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Effect of Adipic Acid on Mechanical Properties and Thermal Aging Resistance of EPDM/NBR Blends

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Abstract

This study examines the effect of adipic acid concentration on the physicomechanical and chemical properties of nitrile–butadiene rubber (NBR) and ethylene–propylene–diene monomer (EPDM) blends reinforced with carbon black. Four composite formulations were prepared with a fixed NBR/EPDM ratio of 70/30 phr, identical amounts of reinforcing fillers, activators, accelerators, and curing agents, while varying adipic acid contents at 0.0, 0.5, 1.0, and 3.0 phr. Mixing was performed on a laboratory two-roll mill, followed by vulcanization under optimized time–temperature conditions. Adipic acid was incorporated as a potential co-curing agent and compatibilizer to improve phase interaction between the polar NBR and non-polar EPDM phases. The influence of adipic acid on vulcanization characteristics, tensile strength, elongation at break, modulus, hardness, crosslink density, and thermal–oxidative aging resistance was systematically evaluated. The results showed that increasing adipic acid content modified the curing kinetics and improved interfacial adhesion, leading to notable enhancements in certain mechanical and aging resistance properties, particularly at moderate concentrations. However, excessive adipic acid levels resulted in a decline in performance due to overplasticization effects. These findings contribute to the optimization of hybrid elastomer formulations for automotive, sealing, and other industrial applications where enhanced durability and environmental resistance are required.

Keywords: NBR, EPDM, adipic acid, vulcanization, carbon black, rubber composites

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Adipin turşusunun EPDM/BNK qarışıqlarının mexaniki xassələrinə və qocalmaya qarşı davamlılığına təsiri

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Xülasə

Məqalədə adipin turşusunun butadien-nitril (BNK) və etilen-propilen kauçukuna (EPDM) fiziki-mexaniki və kimyəvi xüsusiyyətlərinə müxtəlif nisbətlərdə təsiri araşdırılmışdır. Dörd müxtəlif kompozisiya hazırlanmışdır ki, burada BNK/EPDM nisbəti sabit (70/30 phr), doldurucular, aktivləşdiricilər, sürətləndiricilər və vulkanizasiyaedici agentləri sabit miqdarda saxlanılmış, adipin turşusunun miqdarı isə 0.0, 0.5, 1.0 və 3.0 kütlə nisbətində dəyişdirilmişdir. Qarışıqlar laboratoriya vərdənəsində hazırlanmış, optimallaşdırılmış vaxt və temperatur şəraitində vulkanizasiya olunmuşdur. Adipin turşusu, doymuş BNK və doymamış EPDM fazaları arasındakı qarşılıqlı təsiri yaxşılaşdırmaq üçün potensial vulkanizasiyaedici agent və fazalararası uyğunlaşma təmin edilmişdir. Adipin turşusunun təsiri ilə qırılmada möhkəmlik həddi, nisbi uzanma, bərklik, qocalmaya qarşı davamlılıq və s. təsiri sisteməlik şəkildə araşdırılmışdır. Nəticələr göstərmişdir ki, adipin turşusunun artan miqdarı vulkanizasiya kinetikasını dəyişdirir və fazalararası yapışmanı yaxşılaşdırır, bu da xüsusilə orta konsentrasiyalarda bəzi mexaniki xüsusiyyətlər və qocalmaya qarşı müqavimətin əhəmiyyətli dərəcədə yaxşılaşmasına səbəb olur. Lakin, adipin turşusunun həddindən artıq miqdarı plastikləşmə təsirinə güclənməsinə gətirib çıxarır. Burada, avtomobil sənayesi, möhürləmə və digər sənaye sahələrində tətbiq olunan hibrid elastomerlərin mexaniki dayanıqlığının və ətraf mühit amillərinə qarşı uzunmüddətli stabilliyinin artırılması məqsədilə uyğun kompozisiya materialı hazırlanmışdır.

Açar sözlər: BNK, EPDM, adipin turşusu, vulkanizasiya, texniki karbon, rezin kompozisiya

Влияние адипиновой кислоты на механические свойства и стойкость к старению смесей СКЭПТ/БНК

