







N.D. Amanova^{1*} , Kh.Kh. Turaev¹ , A.T. Djalilov² , A.K. Nomozov³ ,
E.S. Sottikulov² , Y.A. Makhmudova⁴ 

¹Termez State University, Termez, Uzbekistan

²Tashkent Research Institute of Chemical Technology, Tashkent, Uzbekistan

³Termez State University of Engineering and Agrotechnologies, Termez, Uzbekistan

⁴National University of Uzbekistan, Tashkent, Uzbekistan

*e-mail: amanova0176@gmail.com

A STUDY ON PHYSICO-CHEMICAL PROPERTIES OF MODIFIED SULFUR CONCRETE

In this article, the polymerization reaction of elemental sulfur with organic modifiers is studied, and the optimal conditions of the modification process are presented. Also, the process of obtaining polymer sulfur concrete from modified sulfur is presented. Initially, elemental sulfur is heated to a temperature of 140-145 °C, aniline hydrochloride is added, and the temperature of the reaction is raised to 160 °C for 45 minutes. The average density of this obtained sulfur concrete sample was 2382 kg/m³. Sulfur-based concrete was obtained using the modified sulfur obtained in this process. Sulfur-based concrete was obtained by taking 56% sand, 14% ash, and 30% modified sulfur. Physico-chemical properties of sulfur concrete obtained based on modified sulfur and modified sulfur were studied and analyzed using X-ray phase analysis, IR-spectroscopy, scanning electron microscope, gamma-spectroscopy, and X-ray analysis. Also, the thermal analysis of modified sulfur-based concrete was studied using differential-thermal and thermogravimetric analysis. It was calculated that the average particle size d (nm) of the obtained polymer sulfur concrete is equal to 84.14 according to the Debaya-Sherrera equation.

Key words: sulfur, sulfur concrete, modifier, Anilinehydrochloride, X-ray phase analysis, diffractogram, electron microscope, IR-spectroscopy, Roman-spectroscopy.

Н.Д. Аманова^{1*}, Х.Х. Тураев¹, А.Т. Джалилов², А.К. Номозов³,
Е.С. Соттикулов², Ю.А. Махмудова⁴

¹Термез мемлекеттік университети, Термез қ., Өзбекстан

²Ташкент химия-технология ғылыми-зерттеу институты, Ташкент қ., Өзбекстан

³Термез мемлекеттік инженерлік және агротехнологиялық университети, Термез қ., Өзбекстан

⁴Өзбекстан Ұлттық университети, Ташкент қ., Өзбекстан

*e-mail: amanova0176@gmail.com

Модифицирленген күкіртті бетонның физика-химиялық қасиеттерін зерттеу

Бұл зерттеуде элементарлы күкіртті органикалық модификаторлармен өңдеу арқылы оның полимерлену процесі тереңірек зерттелді. Нәтижесінде, күкірт бетондары үшін жоғары сапалы байланыстырғыш алуға мүмкіндік беретін оңтайлы модификациялау шарттары анықталды. Модификацияланған күкірт негізіндегі күкірт бетондарын алу технологиясы толық сипатталды.

Тәжірибе барысында элементарлы күкіртті 140-145 °C температураға дейін қыздырып, содан кейін анилин гидрохлориді қосылды. Реакцияны толықтыру үшін температураны 160 °C дейін көтеріп, 45 минут бойы сақтады. Алынған күкірт бетонының орташа тығыздығы 2382 кг/м³ құрады. Оның құрамына 56% құм, 14% күл және 30% модификацияланған күкірт кірді.

Модификацияланған күкірт негізіндегі күкірт бетонның физикалық-химиялық қасиеттерін толықтай зерттеу үшін рентгендік фазалық талдау, инфрақызыл спектроскопия, сканерлеуші электронды микроскопия, гамма-спектроскопия және рентгендік флуоресценттік анализ әдістері қолданылды. Сонымен қатар, материалдың жылудық тұрақтылығын бағалау үшін дифференциалды термоанализ және термогравиметриялық анализ әдістері қолданылды. Дебай-Шеррер теңдеуі бойынша есептелген полимерлі күкірт бетонының бөлшектерінің орташа мөлшері 84,14 нм құрады, бұл оның жоғары дисперстілігін көрсетеді. Жүргізілген зерттеулер нәтижесінде жоғары беріктікке

және әртүрлі сыртқы әсерлерге төзімділігі жоғары жаңа құрылыс материалы – күкірт бетон алуға мүмкіндік беретін тиімді әдіс әзірленді.

