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EFFECT OF BLEND RATIO ON RHEOLOGICAL AND MECHANICAL PROPERTIES OF BUTADIENE RUBBER, ETHYLENE PROPYLENE DIENE RUBBER AND CHLORINATED PARAFFINS BLENDS

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Abstract	Keywords
In the present study, the influence of blend ratio	Rubber, compatibilizer,
(mass percentage ratio 100/0, 80/20, 60/40, 50/50,	blend, covulcanization, chloro-
40/60, 80/20, 0/100) on the curing efficiency and	paraffin
mechanical properties has been studied for two kinds	
of rubber blends, namely butadiene rubber + ethylene	
propylene diene rubber (BR/EPDM) and butadiene	
rubber + ethylene propylene diene rubber + chlorin-	
ated paraffin (BR/EPDM/CP). The efficiency of pro-	
posed compatibilization technique which includes	
two-roll mill mixing and vulcanization under the	
press is verified by the improvement of the mechani-	
cal properties of the blend vulcanizates. The compati-	
bility of blends was also evaluated in terms of their	
cure characteristics, rheological and mechanical	
properties. Rheometric characteristics (ML, MH, t_{c90} ,	
t_{s1} , and CRI) and Mooney viscosity were determined.	
The cure time t_{c90} of BR/EPDM/CP rubber blends	
increases with an increasing of the EPDM content in	
the polymer blend. The relatively shorter cure time	Received 13.12.2022
t_{c90} of BR/EPDM/CP indicates on better co-curing	Accepted 23.06.2023
compared to BR/EPDM rubber blends	© Author(s), 2023

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Introduction. The blending of two or more rubbers could be useful method to improve operational properties of elastomeric materials. It can also fulfill industry needs for materials with a complex of specific properties.

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In general, the elastomer blends show poor mechanical properties due to incompatibility and gross phase separation [1]. In addition to the low interfacial adhesion which is usually caused by the poor thermodynamic incompatibility, these blends mostly present cure rate incompatibility. This drawback stems from the fact that elastomers react with the curing agents in a different way. Another reason is the differences in solubility of the curatives in different elastomer phases.

Butadiene rubber (BR) and its blend compounds have been broadly studied by far as they are used, e.g., in tires applications. Blending of ethylene propylene diene rubber (EPDM) with high-diene rubbers attracts a technological and scientific interest as these species are involved in many industry areas. Blends of high-diene rubbers combined with halobutyl rubber and EPDM can be applied in tire sidewalls production. The latter get more resistance to ozone oxidation and following decay. EPDM rubber has improved resistance to ozone and ageing comparing to natural polyene or synthetic rubber, where double bond ozonolysis has crucial effect. Moreover, ethylene propylene diene monomer (EPDM) rubber has hydrocarbon backbones without multiple bonds, it has good resistance with respect to oxidation [2, 3]. So, when ethylene propylene diene rubber is incorporated into BR blends it leads to considerable better heat and ozone resistance of the resulting blend. The analogous approach takes place in [4], where acrylonitrile butadiene and epichlorohydrin rubbers are combined to get a blend with equivalent properties of neat epichlorohydrin rubber. However, EPDM rubber has too poor solvent, oil and flame resistance which restricts its application. The latter was improved in [5] by using a substance based on synergic action of ammonium polyphosphate (APP)/pentaerythrotol and expandable graphite. Nevertheless, when there is a blend of two rubbers the additional questions are to be solved: curing consistency, adhesion, mechanical stability, solubility of the agent and curing in both phases. One of the ways to overcome curing inconsistency is the introduction of new reactive sites in the cure process which speeds vulcanization up and forms additional cross-linking.

