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Issue Article

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PRODUCTION OF SUSTAINABLE SULFUR CONCRETE BASED ON ORGANIC MODIFIERS

***Abstract**: The article studies the study of the production of modified sulfur concrete from elemental sulfur and its composition using IR spectroscopy, electron microscopy and X-ray phase analysis based on X-rays.*

***Key words**: sulfur, sulfur concrete, ash, color phase analysis, diffractogram, electron microscope, sulfur concrete, sand.*
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Introduction

Sulfur-containing concrete is a thermoplastic composite concrete composed of modified sulfur and fillers. This study focused on the characterization of a new aerated concrete developed as a sustainable construction material. It is made from industrial waste technical sulfur, fly ash, sand, modified with modifiers and active fillers. Organic matter is used as a chemical modifier, added as an additive to cheap industrial products, new sulfur concrete is a stable product that can compete with Portland cement. A series of characteristic analyzes including elemental analysis method, IR spectroscopy, X-ray diffraction and phase-resolved X-ray absorption spectroscopy were carried out to confirm the polymerization of the sulfur produced by the experiment. In addition, mechanical testing, structural analysis and electronic scanning. Microscopic studies are evaluating the effectiveness of this new sulfur concrete as an environmentally friendly sustainable construction material.

Portland cement requires limestone to be heated in a kiln at temperatures above 1400°C for several hours to form clinker materials. Burning fuel for kilns and converting limestone into calcium oxide, releasing carbon dioxide, produces about one ton of carbon dioxide per cement, accounting for 5% of the world's anthropogenic CO_2 emissions [1, 2]. Unlike conventional cement production, sulfur-based cement production does not rely on high energy or direct carbon dioxide emissions [3-5]. In addition, sulfur is in excess globally. Sulfur is the third most common chemical element in petroleum at concentrations greater than 10% and is subject to environmental limitations in oil and gas processing [6]. Thus, there is a large amount of sulfur as a product of these processes. In addition, since sulfur itself is a by-product of industrial production, the amount of carbon dioxide emissions can be reduced by using sulfur-based concrete.

Sulfur-based concrete is a thermoplastic component of mineral aggregates and sulfur. Studies using elemental sulfur show that it has serious durability problems such as freeze-thaw cycles [7]. To form sulfur, sulfur and aggregates are mixed while hot, and upon cooling, the liquid sulfur binder in concrete products is initially converted to monoclinic sulfur (Sb). As we continue to cool, the material transitions to a solid phase as it transitions to rhombic sulfur (Sa), which causes the material to shrink. Therefore, in order to increase the durability of sulfur-based concrete, chemical modifiers that polymerize sulfur were added [8]. This modified sulfur concrete is known as polymer sulfur concrete (POB). It can be used as a building material due to its excellent resistance to acidic and saline environments. Also, these binders effectively stabilize contaminated soil and nuclear waste [9,10,11]. Unlike traditional hydraulic cement concrete, sulfur concrete does not need water and can reach full strength in just a few days, compared to up to 28 days for conventional Portland cement concrete. Several organic chemical modifiers have been developed to efficiently polymerize sulfur.

Experimental method

The raw materials necessary for sulfur concrete are prepared. First of all, we pay attention to the purity level of substances. at first the sulfur dissolves and over time turns to a sticky yellowish-brown state. As a modifier, an aromatic compound, which is an organic substance, is added directly to the dissolved sulfur phase. The resulting mixture is heated at a temperature of 160-180 C for 45 minutes with stirring. This reaction results in a slight decrease in the viscosity of the mixture medium and a dark yellow product characteristic of copolymers of aromatic compounds with sulfur. Then we add ash as an additive, add sand as filler to the resulting product, mix thoroughly for 10 minutes. Pour the resulting mixture into a mold and leave for 45-50 minutes to cool. We examine the obtained sample in the IR spectrum.