Түйін сөздер: күкірт, күкіртті бетон, модификатор, анилин гидрохлорид, рентгендік фазалық талдау, дифрактограмма, электронды микроскоп, ИҚ-спектроскопия, Раман спектроскопиясы.

Н.Д. Аманова^{1*}, Х.Х. Тураев¹, А.Т. Джалилов², А.К. Номозов³,
Е.С. Соттикулов², Ю.А. Махмудова⁴

¹Термезский государственный университет, г. Термез, Узбекистан

²Ташкентский научно-исследовательский химико-технологический институт, г. Ташкент, Узбекистан

³Термезский государственный университет инжиниринга и агротехнологий, г. Термез, Узбекистан

⁴Национальный университет Узбекистана, г. Ташкент, Узбекистан

*e-mail: amanova0176@gmail.com

Исследование физико-химических свойств модифицированного серного бетона

В данной статье исследуется реакция полимеризации элементарной серы с органическими модификаторами, а также представлены оптимальные условия для процесса модификации серы для получения высококачественного связующего вещества для серного бетона. Рассматривается процесс получения серного полимербетона на основе модифицированной серы. Сначала элементарная сера нагревается до температуры 140 – 145 °С, затем добавляется гидрохлорид анилина, и температура реакции повышается до 160 °С на 45 минут. Средняя плотность полученного образца серного бетона составила 2382 кг/м³. Для создания серного бетона использовали модифицированную серу, при этом в составе использовалось 56% песка, 14% золы и 30% модифицированной серы. Физико-химические свойства серного бетона на основе модифицированной серы были исследованы с помощью рентгенофазового анализа, ИК-спектроскопии, сканирующего электронного микроскопа, гамма-спектроскопии и рентгеновского анализа. Также проводился термический анализ серного бетона с применением дифференциального термического и термогравиметрического анализа. Расчет среднего размера частиц полимерного серного бетона по уравнению Дебая – Шеррера позволил определить, что он составляет 84,14 нм, что свидетельствует о формировании мелкодисперсной структуры материала.

Ключевые слова: сера, серобетон, модификатор, анилингидрохлорид, рентгенофазовый анализ, дифрактограмма, электронный микроскоп, ИК-спектроскопия, Раман-спектроскопия.

Introduction

The natural resources are exhausting due to the expansive growth in the construction sector, while waste materials are accumulating in the natural environment because of the endless processing and finishing of raw products [1, 2].

The advancement of technology in the petroleum industry and the installation of new oil refineries have caused the large-scale production of sulfur by-products [3]. Owing to special physical, chemical, and mechanical characteristics, sulfur has applications in the fields of agriculture, pharmaceuticals, chemical, and construction industry, and in the process of rubber vulcanization [4]. The utilization of sulfur as a substitute for cement binders can help in minimizing the abiotic depletion caused by the construction industry [5]. Conventional concrete requires water for the hydration of cement (in order to complete the chemical reactions responsible for strength gain) [6]. However, the experience across the world has

shown that SC has significant strength and durability during exposure to chemicals in comparison to PCC. There have been significant applications of SC in the USA and Canada; however, the use in Europe is limited [7]. The utilization of SC on large scales will be economical as compared to cement concrete, and it can alleviate environmental problems and problems related to sulfur storage [8].

Sulfur melts at 120 °C and transfers into a liquid state. When cooled at 114 °C, it changes from liquid to a monoclinic crystalline form. On further cooling to 98 °C, it changes to an orthorhombic state, which is stable at room temperature. This polymerization helps in attaining high strength at normal temperatures [9]. As sulfur is a thermoplastic material, it has some of its limitations to be used as a binder. Despite this, there are several benefits of SC applications which including a high of strength in a short time, resistivity to aggressive chemicals, waterproof characteristics, and recycling potential [10]. Today, sulfur is considered a good

