites

Also, figures 7, 8 show the variation in tensile strength and elongation at break of the vulcanizates containing HAF as black filler before and after ageing. These figures illustrate that all the vulcanizates show fairly good resistance to aging at 90°C. The vulcanizates containing 2-propyl-1*H*-benzo[*d*]imidazole and 2-heptyl-1*H*-benzo[*d*]imidazole shows good resistance when the aging time is increased more than traditional antioxidant. This again confirms that prepared antioxidants can improve the aging resistance of NBR composites

Phase morphology of NBR Vulcanizates

The change of morphology was monitored

by Scanning Electron Microscope (SEM). The morphology changes on the surface of samples before thermal aging for 1.5 phr antioxidants were obtained by Scanning Electron Microscopy (SEM) as shown in Fig. 9. The homogeneous distribution of ZnO, sulfur, filler, accelerator particles and antioxidants particles are very important for the activation of sulfur vulcanization and the improvement of mechanical properties of elastomer matrix. For NBR/ silica/ antioxidants vulcanize, crosslinking system particles are not homogeneously distributed in the elastomer matrix for 2-propyl-1*H*-benzo[*d*]imidazole. Microsized agglomerates (several micrometers in size) are created. The agglomeration of particles causes their surface area to decrease, followed by a reduction of the interface between zinc oxide, sulfur, and the accelerator. Homogeneous distribution of NBR compound was observed for 2-heptyl-1*H*-benzo[*d*]imidazole. For NBR/ HAF/ antioxidants, it was found that, 2-heptyl-1*H*-benzo[*d*]imidazole displayed less degree of dispersion than 2-propyl-1*H*-benzo[*d*]imidazole. In addition, increase an alkyl chain length for antioxidants form C₃ to C₇ leads to create agglomeration in the rubber matrixes shown in Fig. 10.

Effect of adding 2-Alkylbenzimidazole on dielectric properties of NBR vulcanizates.

This work was extended to study the dielectric properties of NBR vulcanizate upon addition of 2-alkylbenzimidazole. Accordingly, the obtained results for the permittivity ϵ' and dielectric loss ϵ'' were illustrated graphically versus the applied frequency ranging from 10-1 upto 107

