

Formulation of Composite Materials Containing Tengiz Sulfur-Oil Production Waste

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ABSTRACT

The description of additional secondary semi-finished products, or sulfur wastes, which occur because of the work of the oil production and refining industries, which are the main economic sector of the country, was considered in the given article. The negative impact of their vast territory on the environment, flora and fauna and the local population was represented in literary and practical form. It is scientifically proven that storage of sulfur waste in the open by enterprises in this area causes irreparable damage to human life. The ways of using the residues of oil production and refining sulfur as one of the main components of raw materials in the production of rubber products were studied. The results of the study of exemplary sulfur samples using modern physicochemical and electron-microscopic methods showed its applicability. The research was conducted on the serial formulations of rubber compounds for lightweight tire filler cord of Tengiz sulfur and tire tread. The results of the study of the effect of Tengiz waste sulfur on the physicochemical and technical-operational properties of breaker and tread rubber were presented. Tengiz sulfur reduces the amount of sulfur in the formulation without reducing the rate of vulcanization, which eventually leads to an increase in the quality of the rubber. In addition, it was found that the use of Tengiz sulfur allows regulating the elastic properties of rubber.

Keywords: oil production, sulfur waste, environment, life safety, rubber, technology.

INTRODUCTION

One of the most important tasks with a clear socio-ecological direction is the study of the state of the environment, forecasting its changes under the influence of anthropogenic impacts, and determining the safe levels of man-made ecological burdens. The importance of environmental issues and environmental activities is constantly growing, substances that are dangerous to humanity and the natural ecosystem are released into the environment and are constantly accumulating in its various elements in the modern world.

Environmental pollution is increasing due to the widespread introduction of energy-intensive and chemical technologies, production of new chemical products, increased international sales of chemicals and technologies, as well as insufficient

environmental control in almost all areas of human activity [Minister of Environmental Protection of the Republic of Kazakhstan, 2007].

Elemental sulfur from Kazakhstan's oil is a very valuable raw material for chemical companies. However, the bulk of the afore-mentioned chemical substance is still stored near oil production facilities. Sulfur in Tengiz, in the form of large solid blocks, is stored in specially equipped areas called "sulfur emissions". The huge amount of waste generated in oil production, i.e. sulfur (today more than 8 million tons of products are stored in the "sulfur emissions"), is of great concern, because under local climatic conditions, sulfur can be converted into many compounds.

The above-mentioned sulfur massifs are located in the sanitary protection zone of the Tengiz Gas Processing Plant, in the carbonated zone

under the influence of flue gases containing carbon, hydrogen and various metals. The direction of the wind towards the sulfur storage area increases its effect. At the junction of the atmosphere and sulfur, micro-regions of ventilation of different intensities appear along the entire surface of the sulfur massif. During strong winds, sulfur particles are spread over a considerable distance in the air basin. At the same time, they can sink to the surface of earth, water or react with other chemicals resulting in the transition to new harmful substances. Therefore, the main problems in oil production at Tengiz are contaminated soil, groundwater, the release of sulfur dust, as well as the release of sulfur sulfide into the atmosphere [Zharylkasyn et al., 2016a].

A number of methods for desulfurization of oil and oil products are known [ST RK 1052-2002, 2003]. Outdated technologies of oil desulfurization and irrational usage of secondary products have a negative impact on the ecological state of the country's environment. In this regard, the development and implementation of waste-free, low-waste and highly efficient technologies is especially relevant and promising. The technology of processed heat-resistant composite materials must comply with the provisions of the "Sanitary and epidemiological requirements for the establishment of sanitary protection zones of industrial facilities" [Minister of National Economy of the Republic of Kazakhstan, 2015] and the "Environmental Code of the Republic of Kazakhstan" [Parliament of the Republic of Kazakhstan, 2007].

The residues of oil production and refining technologies – the assessment of the environmental impact of sulfur as a secondary product, professional safety and safety of process equipment in the production of heat-resistant composite materials – technical rubbers can be the basis for the development of technology for the production of heat-resistant composite materials.