building material alternative for some applications in the construction industry. As an alternative to traditional concrete with cement as a binder, sulfur-solidified concrete is among other construction materials [11]. The first studies on the evaluation of sulfur as construction material were carried out by Bacon and Davis [12]. A material with high acid resistance and strength was obtained from 60% sand and 40% sulfur mix. The use of sulfur as a binder was investigated by experimental studies performed by various researchers [13]. In this study, it is aimed to determine the durability performances of bitumen-modified sulfur polymer concrete (SPC) Portland cement concrete (PCC). Three different bitumen ratios were selected as 2.5%, 5%, and 7.5% by weight of pure sulfur for the modification. The sulfur modification of the modified sulfur sample prepared with 2.5% bitumen addition among the selected bitumen ratios was successful [14]. The mix designs and specimen preparation for the dry process and wet process of sulfur rubber concrete (SRC) were investigated. Although the compressive strength of SRC decreases with increasing rubber content, the toughness increases instead. Adding micro-filler will improve the compressive strength of SRC. There is a threshold value for the sulfur content, at which the compressive strength and the workability of SRC reach an optimum balance. The bond between rubber particles and surrounding sulfur is strong due to the vulcanization process that generates cross-links through S-C bonds [15]. One of the most perspective methods of enhancing durability of building materials and articles exposed to various aggressive media is application of sulfur for their manufacturing. The results of the experiments have proved that in all the impregnated samples the strength had increased by a factor of 2.0 ÷ 4.8. The data have also demonstrated that depth of sulfur penetration into capillary-porous article had augmented twofold and more. As seen from these data, strength and resistance to aggressive media of arbolites impregnated with sulfur by-products is rather high. This proves that arbolite blocks impregnated with sulfur by-products could be recommended for masonry of inspection manholes, underground engineering structures [16]. The sulfate resistance of concrete was tested using drying-immersion cycles of varying duration in different sulfate solutions. A matrix of 20 concrete mixtures was tested with this protocol. The test permitted to distinguish the effect of cement type, w/c and paste volume on expansion. Measurements of the dynamic E-modulus made it possible to link expansion and mechanical damage and to define a limit value for expansion [17]. Durability of sulfur concrete with different fillers, as well as Portland cement concrete, was tested in the solutions of HCl,

H₂SO₄, and NaCl. Regarding mass changes, in the solutions of HCl and H₂SO₄ sulfur concrete with talc and fly ash exhibited higher durability, while in NaCl samples with alumina and microsilica were better. Strength loss was higher in the solution of HCl comparing to H₂SO₄, while negligible in NaCl which is in accordance with apparent porosity increase. Portland cement concrete after two months lost 20% of mass [18]. The aim of this study was to predict the corrosion of cement concrete (CC) and sulfur concrete (SC) in sewer systems using three artificial intelligence (AI)-based techniques: Adaptive NeuroFuzzy Inference System (ANFIS), Genetic Programming (GP), and Multi Expression Programming (MEP). Two sets of chemical experiments in acidic solutions and biological tests using *Thiobacillus thiooxidans* were conducted to investigate the corrosion in concrete samples. For both tests, weight loss was used as the indicator of corrosion [19,20]. This paper presents the results of the experimental research and analyses indicating the usefulness of polymer sulphur composites to the protection against corrosion of reinforcement. Presented in paper materials, being also the domain of the personal investigations and the methodology are definite. After analyze of the initial results the optimum compositions have been chosen for the experimental research [21-25].

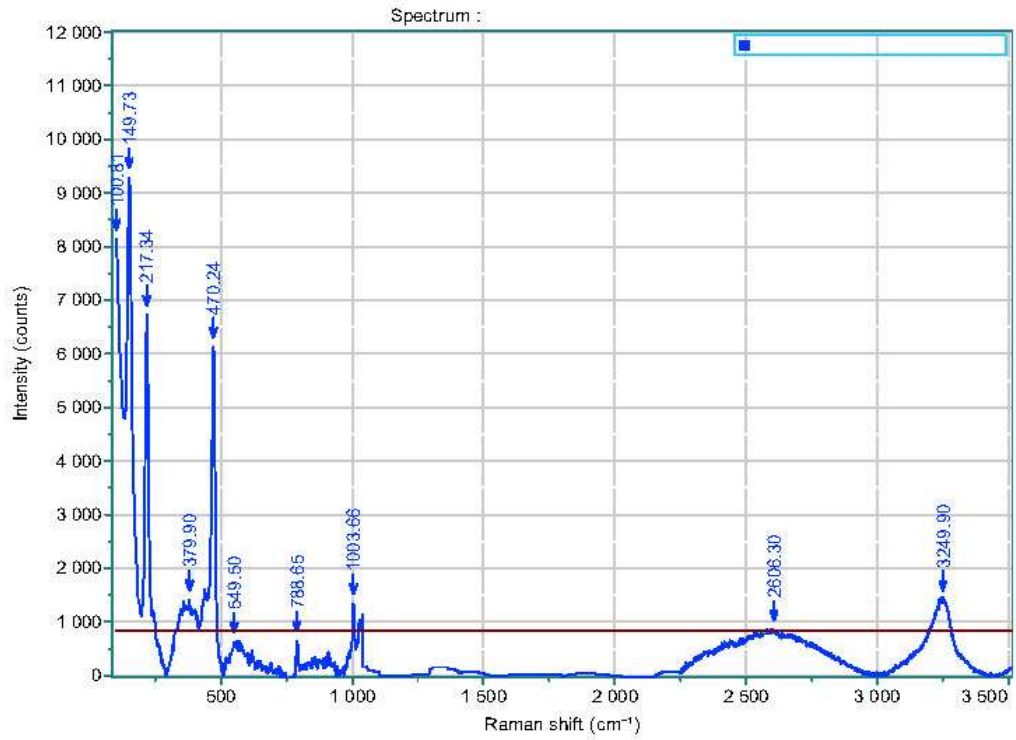
Purpose of work: It consists of obtaining modified sulfur based on aniline hydrochloride, analyzing their composition and structure using the results of infrared spectroscopy and scanning electron microscope analysis, studying their physical and mechanical properties, improving their properties, and developing polymer concrete with sulfur-based on them.

