UTILIZATION OF SULFUR SLUDGE FROM SULFURIC ACID PRODUCTION IN SULFUR CONCRETE FOR BUILDING APPLICATIONS

Moataz F. Ibrahem $^{(1)}$; Osama Hodhod $^{(3)}$; Mohamed M. Ghobashy $^{(4)}$; Taha M. A. Razak $^{(1)}$

1) Faculty of Graduate Studies and Environmental Research, Ain Shams University, Cairo, Egypt 2) Nasr Company for Intermediate Chemicals 3) Department of Structural Engineering, Faculty of Engineering, Cairo University, Gizeh, Egypt 4) Department of polymers, National Center for Radiation Research and Technology (NCRRT), Egyptian Atomic Energy Authority, Nasr City, Cairo, Egypt.

*Corresponding author: mizofito@gmail.com

ABSTRACT

The industrial-scale production of sulfuric acid results in substantial quantities of solid waste, typically discarded in landfills, posing serious environmental concerns. This research investigates the characterization and utilization of sulfur sludge waste in the development of modified sulfur-based concrete. The study aims to explore its potential as a partial substitute for cement, enhance its economic value, contribute to the reduction of CO₂ emissions associated with traditional cement production, and improve the physical and mechanical properties of mortar. A range of analytical techniques, including XRF, EDAX, XRD, FTIR, SEM, and TEM, were employed to examine the sulfur sludge composition. Results indicated that the sludge is primarily composed of sulfur (97.56%), calcium (2.38%), and silicon (0.23%). In parallel, compressive strength and water absorption were evaluated for both Portland cement concrete (PC) and modified sulfur concrete (SC) at intervals of 1, 2, and 28 days. The most effective formulations were identified as those incorporating 40% and 50% sulfur sludge by-products into the Portland mix, labelled SSC40 and SSC50, respectively. These mixtures exhibited enhanced compressive strengths of 21.4 MPa (SSC40) and 23.4 MPa (SSC50), compared to 20.4 MPa for standard Portland concrete. Furthermore, water absorption rates in the modified mixtures were significantly lower, ranging from 0.7% to 1.35%, whereas Portland concrete exhibited absorption rates between 7.58% and 8.06% over the same curing periods. Both concrete types were subjected to identical curing conditions, specifically ambient air curing, and tested at consistent time intervals aligned with the experimental schedule. It can be concluded that sulfur sludge from sulfuric acid industry can be utilized as additive in concrete manufacture.

Keywords: Modified Sulfur Concrete; Sulfur Sludge by-product; Compressive Strength; Water Absorption.

Ibrahem, et al.

INTRODUCTION

Elemental sulfur plays a central role in industrial chemistry, particularly in the large-scale production of sulfuric acid—a critical chemical used across various sectors, including fertilizers, petrochemicals, metal refining, textiles, paper, and water treatment. Globally, over 265 million tonnes of sulfuric acid are produced annually through a multi-step process involving sulfur combustion, catalytic oxidation of sulfur dioxide, and subsequent absorption of sulfur trioxide to produce oleum. However, during storage and handling, elemental sulfur accumulates airborne contaminants, resulting in significant levels of solid impurities. These particulates not only reduce processing efficiency but also contribute to equipment fouling and increased maintenance costs. To mitigate this, solid contaminants are removed, generating substantial amounts of sulfur-rich sludge each year.

Sulfur-rich sludge (SRS) is considered a hazardous waste due to its composition, which may include heavy metals, carcinogenic compounds, and sulfur compounds in the form of sulfates and sulfides. Its improper disposal poses serious environmental and public health risks, particularly in windy regions where particulate dispersion is likely. Transforming this waste into a valuable resource is essential from both ecological and industrial perspectives.

On the other hand, Portland cement concrete (PC) remains the dominant material in construction despite its environmental drawbacks. The production of cement is energy-intensive and contributes significantly to global greenhouse gas emissions—accounting for approximately 82% of the total CO₂ emissions associated with the concrete industry (Kim & Chae, 2016; Kurad et al., 2017; Miller, 2020). In addition to emissions, cement manufacturing generates large quantities of dust and requires heavy fuel consumption (Drack & Vázquez, 2018; Kushnir et al., 2021; Rai, 2016). Furthermore, Portland concrete is prone to cracking due to its high porosity, allowing aggressive agents to penetrate the matrix, corrode reinforcement, and compromise structural integrity (El Gamal et al., 2017).