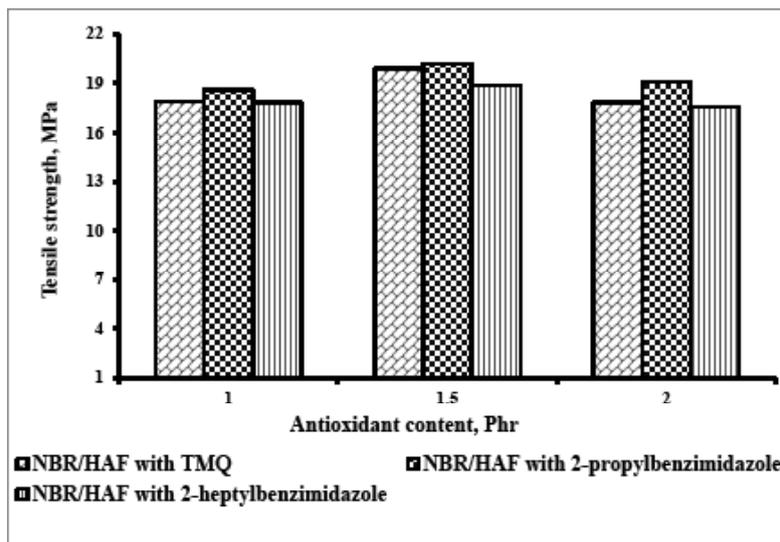


Fig. 5. Tensile strength for NBR/ HAF composites

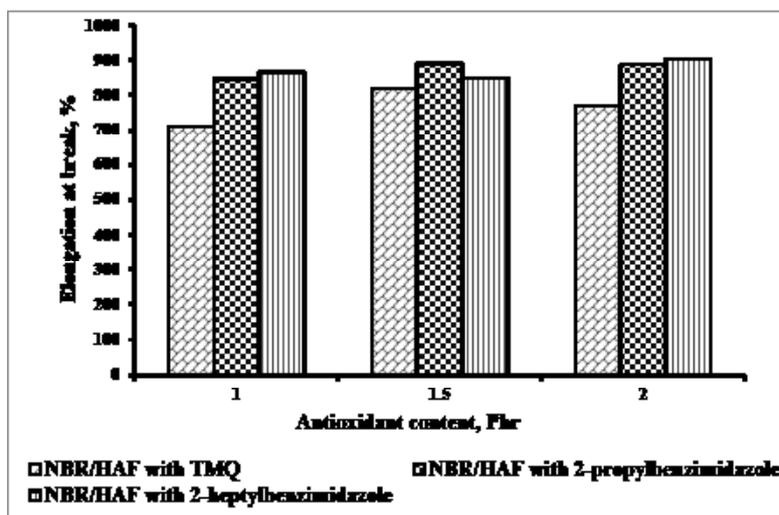


Fig. 6. Elongation at break for NBR/HAF composites

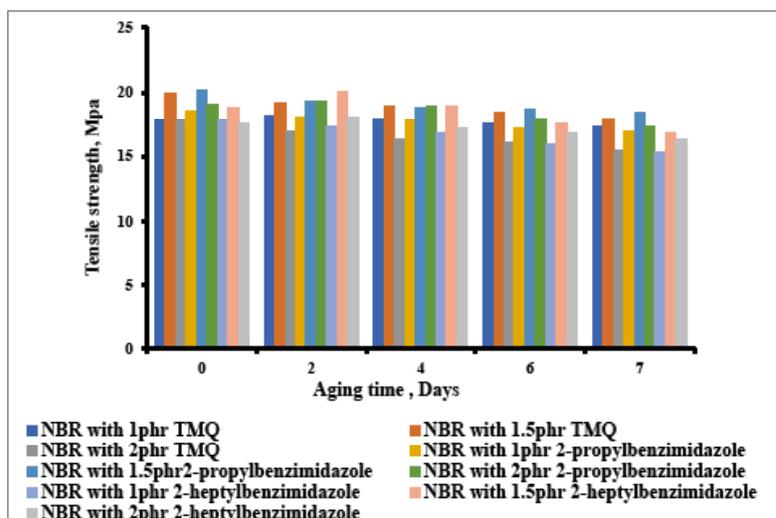


Fig. 7. Variation in tensile strength for NBR/HAF composites before and after thermal aging

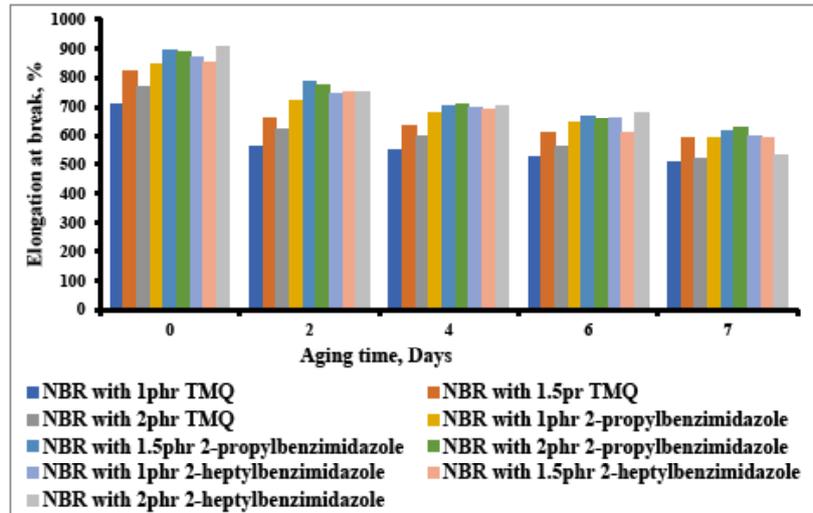


Fig. 8. Variation in elongation at break for NBR/HAF composites before and after thermal aging