It has been reported in the literature that rubber blends of EPDM with natural rubber (NR) and other diene rubbers have good ozone and chemical resistance. Moreover, the compression set is also reduced. Despite the blend incompatibility of EPDM and diene rubbers, blending of EPDM with highly unsaturated elastomers is an important objective. One of the main challenges here — the problem of compatibility of butadiene rubber (BR) with EPDM could be partially solved by additives. Maleic anhydride could also level up compatibility of EPDM with NR [6]. The non-compatibility of mixing EPDM rubber with other rubbers was overcome by the presence of polar group of the

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K.V. Sukhareva, I.A. Mikhailov, E.A. Mamin

filler and polymer [7]. Nevertheless, in study [8] it was shown that NR/EPDM and NR/Brominated EPDM Blends have poor compatibility. In [9] it was shown that the tensile strength of the blends decreased with lower percentage of EPDM. In addition, the compatibilizer (methacrylate-butadiene-styrene) improved the compatibility of the NR/EPDM blends. Thioacetate (EPDMTA) or mercapto groups (EPDMSH) were used in [10] as compatibilizer compound for nitrile rubber. Here, introducing of functionalized copolymers brought about an increasing of thermal aging resistance of the blends. In study [11] the influence of carbon black filler on the curing, mechanical properties were examined. Along with standard mechanical test and thermochemistry the authors assessed the apparent activation energy values (E_a) of degradation for NBR, EPDM and NBR/EPDM/CB rubber blend. Apparent activation energy for the NBR/EPDM/CB rubber blend composite is lower compared to E_a for the rubber based on NBR and EPDM. This fact illustrates easier formation of rubber blend. The data showed increasing of hardness and tensile strength and reducing elongation at break with CB content increasing for all NBR/EPDM blends. The patent [12] describes a polymer blend of an ethylene- α -olefindiene rubber and a conjugated diene which had a distinctive mechanical characteristic (tensile strength) and could possibly replace unblended EPDM rubber in automotive applications. The blend had mechanical characteristics which were close to EPDM rubbers, and also had excellent heat and weather resistance, quite low Mooney viscosity. In the [13], authors investigated the influence of titania nanoparticles on the morphology and properties of vulcanized natural rubber (NR)/ethylene-propylene-diene monomer (EPDM) blends were performed and modern DMA, TGA, TEM techniques were applied for the analysis. Thermogravimetric (TGA) results showed good thermal stability of EPDM blends for the samples with titania nanoparticles, the vulcanization process was however inhibited. Maleic-anhydride modified EPM was a substance which the authors of [14] used to modify mechanical features of the blend NR/BR/EPDM. The tensile and tear strength increased as a consequence of MAH-EPM compatibilizing effect. Homogeneous phase distribution was also found for MAH-EPM.

The question of to overcome too high cure rate mismatch and consequently obtain better covulcanization was boiled down mostly to the adding a new substance, e.g., the bis(diisopropyl)thiophosphoryl disulfide [15], transpolyoctylene rubber (TOR) [16] or liquid BR [17]. The approach of [15] used carbon black and silica along with additive, bis(diisopropyl)thiophosphoryl disulfide (DIPDIS) to avoid cure mismatch in covulcanization of elastomer blends constituting styrene-butadiene rubber (SBR) and ethylene-propylene-

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Effect of Blend Ratio on Rheological and Mechanical Properties of Butadiene Rubber...

diene (monomer) rubber (EPDM). Hopper [18] improved mechanical characteristics of EPDM/NR blends by modifying EPDM adding a set of N-chlorothiosulfonamides. Coran [19] improved cure compatibility for EPDM/NR blends by using maleic anhydride as modificator. The blends which were obtained exhibited higher tensile properties, fatigue life, reduced hysteresis and permanent set, which is probably due to independent EPDM crosslinking mechanism which takes place with the action on ZnO. Morrissey [20] reported that halogenation of EPDM in solution could be a factor of better cure compatibility. In [21], the authors used 3 accelerators of vulcanization: 2,2-dithiobis(benzothiazole) (MBTS), the combination of MBTS and tetramethyl thiuram disulfide (TMTD), and n-tert-butyl-2-benzothiazolesulfenamide (TBBS) and controlled the scorch time, mechanical characteristics, crosslink density. The investigation revealed that binary accelerator MBTS/TMTD was the most appropriate one.