Research methods. The IR-spectrum of modified sulfur was analyzed by a Japanese-made SHIMADZU IR-Fure spectrophotometer in the range of 750-4000 cm⁻¹, a HORIBA scientific novel-spectrometer at 3900-500 cm⁻¹ and MIRA 2 LMU scanning electron microscope (SEM), X-ray phase analysis. Modified concrete was analyzed by differential-thermal and thermogravimetric methods.

Experimental part

Modification of elemental sulfur using aniline hydrochloride

Initially, the raw materials needed to obtain sulfur concrete are measured with high precision in grams. In the process of modification, the first elemental sulfur is heated to a temperature of 140-145 °C. After elemental sulfur changes to a yellowish brown state, aniline hydrochloride is added to the dissolved sulfur phase as an organic



Date	08.04.2023 1...	Acq. time (s)	1.13383	Accumulations	1	Laser (nm)	785
Spectro (cm ⁻¹)		Hole		Slit		Grating	685
ND Filter		Objective		ICS correction	Off	Range (cm ⁻¹)	

Figure 2 – Novel spectrum of Modified Sulfur

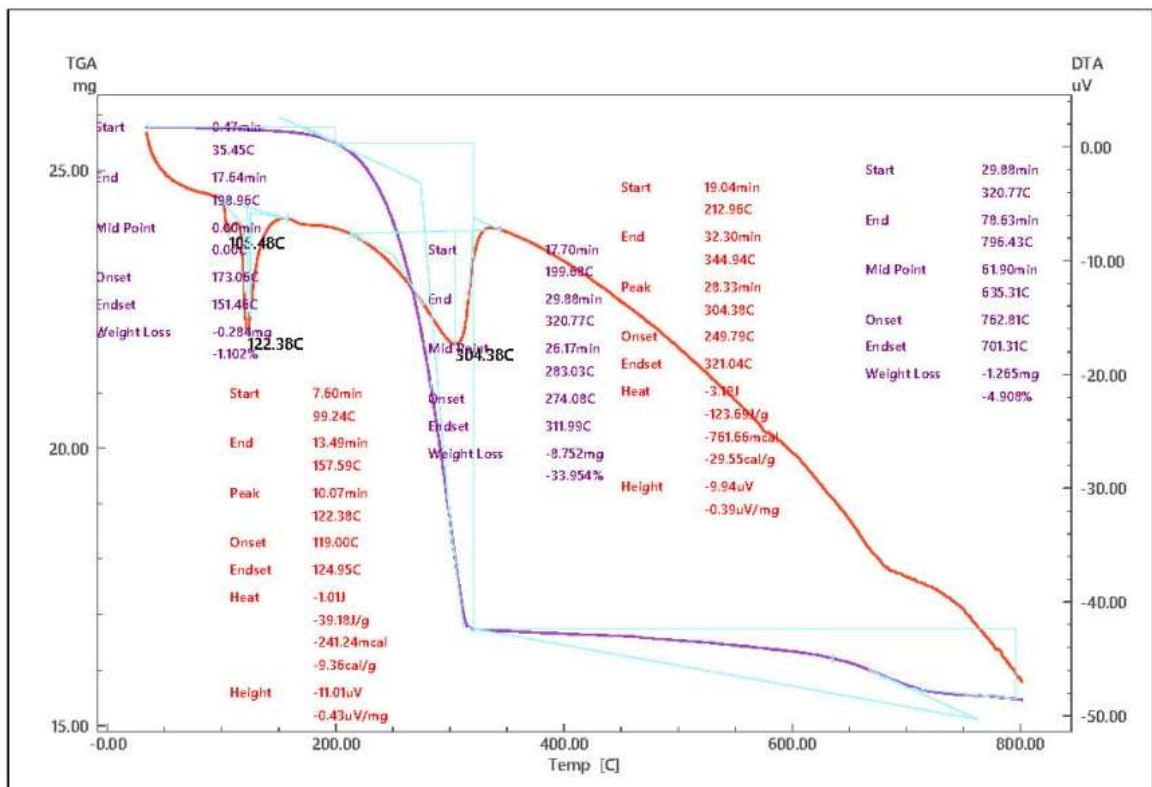


Figure 3 – Derivatogram of modified sulfur concrete

The analysis of the thermogravimetric curve of the organic compound shows that the TG curve mainly takes place in the temperature range of 1 intensive and 2 non-intensive mass losses. The 1st mass loss range is 35.45 - 198.96°C, the 2nd mass loss range is 199.68 - 320.77°C, and the 3rd mass loss range is 320.77 - 796.43° C corresponds to the temperature. The analysis shows that in the mass loss interval 1, a mass loss of 0.284 mg, i.e. 1.102%, is observed, while mass loss 2 is the most intense decay. The main amount of mass loss in this decay is 8.752 mg, i.e. 33.954%. It can be seen that the first mass loss involves the loss of unbound matter. In the second main stage of decomposition, in the case of lower molecular compounds such as hydrogen sulfide, sulfur oxides, and carbon oxides, after the decomposition of the organic and sulfurous part, a mixture of sulfur oxides and partially coal remains.

The results of the thermal analysis show that the field of application of the obtained sulfur concrete is limited to a maximum of 3000 C. This temperature is several times higher than the normal outdoor temperature, which shows the possibility of wide application.

