



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكرو فيلم

بسم الله الرحمن الرحيم



MONA MAGHRABY



شبكة المعلومات الجامعية
التوثيق الإلكتروني والميكروفيلم



شبكة المعلومات الجامعية التوثيق الإلكتروني والميكروفيلم



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جامعة عين شمس

التوثيق الإلكتروني والميكروفيلم

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Synthesis, Characterization and Utility of Novel Organic Antioxidants for Some Rubber formulations

Thesis Submitted by

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B.Sc. (Chemistry) **2012**

M.Sc. (Chemistry) **2017**

For the requirement of Ph.D. Degree of Science in Chemistry

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**Synthesis, Characterization and Utility of Novel Organic
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Ayman Mohammed Mohammed Ali Hassan

Aim of work:

The presence of carbon-carbon double bonds in an organic compound leads to rubber materials susceptible to attack by oxygen and ozone, and to thermal degradation. Traditionally, acrylonitrile butadiene rubber (NBR) suffers from oxidative degradation by formation of free radicals along the polymer chains.

To delay this phenomenon for nitrile rubber, herein, the author was inspired to prepare 2-alkylbenzimidazole derivatives, and benzotriazole to be used as novel organic antioxidants for NBR. These antioxidants were characterized using FT-IR, ¹H-NMR, and melting points.

These novel antioxidants have been incorporated into NBR matrix at various concentrations and their activities were compared to a commercial antioxidant (TMQ) of the same concentrations. In addition, two different types of fillers were used in Nitrile matrix as silica and carbon black (HAF).

Further, the activities of these antioxidants have been evaluated by studying their effects on the physico-mechanical and electrical properties for nitrile rubber.

Abstract:

The present work is devoted to the preparation of novel different aromatic amine moieties and evaluation of their activity as antioxidants for acrylonitrile-butadiene rubber NBR.

In the first part, 2-propyl-, and 2-heptyl-, *1H*-benzo[*d*]imidazole (**3a,b**) were prepared by condensation reaction of *o*-phenylenediamine (**1**) with *n*-butanoic acid (**2a**) and *n*-octanoic acid (**2b**), respectively. The prepared products were characterized by FT-IR, ¹H-NMR spectroscopy and melting points. These products were incorporated into (NBR) composites with two different fillers (Silica and High Abrasion Furnace carbon black “HAF”) as antioxidant additives at different concentrations from 1 up to 2 phr as compared to 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ) as a commercial 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). Swelling and crosslinking density for NBR vulcanizates were evaluated and thermal gravimetric analysis (TGA) for NBR vulcanizates were also evaluated. 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-alkyl benzimidazoles incorporated into acrylonitrile butadiene rubber vulcanizates is 1.5 phr if compared with the same ratio from commercial antioxidant (TMQ).

In the second part, *1H*-benzo[*d*][1,2,3]triazole (**4**) as N- heterocyclic compound was prepared by ring closure reaction of diazonium salt of *o*-phenylenediamine (**1**) by nitrous acid. FT-IR, ¹H-NMR spectroscopy and melting point confirmed the successful preparation of *1H*-benzo[*d*][1,2,3]triazole. The main objective of this study is to investigate the probability of *1H*-benzo-[*d*][1,2,3] triazole to be used as a novel antioxidant for (NBR). The prepared product filled in NBR composite with different content starting

from 1 to 2 phr as compared to the commercial antioxidant (TMQ). The mechanical, thermo-oxidative aging and electrical properties of the composites containing the prepared product were compared with those of composites having commercial antioxidant in the same content. Distribution of the antioxidants into composites was observed by (SEM). Swelling and crosslinking density for NBR vulcanizates and (TGA) analysis for NBR vulcanizates were also evaluated. The results revealed that *1H*-benzo-*[d]*[1,2,3]triazole can act as highly efficient antioxidant in acrylonitrile butadiene rubber vulcanizates comparing with commercial antioxidant TMQ and showed that there was enhancement in mechanical properties of NBR composites, as well.

Keywords

2-propyl-*1H*-benzo-*[d]*imidazole, 2-heptyl-*1H*-benzo-*[d]*imidazole, *1H*-benzo-*[d]*[1,2,3]triazole, 2,2,4-trimethyl-1,2-dihydroquinoline (TMQ), Antioxidant, Physico-mechanical, Crosslinking density and Electrical properties.

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