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Аннотация

В статье исследуется влияние адипиновой кислоты на физико-механические и химические свойства бутадиен-нитрильного (БНК) и этиленпропиленового каучука (ЭПДМ) в различных пропорциях. Были приготовлены четыре различные композиции, в которых соотношение БНК/ЭПДМ поддерживалось постоянным (70/30 мас.ч.), наполнители, активаторы, ускорители и вулканизирующие агенты оставались постоянными, а количество адипиновой кислоты варьировалось в массовом соотношении 0,0, 0,5, 1,0 и 3,0. Смеси готовились в лабораторном смесителе и вулканизовались при оптимизированных временных и температурных условиях. Для улучшения взаимодействия между фазами адипиновой кислоты, насыщенной БНК и ненасыщенного ЭПДМ были предложены потенциальный вулканизирующий агент и межфазная совместимость. Было систематически исследовано влияние адипиновой кислоты на прочность при растяжении, относительное удлинение, твердость, стойкость к старению и т. д. Результаты показали, что увеличение количества адипиновой кислоты изменяет кинетику вулканизации и улучшает межфазную адгезию, что приводит к значительному улучшению некоторых механических свойств и стойкости к старению, особенно при средних концентрациях. Однако избыточное количество адипиновой кислоты приводит к усилению пластифицирующего эффекта. В данной работе был получен подходящий композиционный материал для повышения механической прочности и долговременной устойчивости к воздействию факторов окружающей среды гибридных эластомеров, используемых в автомобильной промышленности, производстве уплотнителей и других отраслях промышленности.

Ключевые слова: БНК, СКЭПТ, адипиновая кислота, вулканизация, технический углерод, резиновая композиция.

Introduction

Nitrile–butadiene rubber (NBR) is a high-performance synthetic elastomer widely recognized for its excellent resistance to petroleum-based oils, fuels, and a broad range of chemicals, as well as its favorable mechanical strength, abrasion resistance, and resilience. These attributes make NBR a preferred material in automotive seals, gaskets, fuel hoses, and other applications demanding oil resistance and mechanical integrity. Nevertheless, NBR suffers from inherent limitations such as low resistance to weathering, ozone attack, and prolonged exposure to elevated temperatures, which restrict its performance in outdoor and high-temperature environments [1].

Ethylene–propylene–diene monomer (EPDM) rubber, on the other hand, is valued for its outstanding resistance to ozone, ultraviolet radiation, oxidation, and thermal degradation, alongside good flexibility over a wide temperature range. EPDM's saturated backbone structure contributes to its exceptional weatherability; however, its non-polar nature results in poor compatibility with polar rubbers and limited oil and fuel resistance.

Blending NBR with EPDM offers a strategy to combine the advantages of both elastomers, producing hybrid materials that balance oil resistance, weatherability, and mechanical durability. Yet, the primary challenge in NBR/EPDM blends arises from the inherent incompatibility between the polar nitrile groups of NBR and the non-polar saturated backbone of EPDM. This mismatch often leads to coarse phase separation, weak interfacial adhesion, and suboptimal property balance.

To address these issues, compatibilization techniques have been explored, including the

incorporation of reactive additives, coupling agents, and co-curing systems. Adipic acid, a bifunctional aliphatic dicarboxylic acid, has potential as both a compatibilizer and co-curing agent. Its reactive carboxyl groups can interact with polar NBR chains and potentially participate in sulfur vulcanization, while its hydrocarbon backbone may enhance interaction with non-polar EPDM domains. Such dual compatibility may promote finer phase dispersion and improved interfacial bonding, ultimately enhancing mechanical performance and aging resistance [2].

This study systematically investigates the influence of adipic acid concentration on the vulcanization characteristics, tensile properties, hardness, crosslink density, and thermal -oxidative aging resistance of NBR/EPDM composites reinforced with carbon black. Four formulations were prepared with identical NBR/EPDM ratios and fixed amounts of curing agents, while varying adipic acid content (0.0, 0.5, 1.0, and 3.0 phr). The results are expected to provide insights into the formulation optimization of hybrid elastomers for demanding industrial applications, particularly in the automotive, sealing, and outdoor equipment sectors where enhanced durability and environmental resistance are critical.

Materials and methods

Nitrile–butadiene rubber (NBR, grade SKN-40) and ethylene–propylene–diene monomer rubber (EPDM, grade SKEPT-60) were used as base polymers [3]. Carbon black (type N-330) served as the reinforcing filler. Zinc oxide (ZnO) and stearic acid acted as activators, while ED-20 epoxy resin was incorporated to enhance adhesion and compatibility [5-6]. Sulfur was employed as the

curing agent, with 2-mercaptobenzothiazole disulfide (MBTS) and tetramethylthiuram disulfide (TMTD) used as primary and secondary accelerators, respectively. Adipic acid (99% purity) was added in varying concentrations (0.0, 0.5, 1.0, and 3.0 phr) to

investigate its role as a co-curing agent and compatibilizer. All chemicals were of industrial grade and used without further purification. The formulations of the four composite samples are presented in Table 1.