In [22], the compatibilizing efficiency for NR–EPDM blends of various compatibilizers; namely butadiene rubber, chlorosulfonated polyethylene, polyvinylchloride, chlorinated rubber, maleic anhydride were compared, and it was shown that addition of compatibilizers with chlorine content enhanced mechanical features and compatibility. In our study, a chlorinated paraffin was taken for that purpose. This could level up the compatibility and speed up covulcanization of BR and EPDM.

Materials and methods. The way in which the blends are obtained in the present work includes three stages:

1. Milling the EPDM in a two-roll mill with or without chlorinated paraffin.

2. Adding NR to the blend and mixing two elastomers.

3. Putting the blend of two resins with additives (CB, ZnO, Stearic acid, MBTS and sulfur) under the press for vulcanization.

The blending was carried out on a laboratory two-roll mill (470 mm diameter and 300 mm working distance). The speed of the slow roll is 24 rev/min, with a gear ratio of 1:1.5. Rubbers were mixed in self-heating mode.

The vulcanization was carried out at 152 \pm 1 °C in an electrically heated press under a pressure of about 4 MPa for their optimum cure time t_{c90} .

Rheometric characteristics (minimum torque (ML), maximum torque (MH), t_{c90} , t_{s1} , and CRI) were determined according to (ASTM), D 1646, 1994, by using a *Monsanto* oscillating rheometer model 1009 (USA). Rheology investigation bring about the optimal degree of vulcanization which provides the rate of vulcanization.

Mechanical properties were examined using a tensile compression testing machine *Devotrans DVT GP UG 5* (Turkey) according to ISO 527–1:2012 at a rate of 100 mm/min (five measurements for each point). The rebound resili-

ence measurements were performed using a Schob pendulum following the standard ASTM D7121 testing method.

The work was carried out using the equipment of the Center of Shared Usage "New Materials and Technologies" of Emanuel Institute of Biochemical Physics, Joint Research Center of Plekhanov Russian University of Economics.

Butadiene rubber. Butadiene rubber (BR) is one of the most useful and second most produced rubber worldwide. Butadiene rubber (BR-1203Ti grade, PJSC SIBUR Holding, Russia) is essentially a polymerization product of butadiene in solution¹. The polymer contains no nitrosamines and substances that may become a source of nitrosamines.

Ethylene propylene diene rubber (EPDM). Ethylene-propylene-diene rubber (EPDM) is a terpolymer of ethylene, propylene, and a non-conjugated diene with relatively small amount of unsaturated carbon–carbon bonds in the side chain. EPDM (EPDM-60 grade, PJSC Ufaorgsintez, Russia) is a product of ethylene, propylene, and DCPD/ENB copolymerization. EPDM rubbers are amorphous polymers. EPDM's properties depend on the ethylene and propylene percentage in the polymer chain.

Compatibilization of EPDM/BR rubber blend. The compatibilization of EPDM/BR rubber blend was carried out on the first stage by mixing EPDM with 2 phr (mass parts per hundred mass parts of rubber) of chlorinated paraffin (CP) in a laboratory two-roll mill. Mean carbon formula of CP (CH_{1.30}Cl_{0.82}), indicates the approximate mean number of chlorine/ hydrogen atoms per carbon atom. Based on the mean carbon formula, 1 g of CP contains 0.695 g of Cl. Then the EPDM + CP was mixed with BR in the same mill. The speed of the slow roll is 24 rev/min, with a gear ratio of 1:1.5. Rubbers were mixed in self-heating mode.

Rubber compounds based on BR, EPDM and EPDM/CP were prepared according to the requirements of GOST 54554–2011 Standart rubber compouds. Materials, equipment, and procedure for mixing and preparing vulcanized sheets².

The blend mixes were vulcanized by using a hydraulic press operated at 4 MPa pressure at 150 \pm 1 °C. The rheological properties of the blends (ML, MH, t_{c90} , t_{s1}) were assessed according to ASTM D2084-95 by using a Monsanto oscillating disc rheometer R-100 (Monsanto Co., Akron).

 $^{^1}$ Ash mass content 0.3 %, volatile matter content, max 0.8 %; Mooney viscosity 39 \pm 4.

² Propylene mass content 42–55 %, ash mass content 0.2 %, DCPD mass content 5.8–7.2 %, Mooney viscosity 56–65.