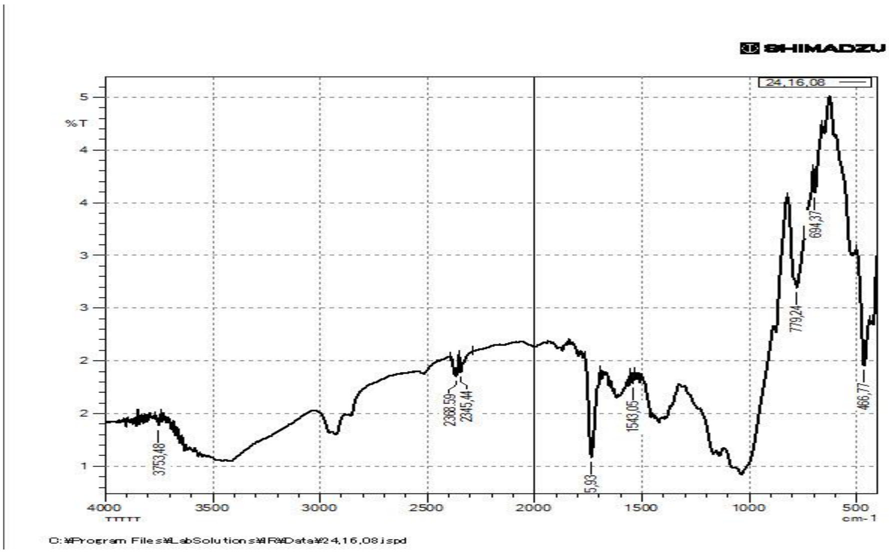

Figure 1.

In the IR spectrum of modified sulfur in the range of 2850-1470 cm^{-1} there are absorption lines confirming the presence of $–CH_2$- groups and in the region of 1650 cm^{-1} there are absorption lines confirming the presence of the -C=O group in the free state. In the IR spectrum, there are absorption lines in the region of 3400 cm^{-1} corresponding to –OH groups. Deformation vibrations of all active –SN–O– groups appear in the form of strongly absorbed lines in the range of 1400-1465 cm^{-1}. The presence of groups with sulfur S=O and S–N in the range of 2343-2688 cm^{-1}, a broad intense peak in the regions of 1200-1100

cm^{-1}, 1040-1060 cm^{-1} confirm compounds containing sulfur.

In addition, in IR-spectroscopy, narrow lines of low intensity appear in the regions of 1060 cm^{-1} and 1015 cm^{-1}, which contain bonds of the sulfur-containing compound. When examining the IR spectra of modified sulfur, intense groups $–SN_2$-O- in the dimer state with indices of 1400-1440 cm^{-1} are visible (Fig. 1).

According to its composition, sulfur concrete belongs to the category of special concrete. The national standard GOST P 59613-2021 is important in the design and preparation of technological documents of sulfur concrete, as well as in the construction work and in determining the quality of the finished material.

Table 1. Compressive strength for sulfur polymer concrete samples

№	Time, Day	Average compressive strength, MPa	Standard deviation, MPa
1	3	33,25	0,16
2	7	37,62	0,67
3	14	39,43	2,23
4	28	41,33	0,98

Material synthesis

In the production of sulfur concrete, it is necessary to prepare fillers in advance. The calculated ratio of the mixture was 58% sand, 12% ash, 29.8% sulfur and 0.2% organic modifier (Table 1). Fly ash is used in conventional cementite concrete for its pozzolanic reaction, which can reduce the carbon fixation of the product, in sulfur concrete (along with sand) fly ash is used to provide potential reaction sites for polymerization and as a filler in the composition. Also, the addition of ash to sulfur concrete is beneficial in improving the strength due to the rounded shape of the mixture and the presence of fillers of suitable size. Electron microscopy shows elemental sulfur and fly ash particles, irregularly shaped particle sizes (micron diameter or less) and spherical shapes of fly ash particles (Figure 2). At the initial stage, filler products and organic modifier are mixed and heated at a temperature of 170-180 °C for 2 hours. To reduce the particle size, the materials were combined with elemental sulfur and processed through a 1 mm long flow mill. Then the mixture is heated and stirred in the molten state at 135-145 °C for an hour and poured into molds for cooling. The average density of solution samples was 2282 (± 41) kg / m^3.