One of the main advantages of modified sulfur concrete over Portland cement is its resistance to acidic and saline environments, especially in industrial environments where traditional cement has a short service life. In addition to industrial enterprises, it is used in freezing and thawing cycles, in food processing enterprises, and in the production of sewage pipes, drainage channels, and marine structures. In terms of sustainability, MOB can be considered an environmentally friendly material as it can replace Portland cement in several construction applications. A few hours after casting, its compressive strength increases to about 80%, and typically 80 to 95% after 24 hours. In this work, studies were conducted on two identical samples hardened for 3, 7, 14, and 28 days. The change in average compressive strength for all samples is presented in Table 1. Table 1 shows that the average compressive strength obtained at 3 days was 32 MPa and 42 MPa at 28 days. The results correspond to a percentage of compressive strength calculated at 80.5% compared to 28 days.

Table 1. Compressive strength indicators of sulfur concrete samples prepared on the basis of modified sulfur

No	Time, day	Average compressive strength, MPa	Standard error, MPa
1	3	32,15	0,16
2	7	37,62	0,67
3	14	39,43	2,23
4	28	42,13	0,98

Durability tests of the samples were carried out by measuring the mass of the sample after 3, 7, 14, and 28 days and comparing it with the initial value (before dissolving); the results are shown in Figures 4, 5, and Table 1. Mass loss was calculated for 10x10x10 mm square samples immersed in a 40% solution of sulfuric acid and ammonium sulfate. The mass changes induced by dissolution were negligible, indicating that the samples obtained did not deteriorate after exposure to acidic and saline environments.

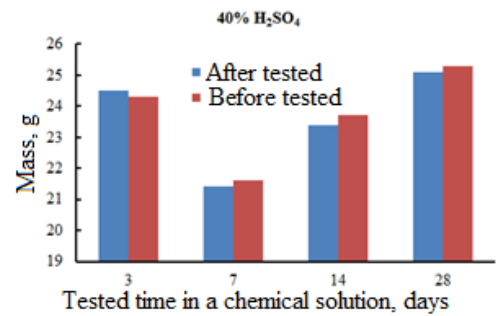


Figure 4 – Mass change after immersion in 40% H₂SO₄

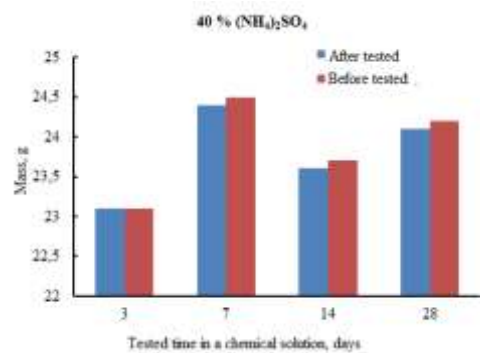


Figure 5 – Mass change after immersion in 40% (HN₄)₂SO₄

SEM photograph and X-ray phase analysis diffractogram of modified sulfur concrete