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Mechanical properties were examined using a tensile-testing machine *Devotrans DVT GP UG 5* (Turkey) according to ASTM D412-98a at a rate of 100 mm/min (five measurements for each point). The components are presented in Table 1.

Table 1

Description of BR/EPDM and BR/EPDM/CP rubber blends in parts per hundred (phr) of rubber (concentration of each additive is given in parts per hundred (phr) of rubber, mass of reach rubber is given in percents; 100 % corresponds to the sum of rubber masses)

Component	BR/EPDM							
Component	100/0	80/20	60/40	50/50	40/60	20/80	0/100	
Component concentration, phr								
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	
Zink oxide	5.0	5.0	5.0	5.0	5.0	5.0	5.0	
MBTS	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
Sulfur	2.0	2.0	2.0	2.0	2.0	2.0	2.0	
Carbon black	40	40	40	40	40	40	40	
Additive concentration, phr								
СР	2.0	2.0	2.0	2.0	2.0	2.0	2.0	
Stearic acid	1.5	1.5	1.5	1.5	1.5	1.5	1.5	
Zink oxide	5.0	5.0	5.0	5.0	5.0	5.0	5.0	
MBTS	3.0	3.0	3.0	3.0	3.0	3.0	3.0	
Sulfur	2.0	2.0	2.0	2.0	2.0	2.0	2.0	
Carbon black	40	40	40	40	40	40	40	

Results and discussion. The most important crosslinking characteristic of rubber blends is the degree of covulcanization which could be assessed by some rheological characteristics such as t_{s1} , t_{c90} , ML, MH. The values of these parameters for BR/EPDM and BR/EPDM/CP are listed in Table 2.

Table 2

Rheometric and mechanical characteristics of BR/EPDM and BR/EPDM/CP rubber blends at 150 °C (mass of reach rubber is given in percents; 100 % corresponds to the sum of rubber masses)

Characteristic	BR/EPDM mass percentage ratio						
	100/0	80/20	60/40	50/50	40/60	80/20	0/100
Minimum torque, dN·m	16.0	16.5	14.8	14.0	14.3	14.3	10.3
Maximum torque, dN·m	42.8	45.0	43.3	39.3	42.8	42.8	48.5

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End of the Table 2

<u>Observatoristic</u>	BR/EPDM mass percentage ratio								
Characteristic	100/0	80/20	60/40	50/50	40/60	80/20	0/100		
Scorch time, min	5.0	5.0	4.5	5.5	5.0	5.0	5.5		
Cure time, min	13.0	15.0	15.0	19.0	25.0	35.0	55.5		
Mooney viscosity	66	70	70	72	72	72	72		
Vulcanization rate, %/min	12	10	9.5	7.4	5.0	3.3	2.0		
M100, MPa	1.6	2.1	2.3	2.9	3.0	3.3	3.9		
M200, MPa	3.7	4.3	5.0	6.2	7.0	7.4	9.0		
M300, MPa	6.3	7.7	8.5	10.6	11.9	11.9	12.0		
Tensile strength, MPa	18.0	16.2	15.5	14.5	14.4	14.4	18.0		
Elongation at break, %	530	420	410	360	360	350	300		
Tear resistance, kN/m	67	46	45	44	42	51	53		
BR/EPDM/CP									
Minimum torque (ML), dN \cdot m	16.5	16.0	16.0	15.5	15.0	14.5	13.0		
Maximum torque (MH), dN · m	43.0	42.8	46.5	48.7	50.1	52.3	58.5		
Scorch time, min	5.0	5.0	6.0	6.0	6.0	6.0	6.0		
Cure time, min	13.3	13.0	14.3	14.7	15.5	17.1	31.0		
Mooney viscosity	66	67	68	68	69	72	76		
Vulcanization rate, %/min	12	12.5	12.0	11.5	10.5	9.0	4.0		
M100, MPa	1.6	2.5	3.2	3.5	3.8	4.0	4.5		
M200, MPa	3.7	5.0	6.5	6.9	7.0	8.9	9.2		
M300, MPa	6.3	10.5	11.5	13.0	14.0	15.5	16.5		
Tensile strength, MPa	18.0	18.0	18.5	19.0	19.5	20.0	21.0		
Elongation at break, %	530	500	420	400	390	390	390		
Tear resistance, kN/m	70	69	65	63	60	60	55		