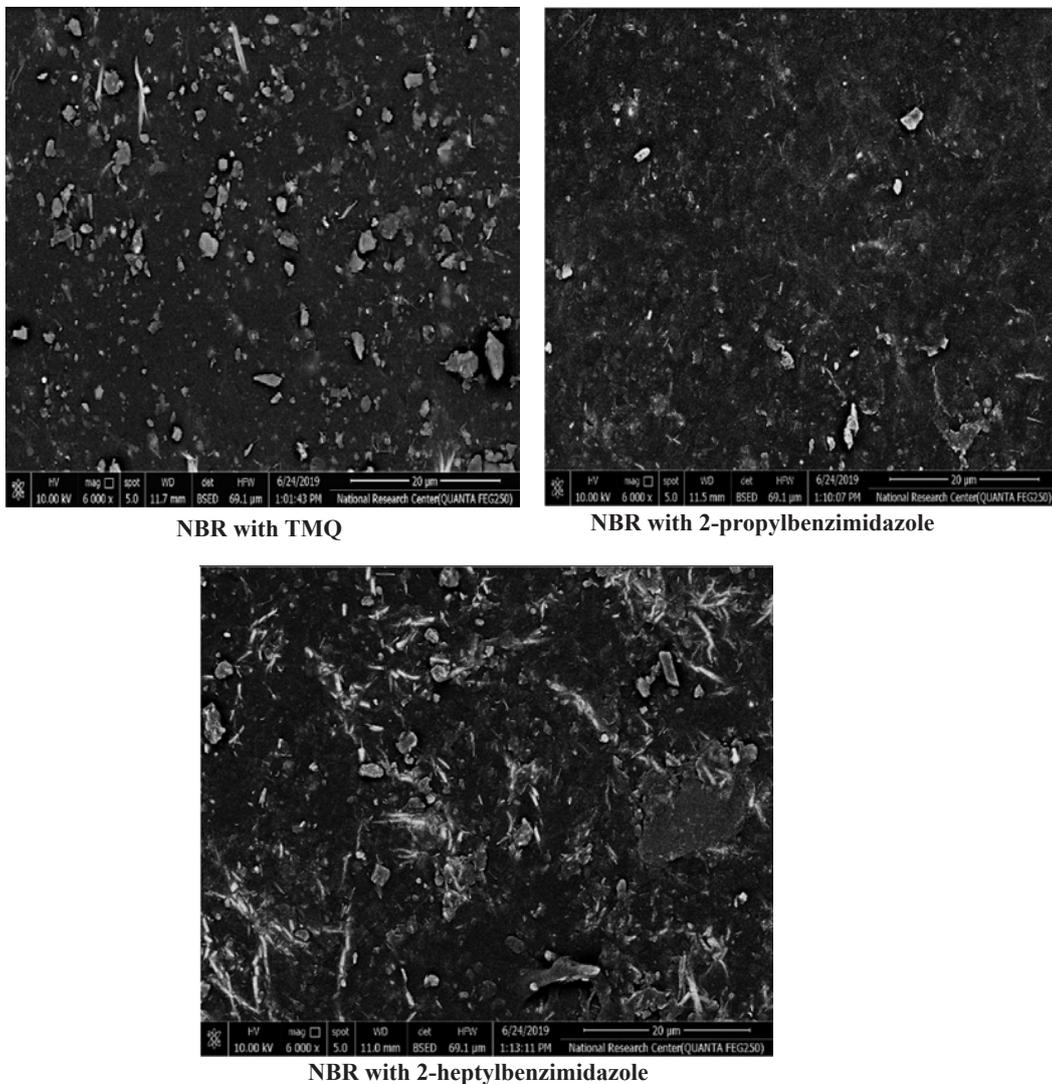


Fig. 9. SEM for NBR/ Silica vulcanizate.