Figure 6 shows electron microscopic elemental sulfur and fly ash particles, irregularly shaped particle elemental sulfur and sizes (micron diameter or less), and spherical shapes of fly ash particles

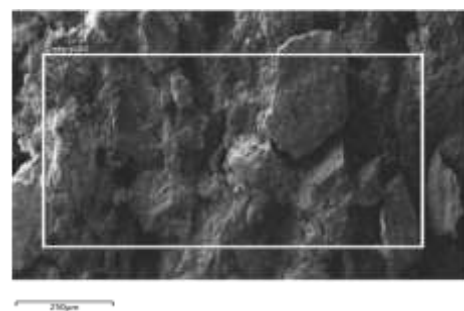


Figure 6 – Photomicrograph of modified sulfur concrete

As can be seen from Figure 7, made on the basis of a roentgenogram, sulfur concrete contains S, SiO₂, CaCO₃, CaSiO₃, Al₂(SiO₄)(OH)₂

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

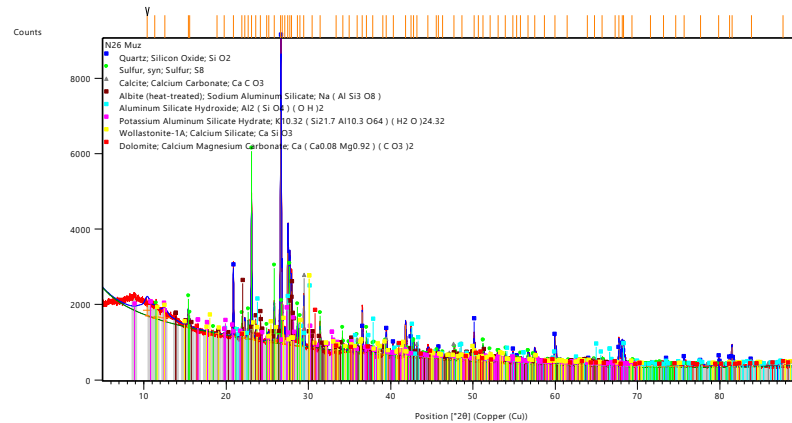


Figure 7 – X-ray phase analysis diffractogram of modified sulfur concrete

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

continuation of Table 2

Table 2 – X-ray phase analysis diffractogram of modified sulfur concrete

Pos. [°2θ]	Height [cts]	FWHM Left [°2θ]	d-spacing [Å]	Rel. Int. [%]
10.4168	278.24	1.0527	8.48544	4.52
11.3083	64.93	1.4870	7.81842	1.05
12.5358	180.96	1.3851	7.05550	2.94
15.4043	366.22	0.0854	5.74749	5.94
15.5815	121.19	0.0687	5.68254	1.97
18.8842	35.71	0.0174	4.69549	0.58
19.7809	60.30	0.0218	4.48461	0.98
20.8749	1414.88	0.0959	4.25200	22.97
21.9421	169.68	0.2839	4.04754	2.75
22.2804	452.31	0.0702	3.98685	7.34
22.6988	361.39	0.0811	3.91429	5.87
23.0824	2988.27	0.1045	3.85010	48.50
23.6199	222.51	0.1426	3.76369	3.61
24.1859	249.73	0.0815	3.67688	4.05
24.9453	191.04	0.0983	3.56664	3.10
25.1764	238.99	0.0686	3.53441	3.88
25.8470	841.52	0.1097	3.44422	13.66
26.6462	6161.00	0.1226	3.34270	100.00
26.8198	1097.28	0.0376	3.32146	17.81
27.1260	247.13	0.1120	3.28465	4.01
27.5053	2426.33	0.1327	3.24021	39.38
27.7372	1873.70	0.1011	3.21365	30.41
27.9385	1420.71	0.1377	3.19095	23.06
28.6655	440.30	0.1750	3.11165	7.15
28.9656	465.42	0.0652	3.08009	7.55
29.4408	1076.35	0.1317	3.03146	17.47
30.4535	253.96	0.0594	2.93291	4.12
31.4077	493.17	0.1252	2.84595	8.00
33.3834	0.00	0.0866	2.68190	0.00
34.1777	239.76	0.1367	2.62136	3.89
34.9373	178.09	0.1248	2.56609	2.89
35.9352	176.72	0.2433	2.49709	2.87
36.5570	1075.52	0.0924	2.45603	17.46
37.0633	373.97	0.1030	2.42363	6.07
37.8374	148.97	0.0975	2.37581	2.42
39.0735	281.03	0.0645	2.30344	4.56
39.4445	485.51	0.1742	2.28263	7.88
40.3105	291.06	0.0875	2.23557	4.72

