

Study of the Influence of Native Polymer Sulfur on the Rheological Properties of Rubber Compounds

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Abstract: Polymer sulfur, obtained from local raw materials, was synthesized in laboratory conditions. The improvement of the physical and mechanical properties of rubber compounds obtained on the basis of natural polymer sulfur has been studied. The physical and mechanical properties of the reference rubber mixture and the sulfur-based polymer rubber mixture were studied.

Keywords: Sulfur, standard, rubber, polymer sulfur, modifier, vulcanization, accelerator.

Introduction : From the literature, dedicated to determining the effect of the type of sulfur vulcanization system on porous rubbers, rubber compounds and vulcanizates were prepared and studied with the addition of three different vulcanization accelerators. As a result of the research, vulcanization properties of rubber compounds, including vulcanization curves, were obtained. The parameters of heat transfer coefficient, apparent density, compressive strength and equilibrium swelling level of the manufactured rubber compounds were determined. The resulting porous rubber samples were found to be 8-40% lighter (apparent density) than the standard. It was found that N-cyclohexyl-2-benzthiazolyl Sulfenamide should be selected as a vulcanization accelerator for porous rubbers because it exhibits a slow rate during the induction period and a high activity during the subsequent vulcanization process[1].

Sulfur is a vulcanizing agent for most rubber products. Special requirements are placed on its quality and chemical composition, which primarily include high purity of the product (minimum amount of harmful impurities, variable valency metals) and high dispersion level. These properties determine the vulcanization activity of sulfur, its distribution in rubber, technological and technical properties of rubber compounds and rubber [2].

Petro Kazakhstan Oil Products PKOP LLC is its use as a component of rubber compounds for the production of rubber products. use as a vulcanizing agent for the production of products allows to produce products at a low price, which ensures their competitiveness in domestic and foreign markets. Small technological additives in the composition of rubber mixtures have a significant impact on the formation of a set of physical, mechanical and operational properties of rubber technical products. These connections perform universal functions. They not only speed up the vulcanization process, but also have a great impact on the physical, mechanical, chemical and operational properties of rubber products [3].

There are several methods for producing polymeric sulfur, including feeding a reactor with powdered sulfur and bulk dicyclopentadiene (DCPD) in a (65-70):(30-35) mass ratio, mixing at 130-140°C, then adding 5% technical carbon and simultaneously cooled in air. Then the resulting mixture is crushed to a size of 0.17-0.25 mm [4-5].

In order to obtain polymer sulfur, 20 grams of sulfur was added to a laboratory reactor with a tightly closed lid, equipped with a thermometer, after mechanical mixing in a sulfur oil bath. At a temperature of 140 $^{\circ}$ C, sulfur gradually changed from a monoclinic state to a plastic state, at a temperature of 165 $^{\circ}$ C was delivered to, and aliphatic ether with a double bond was added to it in the amount of 1-10% by mass of sulfur. The reaction was carried out with gentle stirring for 30 min. During the reaction, the substance being added as a modifier was slowly added. As a result, a dark red-brown homogeneous mass with high viscosity was formed. The resulting mass was cooled in the open air.

In order to determine how much of the synthesized polymer sulfur was polymerized, 24 hours after the synthesis process, it was extracted by the soxhlet method, as a result, when 10% modifier was added, the amount of polymerized sulfur reached 71%. The obtained results are presented in Table 1.

No	Amount of Modifier (%)	Amount of polymerized sulfur (%)	Modifikatsya temperature
1	1%	12%	165 °C
2	2%	18%	165 °C
3	3%	24%	165 °C
4	4%	32%	165 °C
5	5%	45%	165 °C
6	6%	56%	165 °C
7	7%	63%	165 °C
8	8%	65%	165 °C
9	9%	68%	165 °C
10	10%	71%	165 °C

 Table 1. Results of the influence of the amount of modifier on the amount of polymerization

From the data of Table 1, it can be seen that when the amount of modifier is 1%, the amount of polymerized sulfur is 12%, when the amount of modifier is 45%, when the amount of modifier is 10%, the amount of polymerized sulfur is 71%. The resulting polymer sulfur can be used as a vulcanizing agent in the preparation of rubber compounds.

First of all, chemical analysis is carried out according to the required technical methods and guests before testing polymer sulfur in rubber compounds, in which the total sulfur content, acidity, heat tolerance and ash content are checked by indicators. After the results of these tests meet the technical standards, tests are carried out on rubber compounds of different brands. Considering the above, the chemical analysis of polymer sulfur was carried out and the results are presented in Table 2 below.

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		Indicators			
No	Test names	Required technical norm	In practice	Technical method	
1	Total sulfur content, %	≥ 92	96	ISO 8332	
2	Acidity, %	≤0.05	0.04	ASTM D4569	
3	Heat tolerance, 110 °C	≥72.00	82	ISO 8332	
4	Ash amount, $\% \leq$	≤0. 30	0.25	ASTM D4574	

Table 2. Physico -chemical properties of polymer sulfur analysis results

After the technical parameters of polymer sulfur were in accordance with all the required norms, it was mixed into the rubber mixtures and the effect on the physico-mechanical and rheological properties of the rubber was studied.

The synthesized polymer sulfur was prepared in laboratory conditions based on a standard recipe, a rubber mixture was compared with the technical parameters of the standard rubber mixture, and the results of the analysis of the physical and mechanical properties of the rubber mixture are presented in Table 3 below.

Table 3. Test results of the physical and mechanical properties of the standard rubbermixture and the polymer sulfur-based rubber mixture

Nº	Test names		Technical parameters of the standard rubber mixture	Polymeric sulfur based on technical indicators of the obtained rubber mixture
	Vulcanization process at 185 °C for 3			
1	minutes			
	T30/		43-57	46
	T60/s		55-69	61
	ML/ dN·m		0.8-1.8	1.15
	MH/ dN·m		12-20	14.08
2	Muni according to viscosity (1+4) 100 °C		41-56	45.68
3	Muni according to volcanism period scorch T 5 (127 °C)		14-30	23.49
4	Strength , \geq MPa		12	17.84
5	Relative elongation , \geq %		250	310
6	Hardness level (Shoru A)		55-65	62
7	Dispersion level, $X \ge$		4.5	6.5
8	Modulus of elasticity, \geq	100%	2.5	2.81
	Мра	300%	11.5	14.39

The above test experiments showed that the physical-mechanical parameters of the rubber compounds obtained on the basis of local polymer sulfur are improved and meet the required technical standards.

In conclusion, it was determined that the physico-mechanical parameters of the rubber compounds obtained on the basis of local polymer sulfur were improved, and it was considered that it was possible to see if they met the required technical standards. The synthesis conditions of polymer sulfur obtained in laboratory conditions were determined. The results of the influence of the amount of sulfur modifier on the amount of polymerization of the synthesized polymer were determined.

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