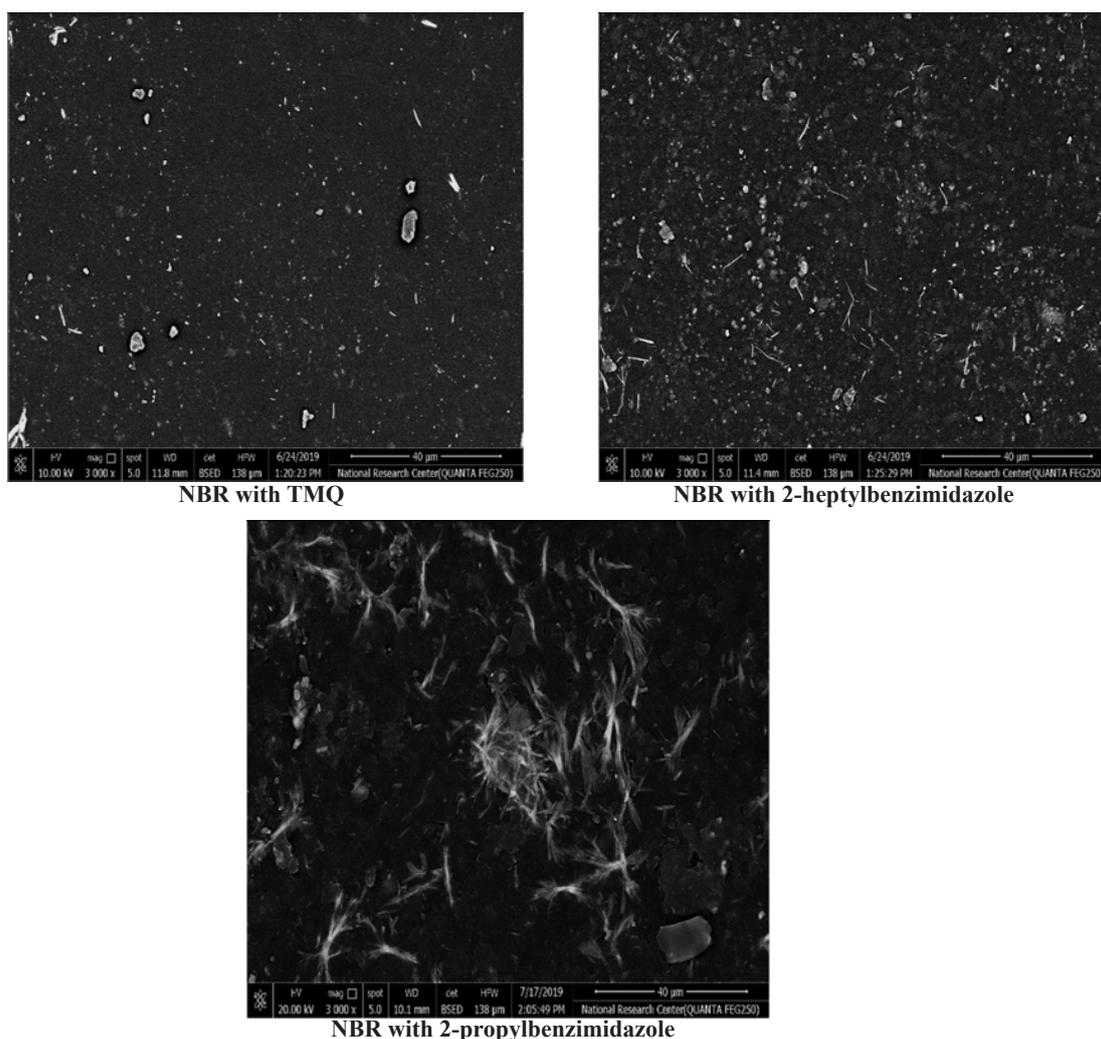


Fig. 10. SEM for NBR/ HAF vulcanizate.

Hz and at room temperature $\cong 25\text{ }^{\circ}\text{C}$ in Figure (11) for various composites with fixed amount of different prepared antioxidants including the 2, 2, 4-trimethyl-1,2-dihydroquinoline as a traditional antioxidant and with fixed amount of silica.

The permittivity ϵ' was found to decrease by increasing the applied frequency showing anomalous dispersion [12]. The decrease in ϵ' values at lower frequency could be due to either space charge effects [13] and or the interfacial polarization [14].

From this figure it is seen that the ϵ' values record the highest value for the traditional antioxidant followed by NBR with 2-propyl-1H-benzo[d]imidazole and the lowest value obtained for NBR with 2-heptyl-1H-benzo[d]

imidazole which shows inverse proportional with the number of carbon atoms in the molecule. This is logic due to the lower polarity of 2-heptyl-1H-benzo[d]imidazole with respect to 2-propyl-1H-benzo[d]imidazole

The dielectric loss ϵ'' as a function of the applied frequency was shown in Figure (11) illustrates a very complicated curves indicating more than one relaxation process. The higher values of ϵ'' at low frequency may be due to a combination between interfacial polarization and the dc electrical conductivity [15, 16].

Also, it is seen that at high frequency range a pronounced peak was detected. This relaxation process ascribes the movement of the side chain and the attached groups and found to be unaffected