Sulfur is a vulcanizing agent for many rubber products, including tires. The ever-increasing demands on the quality of tires require the development of effective components of rubber compounds. Particular attention is paid to the development of vulcanizing agents [Zharylkasyn et al., 2015, Zharylkasyn et al., 2016b].

It should be noted that the problems of sulfur storage, which belongs to the IV hazard class, is still relevant today. This chemical causes inflammation of the mucous membranes of the eyes and upper respiratory tract, skin irritation,

and diseases of the gastrointestinal tract. In addition, the storage, accumulation, storage of sulfur in the open has a negative impact on the environment and human life [Babina, 2010].

Petroleum and associated petroleum gases contain up to 14% hydrogen sulfide. Hydrogen sulfide, separated from the oil supplied to the production site by associated gases, decomposes into water and sulfur in the Klaus installation. The separated liquid sulfur is directed to granulation or to tanks or sulfur waste storage facilities. Lump sulfur is sulfur stored in the form of blocks in waste dumps [Podavalov, 2010].

Today, the world's oil and gas refineries produce about 50 million tons of sulfur a year. As a result of the initial refining of oil from associated components, 1 million tons of sulfur are produced only at the Tengiz gas and oil refinery in the country [Speight, 2020].

Sulfur is a major vulcanizing agent for many composite compounds based on synthetic rubbers, including technical rubber products [Turebekova, 2016]. There are special requirements for its quality and chemical composition: the degree of purity and high degree of dispersion of the product. These characteristics determine the vulcanizing activity of sulfur, its dispersion in rubber, as well as technological and technical properties of rubber and rubber compounds. Polymeric sulfur allows reducing the amount of sulfur during the process without decreasing the rate of vulcanization, which leads to an increase in the quality of rubber. The usage of polymer sulfur allows adjusting the elastic properties of the rubber produced [Behera and Prasad, 2020, Nadirov, 2003, Otalbaev et al., 2019].

On the basis of the preliminary study of the physical and chemical properties of oil production and refining wastes and the activation of dispersing properties in the specified direction, a new technology for obtaining thermostable composite materials associated with using of Tengiz sulfur will improve the technical and economic performance of oil and refining industries; moreover, it has economic, social and environmental significance [Vetoshkin, 2019, Wandelt, 2018, Kalygin, 2004].

The main goal of the study was to eliminate the remains of oil production and processing – a heat-resistant composite material of sulfur – by using it as a vulcanizing agent in the process of producing technical rubber. In order to achieve this goal, the study set the following tasks: to

study the possibility of using sea sulfur in rubber compounds; composite material containing sea sulfur—research of technological and physical and mechanical properties of technical rubber.

MATERIAL AND METHODS

Research methods include physical, chemical, physical and mechanical analyses, as well as technical and operational tests. The physicochemical analysis of raw materials was carried out in a specialized laboratory for methods of physical and chemical analysis “Sapa”. The technical and operational test methods were carried out specifically in the “Complex laboratory of modern test methods” of the South Kazakhstan University named after M. Auezov. The preparation of the rubber mixture was carried out in the laboratory mixer PD 630315/315. The temperature of the front shafts of the shaft is 50–60°C and the back ones is 60–70°C.

The mixing was carried out on laboratory shafts with the following characteristics:

- Shaft diameter-160 mm.
- Shaft length -320 mm.
- Fraction - 1: 1.24.
- Drive power - 4.6-7 kW.
- Effective load - 1 kg (minimum).

During mixing, the following process parameters were observed:

- mixing time;
- shaft clearance;
- shaft surface temperature.

The cleaned and cut rubber passes through the shaft holes until a thin layer is formed. The mixture is often cut for high-quality plasticization, thereby the direction of the deformation force is changed. The procedure of adding the

ingredients was carried out in accordance with theoretical rules: first, emollients, accelerators, activators, plasticizers were added. Technical carbon was added several times in small amounts, scattered on a flat sheet technical carbon was re-added into the mixture. The vulcanizing agents were added at the end. The rubber compound was regularly cut during the mixing process. Ready mixtures were obtained from shafts in the form of rolled blanks.