Pos. [°2θ]	Height [cts]	FWHM Left [°2θ]	d-spacing [Å]	Rel. Int. [%]
41.8126	845.68	0.1387	2.15866	13.73
42.4746	676.94	0.0911	2.12654	10.99
42.7673	341.60	0.1573	2.11266	5.54
43.1937	223.10	0.1447	2.09279	3.62
44.5266	218.61	0.4349	2.03318	3.55
45.5493	100.11	0.6770	1.98988	1.62
45.8095	213.45	0.0830	1.97919	3.46
46.3318	75.91	0.1210	1.95808	1.23
47.7158	169.55	0.5106	1.90447	2.75
48.6674	50.41	0.6848	1.86943	0.82
50.1297	492.86	0.2369	1.81827	8.00
50.6762	111.09	0.1579	1.79994	1.80
51.2307	363.80	0.1558	1.78175	5.90
52.0720	220.00	0.1440	1.75492	3.57
53.0915	215.12	0.3661	1.72360	3.49
53.9629	190.69	0.1665	1.69781	3.10
54.8758	234.90	0.1190	1.67170	3.81
55.3307	119.32	0.2359	1.65903	1.94
55.8037	110.46	0.3832	1.64608	1.79
56.7051	271.22	0.2112	1.62204	4.40
57.5127	345.16	0.2237	1.60117	5.60
58.7079	93.60	0.3515	1.57139	1.52
59.9677	755.47	0.1092	1.54135	12.26
61.4463	88.31	1.8692	1.50776	1.43
63.9360	56.20	0.7723	1.45492	0.91
64.7657	52.38	0.1737	1.43827	0.85
65.6865	109.93	1.2279	1.42032	1.78
67.2711	56.07	0.3167	1.39066	0.91
67.7508	651.23	0.1081	1.38198	10.57
68.1580	590.92	0.1159	1.37471	9.59
68.3210	309.46	0.1308	1.37183	5.02
69.3452	90.25	1.2107	1.35405	1.46
71.6015	67.43	0.9476	1.31682	1.09
73.1890	60.83	1.4268	1.29213	0.99
74.6452	49.77	2.6351	1.27049	0.81
75.6685	198.87	0.1287	1.25583	3.23
77.6887	74.77	0.6441	1.22814	1.21
79.8904	192.57	0.2219	1.19974	3.13
81.1769	223.36	0.1106	1.18394	3.63
81.5006	526.76	0.1264	1.18006	8.55
83.8430	129.26	0.2145	1.15295	2.10
87.7163	0.00	0.5628	1.11174	0.00

According to table 2, we can quote table 3.

Table 3 – Diffractogram data of sulfur concrete

№	2 theta scan angle [20°]	Peaks [cts]	FWHM left [20°]	d is the distance between planes [Å]	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-Scherrer equation, it is calculated according to the values given in the formula. Example:

$$d = K\lambda/\beta\cos\theta$$

$$d = 0.94 * 1.54178 / 0.951 * 0.2 = 76.19.$$

Table 4 – Calculation of the particle size of sulfur concrete according to the Debaya-Sherrera equation

№	2 theta scanning angle [20°]	FWHM left [20°]	average size of d(nm) crystals	d (nm) average
1	20.9291	0.0951	76.19	84.14
2	23.1432	0.1398	51.83	
3	26.7030	0.0883	85.25	
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-Sherrera equation.

Gamma spectrometer analysis of modified sulfur

Taking into account the advantages and convenience of modified sulfur, we analyzed it in a Gamma spectrometer in order to study its impact on the environment. Accordingly, the test conditions are T-22°C, humidity-63%, light-300 lux, relative activity Bk/kg.

Table 5 – Analysis of radionuclides in modified sulfur using a gamma spectrometer

Product name	Sample order	Ra-226	Th-232	K-40	Aeff. bk/kg	Aeff.mb k/kg
Modified Sulfur	1	4.76	5.44	137	18.7	84.4
	2	5.24	6.34	127	24.4	86.8
	3	3.74	4.53	115	19.5	103
	4	5.34	5.29	126	23.0	96.4
	5	3.54	5.36	119	20.7	36.9
Av.	4	5	124	Aeff	Aeff.m	
				σ _r =	σ _r =	
				=108.8	=160.8	

According to SanPin 0193-06, it is used in construction if it is less than 350 Bk/kg. When we analyzed Modified Sulfur, the radionuclides contained in it corresponded to an average of 160.8 Bq/kg.