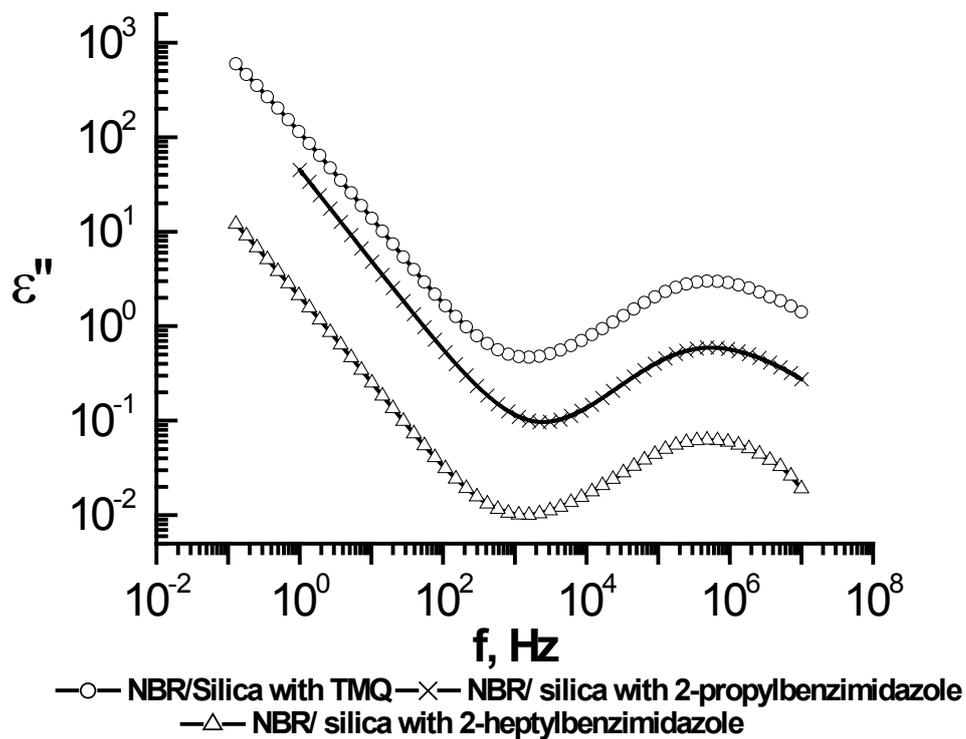
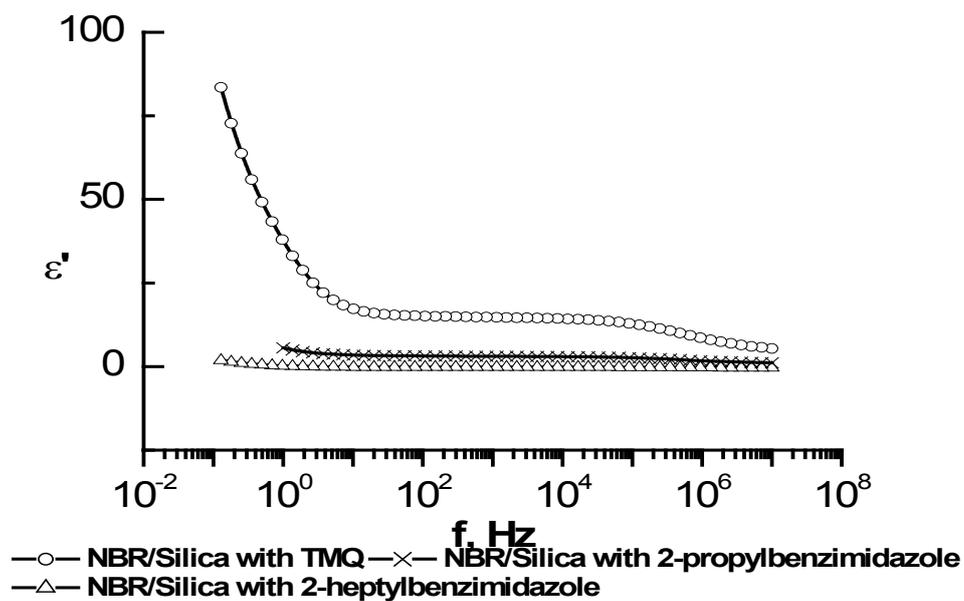


Fig. 11. The permittivity ϵ' and the dielectric loss ϵ'' of NBR/Silica vulcanizates with 1.5phr from different antioxidants