It is well known that the maximum torque value of rubber blend is an indication to the stronger interactions between blend components. As follows from the Table 2 the addition of the compatibilizer improves the rheometric properties. Namely, it leads to the increase in maximum torque and decrease in cure time. Increase of BR/EPDM/CP (50/50 blend ratio) maximum torque for 20 % compared to BR/EPDM (50/50 blend ratio). It could be caused by better curing compatibility and possible reactions between the chlorine groups of polymer blend segments. In addition, the interfacial adhesion is improved because the compatibilizer segments have covalent bonds which are in separate phases.

The cure time of BR/EPDM/CP rubber blends increase with an increasing of the EPDM content in the polymer blend. The relatively shorter cure time of BR/EPDM/CP indicates on better co-curing compared to BR/EPDM rubber blends. Furthermore, chlorine atoms in BR/EPDM/CP rubber blends can act as efficient crosslinking sites which readily react with zinc oxide to form ether-type crosslinks. The chlorines seem to increase the reactivity and solubility of sulfur in the rubber, resulting in the formation of sulfur crosslinks.

The different mechanical properties of EPDM/BR and EPDM/BR/CP blends with different blend ratios including Modulus 100, 200, 300 %, the tensile strength and elongation at break are displayed in Table 2 and partially presented in Figure.

The data show that the tensile strength depends on the ratio of blend components and the more EPDM content is in the blend the more the sample tensile strength increases. The maximum tensile strength is observed for pure EPDM rubber with 2 phr CP. Apparently, the stresses in the BR/EPDM/CP rub-



Tensile strength of the BR/EPDM (**•**) and BR/EPDM/CP (**•**) blends

ber blends at all given relative strain (M100, M300 and M500) were higher than those in EPDM/BR rubber blends, suggesting that the attachment of chlorine groups the polymer chains leads to an increase in tensile strength. The tear resistance of the vulcanized rubber mixture was determined in accordance with GOST 262–93 Rubber, vulcanized. Determination of tear strength (trouser, angle and crescent test pieces). Tear resistance of rubber is defined as the maximum force required to tear a test specimen in a normal direction to stress applying line. As could be seen from the Table 2 the tear resistance depends on the blend ratio, whereas maximum tear resistance is observed for 100/0 blend ratio BR/EPDM/CP and slightly lower for 80/20 blend ratio. Introduction to mixtures up to 2 phr of chlorinated paraffin leads to an increase in rubber resistance by an average of 5 % for individual rubbers and up to 50 % in the mixture. The increase in tear resistance can be attributed to additional crosslinks which are forming in the presence of chlorine atoms during the vulcanization. Moreover, an increase of the Mooney viscosity of the EPDM (from 72 to 76) due to addition of chlorinated paraffins results in an increase of the crosslink density of the EPDM phase in covulcanization. The more expressed acceleration of

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curing rate for mixtures of rubbers is probably boiled down to the C–Cl dissociation (327 kJ/mole) in BR/EPDM/CP system which levels up concentration of reactive sites for vulcanization whereas the C–H dissociation in BR/EPDM (413 kJ/mole) proceeds not so intensive. The mechanism of chlorine atoms influence needs further elucidation and the possibly involves another experimental technique, e.g., X-ray absorption near-edge spectroscopy (XANES).

Conclusion. The BR/EPDM and BR/EPDM/CP blends were prepared with different ratios (100/0, 80/20, 60/40, 50/50, 40/60, 20/80, 0/100) by a two-roll mill mixing technique. The compatibility of blends was also be evaluated in terms of their cure characteristics and mechanical properties. The introduction of compatibilizers with chlorine content (chlorinated paraffins, CP) to the BR/EPDM rubber blends leads to an increase in the curing rate of rubber compounds and an increase of their mechanical properties.

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