The results of the gamma spectrometry examination of the sample taken above meet the requirements of sanitary rules and norms No. 0193-06. The obtained result can be used in housing construction if the gamma spectrometer indicator is up to 300 Bk/kg. If it is < 350 Bk/kg, it can be used for baryors (fences), ditches, underground pipe protection means, and stone pavements (Bruschatka) on the streets.

Conclusion

In this study, the advantages of obtaining sulfur concrete from modified sulfur were studied. A new sulfur concrete was developed using technical sulfur, ash, and industrial additives extracted during the production of petroleum products. In the production of sulfur concrete, an inexpensive chemical modifier was used, this raw material can be a practical and cost-effective solution for a sustainable building material. Sulfur concrete products obtained on the basis of sulfur, ash, and fillers polymerized at high temperatures have been confirmed to have high strength in a short time, resistance to salty and acidic environments, water absorption, and recyclability. Results of gamma spectrometric studies 0193-06 - meet the requirements of sanitary regulations. The results obtained were less than <350 Bq/kg, indicating that it can be used for street barriers, ditches, underground pipe protection devices, and paved roads.

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- 25 A.K. Nomozov, Kh. Beknazarov, S. Khodjamkulov, Z. Misirov, and S. Yuldashova, Baghdad Sci. J. 22, (2024).

Article history:

Received 22 August 2024

Accepted 8 September 2024

Мақала тарихи:

Түсті – 22.08.2024

Қабылданды – 08.09.2024

Information about authors:

1. **N.D. Amanova** (corresponding author) – PhD, associate professor, Faculty of Chemistry, Termez State University, Termez, Uzbekistan. email: amanova0176@gmail.com

2. **Kh.Kh. Turaev** – Doctor of Chemical Sciences, Professor, Faculty of Chemistry, Termez State University, Termez, Uzbekistan. email: hhturayev@rambler.ru

3. **A.T. Djaliilov** – Doctor of Chemical Sciences, Professor, Academician of the Academy of Sciences of the Republic of Uzbekistan, Director of Tashkent Research Institute of Chemical Technology. Tashkent, Uzbekistan. email: gup_tniixt@mail.ru

4. **A.K. Nomozov** – PhD associate professor, Head Department of Chemical Engineering, Termez State University of Engineering and Agrotechnologies. Termez, Uzbekistan. email: abornomozov055@gmail.com

5. **E.S. Sottikulov** – Associate professor, Head Department of Chemical Engineering Laboratory of Tashkent Research Institute of Chemical Technology. Tashkent, Uzbekistan. email: gup_tniixt@mail.ru

6. **Y.A. Makhmudova** – Student of the Faculty of Chemistry of the National University of Uzbekistan, Tashkent, Uzbekistan. email: amanova0176@gmail.com

Авторлар туралы мәлімет:

1. **Н.Д. Аманова** (автор-корреспондент) – PhD, доцент, Термез мемлекеттік университетінің химия факультеті, Термез қ., Өзбекстан. email: amanov0176@gmail.com

2. **Х.Х. Тураев** – химия ғылымдарының докторы, Термез мемлекеттік университетінің химия факультетінің профессоры, Термез, Өзбекстан. email: hhturayev@rambler.ru

3. **А.Т. Джалилов** – химия ғыл. докт., профессор, Өзбекстан Республикасы Ғылым академиясының академигі, Ташкент химия-технология ғылыми-зерттеу институтының директоры. Ташкент қ., Өзбекстан. email: gup_tniixt@mail.ru

4. **А.К. Номозов** – PhD, доцент, Термез мемлекеттік инженерлік және агротехнологиялар университетінің «Химиялық инженерия» кафедрасының меңгерушісі. Термез қ., Өзбекстан. email: abornomozov055@gmail.com

5. **Е.С. Соттикуллов** – доцент, Ташкент химия-технология ғылыми-зерттеу институтының «Химиялық инженерия» зертханасы кафедрасының меңгерушісі, доцент. Ташкент қ., Өзбекстан. email: gup_tniixt@mail.ru

6. **Я.А. Махмудова** – Өзбекстан Ұлттық университетінің химия факультетінің студенті. Ташкент қ., Өзбекстан. email: amanova0176@gmail.com