The determination of salt hardness was carried out according to GOST 263-75 [1988]. The hardness of rubber is characterized by the resistance of the rubber to the compressive force of a metal needle or ball indenter under the action of a spring force or load. The hardness is determined by the depth of compression of the needle by the compressed spring when the base of the instrument touches the surface of the specimen. Pressing the needle moves the link proportionally on the instrument scale.

The test piece is a parallel surface plate. When measuring, the distance between the measuring points must be at least 5 mm, and the distance from any measuring point to the edge of the sample must be at least 13 mm. The sample thickness should be at least 6 mm. The test temperature should be $(23 \pm 2)^\circ\text{C}$. The hardness is measured at at least three points in different parts of the sample. The test result is the arithmetic mean of all measurements, rounded to the nearest whole number.

The tensile strength of the samples is determined according to GOST 261-79 [1980]. For testing, the samples are taken according to permits and marked in accordance with GOST 270-75 [2008]. The test pieces are cut from rubber plates with a thickness of 2 ± 0.2 mm or 1 ± 0.2 mm.

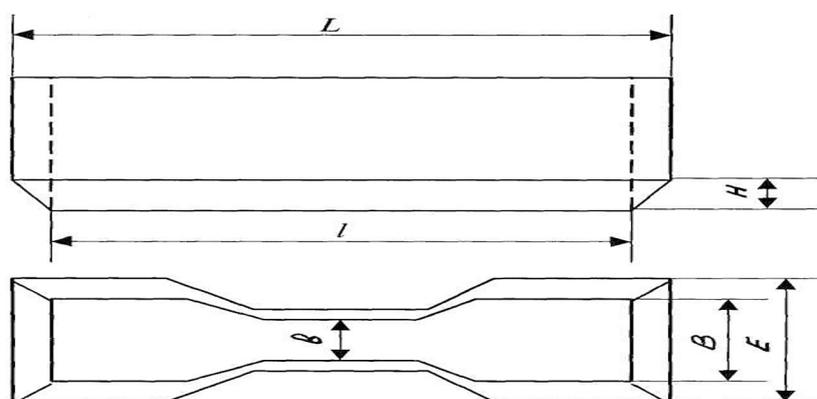


Fig. 1. Device for determining tensile strength and flexibility

The frequency of the deformation is set. The temperature in the chamber is brought to the set value. The amount of dynamic and static displacement of the clamps of independent machines in accordance with the specified deformations of the samples is set, which are determined by the length of the working zone. According to the calculation formulas, the values of the following indicators were determined:

$$f_z = R_z/S_0 \quad (1)$$

where: S_0 – initial state of the sample, m^2 ;

$$S_0 = b_0 \cdot h_0 \quad (2)$$

where: b_0 – initial width of the sample, mm; h_0 – the initial thickness of the sample, mm.

Relative elongation at break, E.

$$E_z = (l_2 - l_0)/l_0 \quad (3)$$

where: l_2 – the length of the working area of the sample at the breaking time, mm; l_0 – the initial length of the working area, mm.

Relative elongation at expansion, Q.

$$E_z = (l_1 - l_0)/l_0 \cdot 100\% \quad (4)$$

where: l_1 – the length of the working area of the sample in a minute after “rest”.

The tensile test method for rubber according to the GOST 262-93 [2002] consists of stretching a cut piece and measuring the load at which the test piece breaks. The tests are carried out on a cutting machine. The samples are cut with a special tool.

Durability of rubber to rupture Σ_z (N/m) of rubber is calculated using the following formula:

$$\Sigma_z = P_r/h_k \quad (5)$$

where: P_r – load causing rupture of the cut specimen, N; h_k – initial sample thickness, m.