Table 1 – Composition of NBR/EPDM Composites with Different Adipic Acid Concentrations and Constant Filler and Curing System Content (phr)

Component	1	2	3	4
NBR (SKN-40)	70.0	70.0	70.0	70.0
EPDM (SKEPT-60)	30.0	30.0	30.0	30.0
Carbon black (C, N-330)	90.0	90.0	90.0	90.0
ZnO	5.0	5.0	5.0	5.0
Stearic acid	2.0	2.0	2.0	2.0
Adipic acid	-	0.5	1.0	3.0
ED-20 (epoxy resin)	3.0	3.0	3.0	3.0
Sulfur	3.6	3.6	3.6	3.6
MBTS	2.4	2.4	2.4	2.4
TMTD	1.9	1.9	1.9	1.9

The compounding process was performed on a laboratory two-roll mill (160×320 mm). Initially, NBR and EPDM were masticated for 3–4 minutes to achieve uniform blending. Carbon black, ZnO, stearic acid, and adipic acid (as per formulation) were then incorporated sequentially [7].

ED-20 epoxy resin was added to improve interfacial adhesion. After thorough mixing, the curing system (sulfur, MBTS, and TMTD) was incorporated at the final stage to minimize premature crosslinking. The total mixing time was approximately 15 minutes, and the batch temperature was maintained below 60 °C to prevent scorch.

Vulcanization

The compounded sheets were vulcanized in a hydraulic press at the optimum cure time (t_{90}) and temperature determined from rheometric analysis (Moving Die Rheometer, ASTM D5289). Standard test sheets of 2 mm

and 6 mm thickness were prepared for mechanical and aging tests, respectively.

Characterization

Cure characteristics were measured using a moving die rheometer at 160 °C to determine scorch time, optimum cure time, and torque values [8].

Mechanical properties (tensile strength, elongation at break, modulus) were tested according to ASTM D412.

Hardness was measured on the Shore A scale (ASTM D2240).

Crosslink density was determined via equilibrium swelling in toluene using the Flory–Rehner equation.

Aging resistance was evaluated by subjecting vulcanized samples to hot air aging at 100°C for 72 h (ASTM D573), followed by re-testing of mechanical properties.

Table 2 – Cure Characteristics, Mechanical Properties, Hardness, Crosslink Density, and Aging Resistance of NBR/EPDM Blends with Different Adipic Acid Contents

Adipic Acid (phr)	Scorch Time (min)	Optimum Cure Time t_{90} (min)	Max Torque (dN·m)	Tensile Strength (MPa)	Elongation at Break (%)	Modulus (MPa)	Hardness (Shore A)	Crosslink Density (mol/m ³)	Tensile Retention After Aging (%)
-	4.5	12.0	15.0	14.0	280	5.0	68	3.5	65
0.5	4.8	11.5	16.2	15.5	300	5.4	70	3.8	75
1.0	5.2	10.8	17.0	16.8	320	5.8	72	4.1	82
3.0	5.0	10.5	16.0	15.0	290	5.2	71	3.7	70

Results and Discussion

Cure Characteristics

The cure characteristics of the NBR/EPDM blends [10] with varying adipic acid content are summarized in Figure 1 and Table 2.

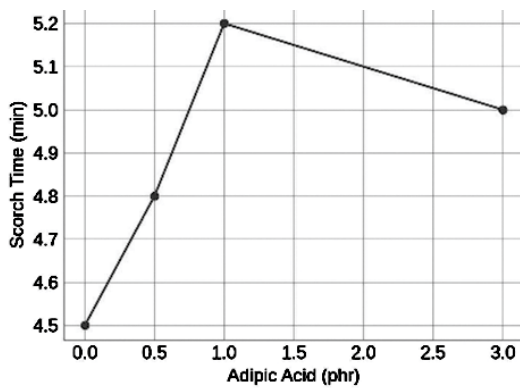


Figure 1 – Effect of adipic acid content on tensile strength of the rubber compound

Scorch time showed a slight increase from 4.5 min (0 phr) to 5.2 min at 1.0 phr adipic acid, indicating improved processing safety and delayed onset of vulcanization. The optimum cure time (t_{90}) decreased with increasing adipic acid, suggesting that adipic acid participates in the vulcanization network, accelerating crosslink formation at higher concentrations. Maximum torque, representing the crosslink density and stiffness of the cured rubber, increased from 15.0 dN·m to 17.0 dN·m at 1.0 phr adipic acid, confirming enhanced network formation. However, a slight decline at 3.0 phr

suggests possible over-curing or plasticization effects [11].

Mechanical Properties

The tensile strength and elongation at break increased with adipic acid addition up to 1.0 phr, reaching peak values of 16.8 MPa and 320%, respectively (Figure 2). This improvement is attributed to better interfacial adhesion and compatibilization between NBR and EPDM phases, likely promoted by adipic acid's bifunctional reactivity. Modulus also increased, indicating stiffer materials with improved crosslink density. At 3.0 phr adipic acid, a decrease in tensile strength and elongation was observed, possibly due to excess acid acting as a plasticizer or interfering with the crosslink network [9].