Table 1. Sulfur concrete mix ratio

Oxide	Sulfur	Sand	Fly Ash	Organic modifier	Total
mas.%	29,8	58	12	0,2	100

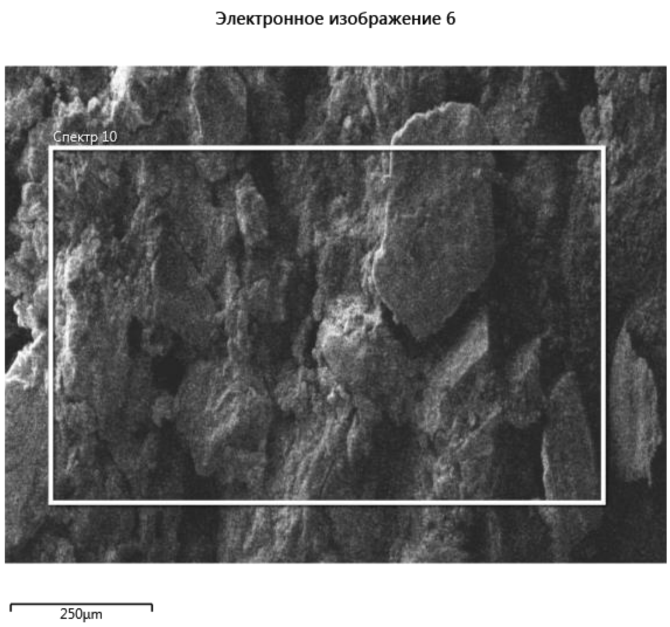

Figure 2. X-ray diffraction pattern of sulfur-containing concrete

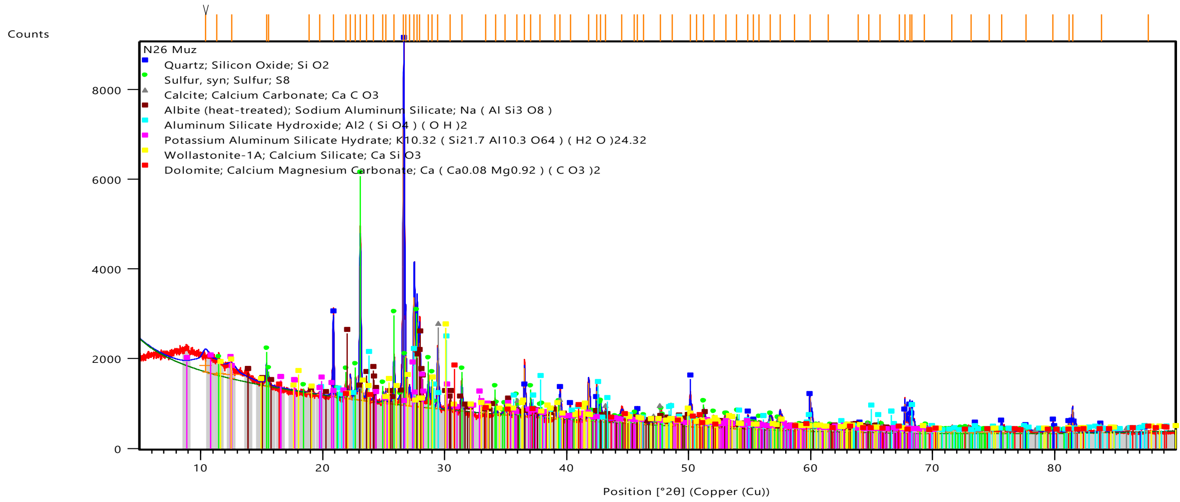

Figure 3. Shows the x-ray phase analysis diffractogram of sulfur-containing concrete. It can be seen from the X-ray diffraction image that sulfur concrete contains S, SiO_2, $CaCO_3$, $CaSiO_3$, $Al_2(SiO_4)(OH)_2$ molecules.

Table 2. Below contains data on absorption peaks of sulfur-containing concrete in X-ray phase analysis.

№	2 theta scanning angle[$2 0^0$]	Pics[cts]	FWHM left[$2 0^0$]	d distance between planes [A]	Density of I-peaks
1	20,9291	1152,26	0,0951	4,24110	26,82
2	23,1432	2619,77	0,1398	3,84013	60,97
3	26,7030	4296,72	0,0883	3,33572	100,00
4	27,5641	4247,18	0,06130	3,23348	98,85
5	27,8072	1068,75	0,1858	3,20572	24,87
6	39,5710	4245,56	0,0550	2,27563	98,81
7	75,7682	1162,20	0,0833	1,25442	27,05

When calculating the particle size of sulfur concrete according to the Debye-Scherre equation, it is calculated according to the values given in the formula. Example:

d=Kl / bcost d=0.94*1.54178 / 0.951*0.2=76.19

Table 3. Calculation of the particle size of sulfur concrete according to the Debaya-Scherre equation:

№	2 theta scanning angle[$2 0^0$]	FWHM left[$2 0^0$]	average size of d(nm) crystals	d (nm) average
1	20,9291	0,0951	76,19	
2	23,1432	0,1398	51,83	
3	26,7030	0,0883	85,25	84,14
4	27,5641	0,06130	118,21	
5	27,8072	0,1858	39,00	
6	39,5710	0,0550	131,7	
7	75,7682	0,0833	87,30	

It was calculated that the average particle size d(nm) of sulfur concrete is equal to 84.14 according to the Debye-Scherre equation.

Summary: In this study, a new sulfur concrete using sulfur, fly ash and industrial additives during the catalytic cracking of petroleum and petroleum products was characterized. As the production of sulfur concrete relies on an inexpensive chemical modifier (i.e., a by-product of processing plants), this material can be a practical and cost-effective solution for a sustainable construction material.

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