RESULTS AND DISCUSSION

The experiments were carried out to determine the optimal amount of polymeric sulfur to obtain vulcanizate with the best usage properties. Polymeric sulfur is obtained from the melt during the sudden cooling of sulfur melted in a hardened medium. Polymeric sulfur was obtained in the form of a light yellow dispersed powder. Tengiz sulfur was added to the rubber mixture to partially or completely replace ordinary sulfur. Several types of primary reference rubber compounds were used in the following tables in the course of research on the production of composite materials (Table 1-4).

The preparation of rubber additives in stages 1 and 2 was carried out on the PD 630315/315 laboratory crusher. The temperature in the front shaft crusher is 50-60°C, in the back 60-70°C. The crushing was carried out on the laboratory crusher with the following characteristics:

- Shaft diameter – 160 mm;
- Shaft length – 320 mm;
- Friction ratio – 1: 1.24;
- Engine power – 4.6 - 7 kW;
- Optimal download – 1 kg.

The cleaned and chopped rubber passed through the holes between the shafts until a thin skin was formed. The additive was often cut for quality plasticization, thus the direction of the deformation force was changed. The procedure for the introduction of the ingredients of the first stage was carried out in accordance with the theoretical principles: initially, softeners, bulk ingredients,

Table 1. Standard rubber compound

Name of ingredients	Parts of mass	Mass, %	The exact amount of obtained substance, kg	
			1 stage	2 stage
Crude rubber SRI	32.70	23.61	118.10	
Reclaimed rubber of belaze	37.30	26.93	134.70	
Zinc whitening	5.00	3.61	18,10	
Stearic acid	2.00	1.44	7.20	
Micro wax	1.00	0.72	3.60	
Fuel oil	5.00	3.61	18.10	
Technical carbon	20.00	14.44	72.20	
Chalk	20.00	14.44	72.20	
Silica	11.00	7.94	39.70	
Additives after 1 stage			483.90	483.90
Sulfenamide	2.00	1.44		7.20
Sulfur	2.50	1.81		9
Total	138.5	100.00		
The set of the additives after 2 stage				500.10

activators, plasticizers were introduced. Technical carbon was introduced several times in small quantities, technical carbon scattered in a flat tin pan was re-introduced into additive. There were no problems during the mixing process.

Polymeric sulfur and accelerator were introduced in the second stage. The positive effect of the studied polymeric sulfur on the technological properties of standard rubber compounds was observed. Polymeric sulfur easily penetrates into the rubber mixture. The separation of polymer sulfur

in the mixture is satisfactory, it does not require changes in the order of crushing and vulcanization. The rubber mixture was cut regularly during the mixing process. The finished mixture was obtained from the grinder in the form of leaflet, sheets, and blanks. After the first stage, the rubber mixture rests for at least 2 hours. The samples were vulcanized in the process of electric vulcanization RDE 800x800 at a temperature of 155°C and in the order of 20 minutes to determine the physical and mechanical parameters.

Table 2. Version 1 of rubber compound

Name of ingredients	Parts of mass	Mass, %	The exact amount of obtained substance, kg	
			1 stage	2 stage
Crude rubber SRI	32.70	23.78	118.90	
Reclaimed rubber of belaze	37.30	27.13	135.60	
Zinc whitening	5.00	3.64	18.20	
Stearic acid	2.00	1.45	7.30	
Micro wax	1.00	0.73	3.65	
Organic part of oil sludge	4.00	2.91	14.55	
Technical carbon	20.00	14.55	72.70	
Chalk	20.00	14.55	72.70	
Silica	11.00	8.00	40.00	
Additives after 1 stage			483.60	
Sulfenamide	2.00	1.45		7.30
Tengiz sulfur	2.50	1.82		9.10
Total	137.5	100.00		
The set of the additives after 2 stage				500.00