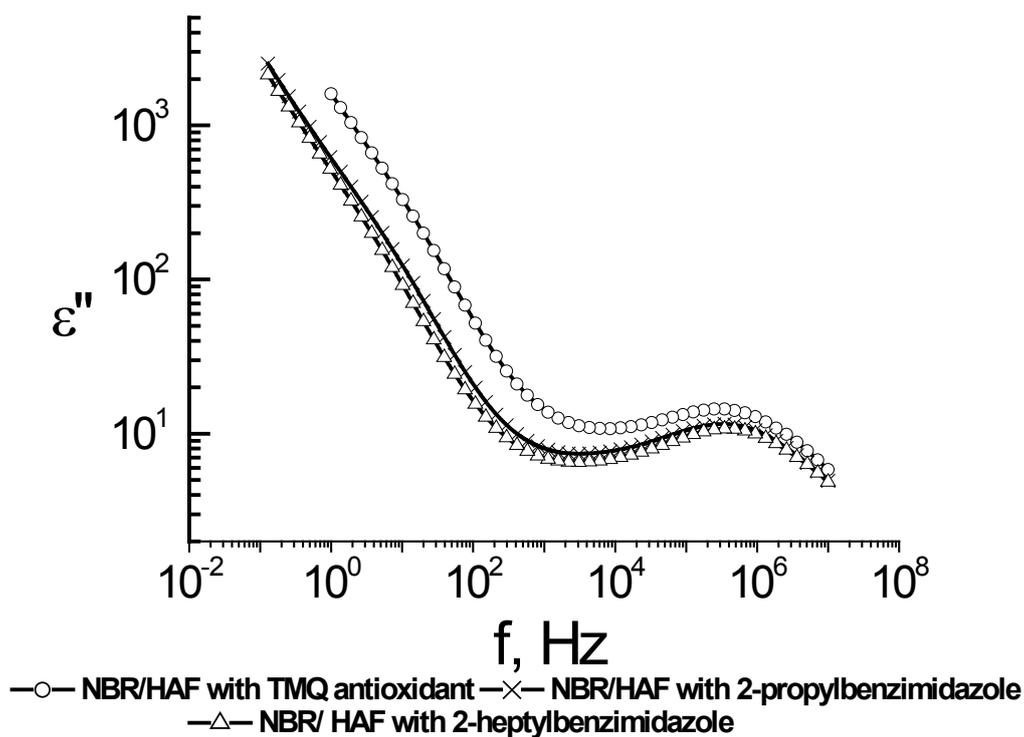
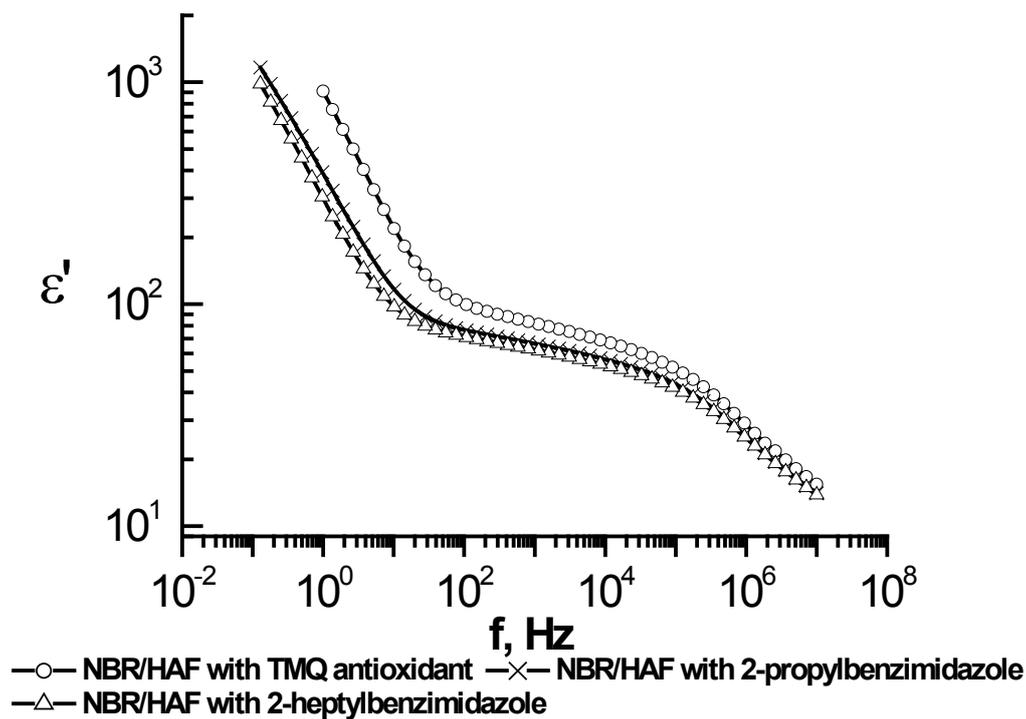


Fig. 12. The permittivity ϵ' and the dielectric loss ϵ'' of NBR/HAF vulcanizates with 1.5phr from different antioxidants.

by the type of antioxidant.