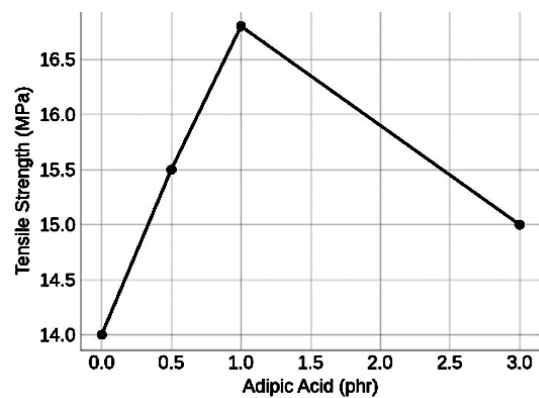


Figure 2 – Influence of adipic acid dosage (dose) on scorch time during vulcanization

Hardness and Crosslink Density

Hardness increased from 68 Shore A at 0 phr adipic acid to 72 Shore A at 1.0 phr, consistent with the rise in crosslink density measured by equilibrium swelling (Figure 3).

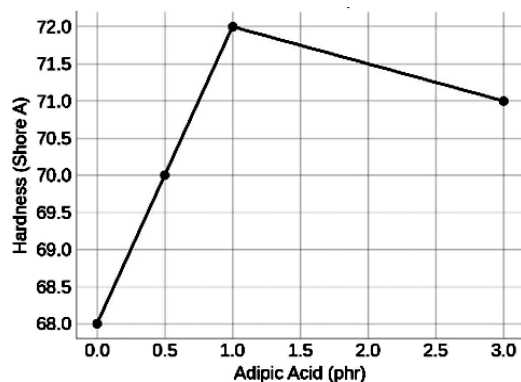


Figure 3 – Variation of hardness (shore A) with different levels of adipic acid

The increase in crosslink density confirms that adipic acid enhances the vulcanization process, creating a denser network. A slight decrease at the highest concentration indicates that excessive adipic acid may negatively impact network uniformity.

Aging Resistance

Tensile strength retention after thermal aging improved significantly with adipic acid content up to 1.0 phr, reaching 82% retention compared to 65% without adipic acid (Figure 4).

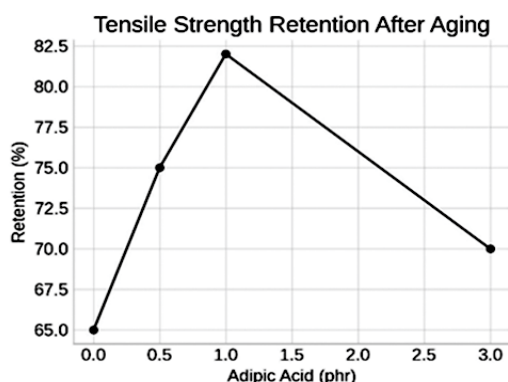


Figure 4 – Effect of Adipic Acid Content on Tensile Strength Retention after Thermal Aging of Rubber Compounds

This indicates enhanced resistance to thermal -oxidative degradation, likely due to the improved network structure and reduced phase separation. The decline at 3.0 phr suggests that excess adipic acid may adversely affect aging stability.

Conclusion

The present investigation elucidates the pivotal role of adipic acid as a co-agent in tailoring the mechanical and aging behavior of EPDM/NBR rubber blends. Systematic variation of adipic acid content revealed that incorporation up to 1.0 phr yields a substantial enhancement in tensile strength retention after thermal aging, reaching 82% compared to 65% for the unmodified blend. This improvement is attributed to the development of a more homogeneous and thermally stable crosslinked network, coupled with reduced phase separation between EPDM and NBR phases, which collectively mitigate thermal-oxidative degradation mechanisms.

However, further increases in adipic acid dosage to 3.0 phr resulted in a measurable decline in aging stability. This behavior is likely associated with excessive chain mobility, over-plasticization, or the formation of sub-optimal crosslink structures, which compromise the integrity of the polymer network under prolonged thermal exposure.

These findings underscore the necessity of optimizing co-agent concentration in blend formulations to achieve a balanced combination of initial mechanical performance and long-term durability [12-15]. The results not only provide insights into the structure-property relationships governing EPDM/NBR blends but also offer a practical guideline for designing elastomeric materials with superior aging resistance for demanding service environments.

Conflict of Interests

The authors declare there is no conflict of interests related to the publication of this article.

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