Table 3. Version 2 of rubber compound

Name of ingredients	Parts of mass	Mass, %	The exact amount of obtained substance, kg	
			1 stage	2 stage
Crude rubber SRI	32.70	23.61	118.10	
Reclaimed rubber of belaze	37.30	26.93	134.70	
Diaphen	0.00	0.00	0.00	
Zinc whitening	5.00	3.61	18.10	
Stearic acid	2.00	1.44	7.20	
Micro wax	1.00	0.72	3.60	
Fuel oil	1.00	0.72	3.60	
Organic part of oil sludge	4.00	2.88	10.90	
Technical carbon	20.00	14.44	72.20	
Chalk	20.00	14.44	72.20	
Silica	11.00	7.94	39.70	
Additives after 1 stage			483.80	
Sulfenamide	2.00	1.44		7.20
Tengiz sulfur	2.50	1.81		9.00
Total	138.5	100.0		
The set of the additives after 2 stage				500.00

Table 4. Version 3 of rubber compound

Name of ingredients	Parts of mass	Mass, %	The exact amount of obtained substance, kg	
			1 stage	2 stage
Crude rubber SRI	32.70	23.61	118.10	
Reclaimed rubber of belaze	37.30	26.93	134.70	
Zinc whitening	5.00	3.61	18.10	
Stearic acid	2.00	1.44	7.20	
Micro wax	1.00	0.72	3.60	
Fuel oil	2.00	1.44	7.20	
Organic part of oil sludge	3.00	2.16	10.50	
Technical carbon	20.00	14.44	72.20	
Chalk	20.00	14.44	72.20	
Silica	11.00	7.94	39.70	
Additives after 1 stage			483.80	
Sulfenamide	2.00	1.44		7.20
Tengiz sulfur	2.50	1.81		9.00
The set of the additives after 2 stage	138.50	100.00		
Fuel oil				500.00

The determination of strength of vulcanizates was carried out in accordance with GOST 270-75 [2008]. For testing, the samples were taken according to the tolerances and marked according to GOST 270-75 [2008]. The samples of rubber plates with a thickness of 2 ± 0.2 mm or 1 ± 0.2 mm were used for the test. The results of determination of physical and mechanical properties of vulcanizates are shown in Figures 2-4. The results shown in the figures prove that the physical and mechanical properties of vulcanizate meet the control standards when using sulfur as a vulcanizing agent. Thus, the usage of polymeric sulfur as a vulcanizing agent and its positive effect on the properties of rubber were determined.

As can be seen in Figure 2, the sample of 3, which corresponds to the control norm, is 2 MPa lower than the reference value. The content of polymeric sulfur is 2-2.5 ms compared to the control norm has the highest conditional tensile strength. Increasing the amount of polymer sulfur to 3.0 units of mass leads to a decrease in this strength. According to the data in Figure 3, the relative elongation at break of vulcanizates in all versions is higher than the control norm and the reference version. However, version 2 can be noted with the highest rate of 287%.

Figure 4 shows that the relative elongation at break is 265% and the tensile strength is 118 MPa. The hardness index on the Shore corresponds to the control norm and 72 MPa to the reference version. Extending the conditional