The permittivity ϵ' and the dielectric loss ϵ'' were measured for the same composites after replacing silica as reinforcing filler with HAF black one. The obtained data are given in Figure (12). Same trend is obtained but the values of ϵ' and ϵ'' are found to be higher when compared with those when silica was used as reinforcing filler.

The dc conductivity σ was calculated from the measured ac conductivity and the obtained data were illustrated graphically in Figure (13). From this figure it is obvious that the electrical conductivity σ follows the trend, TMQ > 2-propyl-1H-benzo[d]imidazole > 2-heptyl-1H-benzo[d]

imidazole which confirms the obtained dielectric data. Also it is clear that σ values are much higher for HAF as reinforcing filler when compared with those of silica. This finding is logic due to the conducting nature of HAF [17-19].

Conclusion

Acrylonitrile butadiene rubber (NBR) has various desired properties but low resistant to oxidation, thus, the incorporation of antioxidants into NBR enhance some of its properties. Series from 2-alkylbenzimidazole were prepared by condensation reaction and characterized by utilizing FT-IR, $^1\text{H-NMR}$ spectral techniques and melting points measurements.

The optimum ratio from 2-alkylbenzimidazole incorporated into acrylonitrile butadiene rubber

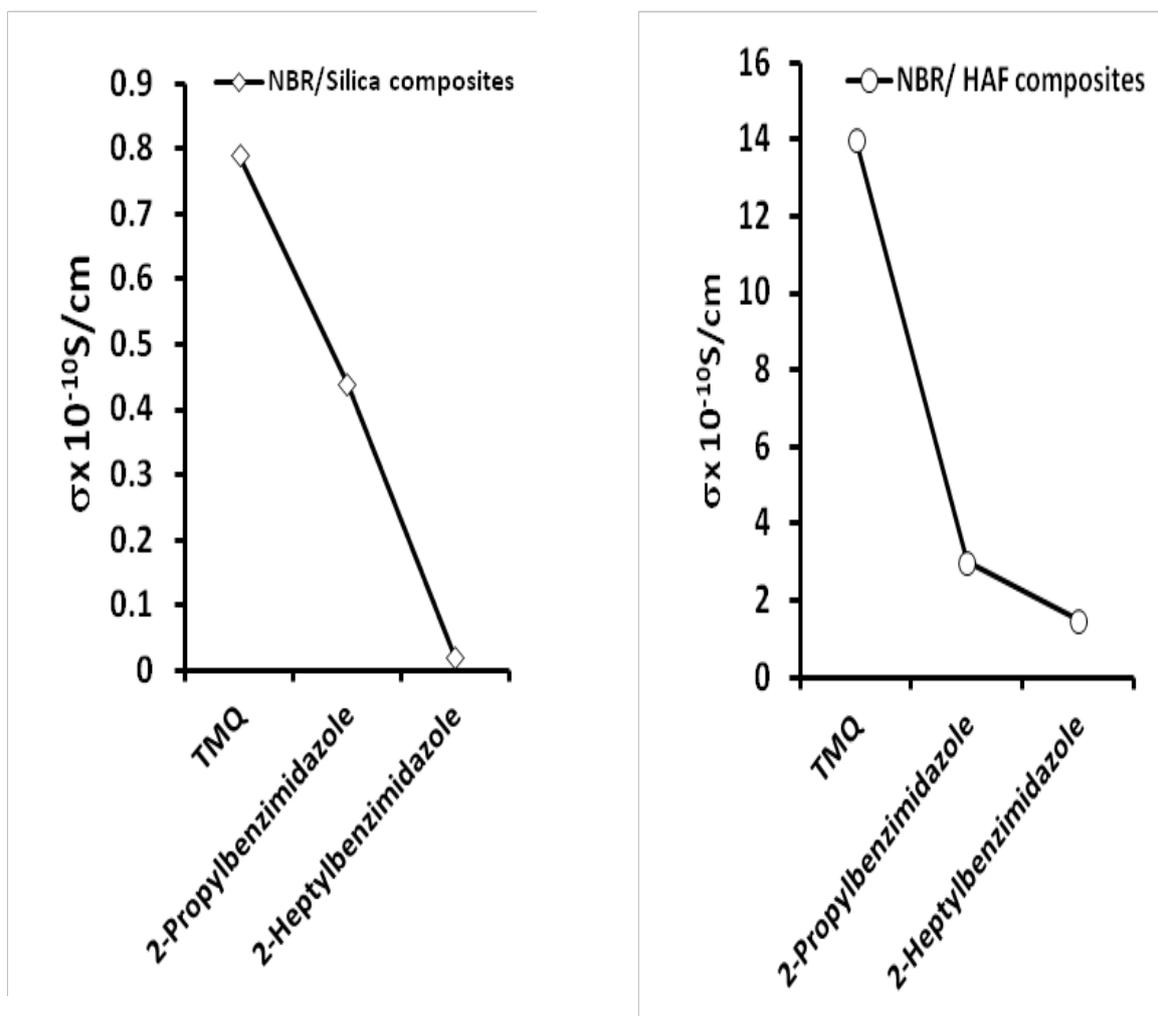


Fig. 13. Electrical conductivity versus antioxidant type for NBR composites.

vulcanizates is 1.5 phr if compared with the same ratio from traditional antioxidant (TMQ).

The vulcanizates containing 2-propyl-, and 2-heptyl-, 1*H*-benzo[*d*]imidazole show good resistance when the aging time increased more than traditional antioxidant.

The synthesized compounds can act as highly efficient antioxidants in acrylonitrile butadiene rubber vulcanizates as compared to TMQ, a commercial antioxidant.

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تحضير 2-ألكيل بنزايميدازول كمضاد أكسدة جديد وتأثيره على الخواص الفيزيقي ميكانيكية والكهربية لمطاط الأكريلونيتريل بيوتادين