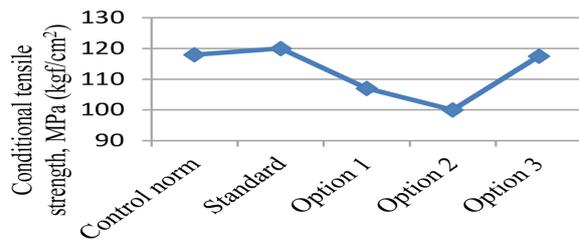


Fig. 2. Conditional tensile strength of vulcanizates

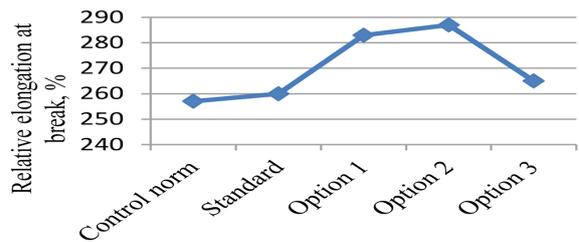


Fig. 3. Relative elongation of vulcanizates during elongation

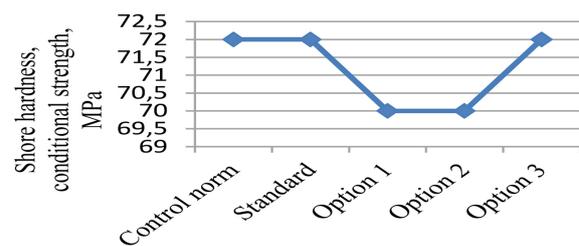


Fig. 4. Shore hardness of vulcanizates

stress value by 300% and further increasing the amount of polymer sulfur leads to a decrease in hardness to 70 conventional units. If the data in the figures are considered, the test results show that the amount of polymeric sulfur in the rubber is 1.5 mass units has the best set of physical and mechanical properties.

According to the results of the study, the optimal amount of polymeric sulfur, which achieves the maximum physical and mechanical properties of vulcanizates, the optimal size is 1.3 parts per 100 mass parts of rubber. Dosing of technical sulfur in the reference recipe of this order is 100 masses of rubber 1.8 times the mass per unit area, which is 0.5 times more than the using polymer sulfur. This is probably due to the activity of polymeric sulfur, which is completely involved in the vulcanization reaction, forming very strong sulfur bubbles, resulting in a partial increase in the strength properties of small amounts of vulcanizates.

As a practical application of the research results, we are sure that the introduction of technology for the production of rubber compounds used in the production of rubber products at the OJSC “Elastopolimet” confirmed by ICT industrial tests may result in a reduced technogenic load on the environment due to sulfur consumption.

The positive effect of polymer sulfur on the technological properties of the investigated rubber compounds was established. Polymer sulfur is easily added into the rubber compound. The distribution of polymer sulfur in rubber is satisfactory, it does not require changing the order of grinding and vulcanization.

According to the tables, the amount of polymer sulfur is 2-2.5 ms, in comparison with the reference sample, has the highest conventional tensile strength. Increasing the amount of polymer sulfur to 3.0 ms leads to the decrease of the given strength index. It is optimal at increasing in the value of the conditional voltage by 300% and the amount of polymer sulfur in salt hardness is 2-2.5 ms. Further increase in the amount of polymer sulfur leads to a decrease in hardness from 78.5 to 75 conventional units. The test results show that the best set of physical and mechanical properties in rubber is achieved if the amount of polymer sulfur is 1.5 ms.

According to the research results, the optimal amount of polymer sulfur at which the maximum physical and mechanical properties of achieved tread rubber is 1.3 parts per 100 masses of rubber. In this standard reference formula, the

dosage of technical sulfur is 1.8 parts per 100 parts by weight of rubber, which is 0.5 times higher than with polymer sulfur. This is probably due to the activity of polymer sulfur, which fully participates in the vulcanization reaction, forming very strong sulfur bubbles, from which, in small amounts, a partial increase in the strength properties of vulcanizates occurs.

CONCLUSIONS

Formulations of composite materials with the content of sea sulfur were developed, technological and physical-mechanical tests were carried out to confirm the quality of the obtained composite material -technical rubber. The influence of sea sulfur on the vulcanization time and properties of rubber was studied. The dependence of the content of sea sulfur on the quality of composite materials was determined. The optimal dose of sea sulfur was determined by the formula of the cord filler 3.5 mass per 100 mass rubber.

It was found that the usage of sea sulfur leads to an increase in the strength properties, including the heat resistance of the rubber filler, due to an increase in the number of intermolecular bonds in the elastomeric matrix due to the reaction of sulfur in the formulation. The technology of manufacturing rubber compounds and their vulcanization was considered. The quality of the obtained composite materials meets the requirements of technological regulations. The recommended filler compositions significantly save the resource on the operation of the external car tire .

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