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² قسم البلمرات والمخضبات - المركز القومي للبحوث - القاهرة - مصر

تتعرض منتجات المطاط إلى تدهور ملحوظ يحدث بشكل عام بواسطة الأكسجين، الأوزون والحرارة وكذلك الإجهاد الميكانيكي. لذا يضاف الي خلطات المطاط بعض المواد التي تلعب دورا هاما للحفاظ علي خواص المطاط الميكانيكية من التدهور أثناء التخزين أو التشغيل وهذه المواد تدعي المواد المضادة للأكسده و تضاف بكميات صغيرة من 1-3 ٪. كما يمكن تصنيف مضادات الأكسدة المستخدمة بشكل شائع في البوليمرات إلى فئتين رئيسيتين هما الفينولات المعطلة والأمينات الاروماتية وهذا يتوقف على طبيعة سلسلة البوليمر.

لذا فقد تناول هذا البحث تحضير كلا من 2 - بروبييل و 2 - هيبتييل - 1 - بنزو [د] إيميدازول بواسطة تفاعل تكثيف ارثو فينيلين داي امين مع حمض بوتانويك وحمض الأوكتانويك، على التوالي. كما تم اثبات التركيب الكيميائي للمركبات المحضرة بواسطة استخدام مطياف الأشعة تحت الحمراء و الرنين النووي المغناطيسي وكذلك ونقطة الانصهار. كما تم خلط المواد المحضرة داخل مطاط الأكريلونيتريل بيوتادين بنسب مختلفة (1- 1,5 - 2) الذي يحتوي علي نوعين مختلفين من المادة المألثة (السيلكا و اسود الكربون) وتم استخدام 2,2,4,4-ثلاثي ميثيل 2,1 ثنائي هيدروكينولين كمادة مانعة للاكسدة كمادة مرجعية.

كما تناول البحث دراسة تأثير اضافة المواد المحضرة علي الخواص الفيزيقي ميكانيكية (قوة الشد والاستطالة) وكذلك الخواص الكهربائية لخلطات المطاط المختلفة. علاوة على ذلك تم دراسة تأثير إضافة هذه المركبات على الخواص الميكانيكية بعد التعمير الحراري عند درجة 90 ± 1 درجة مئوية لفترات زمنية مختلفة (2- 4 - 6 - 7 أيام). كما تمت دراسة توزيع المركبات المحضرة داخل خلطات المطاط باستخدام الميكروسكوب الإلكتروني الماسح

وأظهرت النتائج أن المركبات المحضرة يمكن أن تعمل كمضادات أكسدة عالية الكفاءة في مطاط الأكريلونيتريل بيوتادين مقارنة بمضادات الأكسدة التقليدية كما اظهرت ايضا تحسن في الخواص الميكانيكية لخلطات المطاط التي تحتوي على المركبات المحضرة، كذلك. أوضحت النتائج أيضاً أن النسبة المثلى من 2-ألكيل بنزايميدازول المدمجة في مطاط الأكريلونيتريل بيوتادين تبلغ 1,5 جزء من المائة جزء إذا ما قورنت بنفس النسبة من مضادات الأكسدة التقليدية (TMQ).