These challenges have driven interest in alternative construction materials that are more sustainable, durable, and cost-effective. Among them, **modified sulfur concrete** (**SC**) has emerged as a promising candidate. SC is known for its fast setting time (Yang et al., 2014), recyclability, resistance to acid and radiation (Dehestani et al., 2017), low permeability, and 3506

Vol. (54); No. (12); Dec. 2025 Print ISSN 1110-0826 Online ISSN 2636 - 3178

Ibrahem, et al.

durability under frost and wear conditions (Shin et al., 2014; Szajerski et al., 2019; Toutanji et al., 2012). It also performs well in environments with low temperatures (Al-Otaibi et al., 2019) and achieves its final strength within hours without specific curing requirements (Fediuk et al., 2020). From an environmental and economic standpoint, sulfur concrete offers a dual advantage: reducing sulfur waste accumulation and lowering the reliance on cement-based materials (Sabour et al., 2019).

Utilizing industrial by-products such as sulfur sludge in concrete production contributes to waste minimization, resource conservation, and carbon footprint reduction (Khalil et al., 2018; Kurda et al., 2018). In this context, the present study aims to explore the feasibility of using sulfur sludge, generated from the sulfuric acid production process at EL-NASR COMPANY FOR INTERMEDIATE CHEMICALS (NCIC), as a partial replacement for cement and a source of sulfur in modified sulfur concrete.

The main objective of this study is to develop and evaluate a sustainable sulfur concrete mix incorporating sulfur sludge as a binder component. Specific goals include:

- Characterizing the physical and chemical properties of sulfur sludge.
- Assessing the influence of sludge content on compressive strength and water absorption.
- Optimizing the sulfur–cement ratio to balance mechanical performance and environmental benefit.
- Investigating the potential of this concrete for use in chemically aggressive or high-moisture environments such as wastewater treatment plants and coastal infrastructure.

To achieve this, sulfur sludge was pre-treated using limestone to neutralize pH levels, and its inherent moisture content was leveraged to eliminate the need for external water during mixing. The rapid-setting properties of sulfur concrete (approximately 60 minutes) and its ability to achieve strength without conventional curing offer practical benefits for field applications.

Through this investigation, the study seeks to contribute to the advancement of sustainable construction materials by proposing a viable, durable, and eco-friendly alternative to conventional concrete, while simultaneously addressing industrial waste management challenges.

MATERIALS& METHODS

1. Collection of material

Sulfur sludge samples were obtained from the filtration stage of the crude sulfur melting unit within the sulfuric acid production facilities at El-Nasr Company for Intermediate Chemicals (NCIC). Following collection, the samples were finely ground using a mechanical grinder to prepare them for further characterization. for the preparation of sulfur-based concrete, as detailed in Table 1. The methodology employed for sample preparation and mix design is illustrated in the flow diagram presented in Figure 1. Table 1 outlines the specific compositions of the concrete mixtures, including Portland cement (PC), modified sulfur sludge (SS), and water—added only in the PC mixtures."

2. Characterization of sulfur sludge:

In this study, a comprehensive characterization of the sulfur sludge was conducted using advanced analytical techniques to elucidate its structural and morphological properties and to evaluate its potential for manufacturing sulfur concrete. Sulfur sludge was characterized using X-ray fluorescence analysis (XRF), Energy Dispersive X-ray Spectroscopy (EDAX), X-ray diffraction (XRD), FTIR Spectroscopy, scanning electron microscope (SEM), and Transmission electron microscopy (TEM). To evaluate the water absorption of sulfur sludge concrete (SSC) and Portland cement (PC), concrete cubes measuring ($5 \times 5 \times 5$ cm) were immersed in water for durations of 1, 2, and 28 days. The water absorption was calculated as grams of water per gram of concrete sample (g/g) using the following formula(Elliott, 2019):

Water absorbance ratio (%) =
$$\left(\frac{W_{after} - W_{before}}{W_{before}}\right) * 100$$
) (1)

Were water, the weight of the concrete specimens after being immersed in water, and before, weight of the concrete model before being immersed in water. In addition, to assess the mechanical strength of the SC and PC mixtures immersed in water, compressive strength tests were conducted using a CHANDLER Model 4207D on concrete cubes of the same size, with two test specimens for each sample, following the guidelines of ASTM C109 and hardness testing procedures established by the Faculty of Engineering at Cairo University(Rasheed et al., 2022).

Ibrahem, et al.

3. Preparation of modified sulfur concrete:

The experimental process begins by melting sulfur sludge on a hotplate until it reaches approximately 130 °C, transitioning from a solid to a molten phase. Once fully liquefied, cement is incrementally incorporated into the molten sulfur to form a uniform blend. This mixture is then used to cast concrete cubes. The proportion of sulfur sludge varies across the prepared mixtures, ranging from 0% (denoted as PC) up to 70% (denoted as SSC70), as indicated by the corresponding sample labels. For each formulation, a 1 kg batch of the molten mix is sufficient to produce three test specimens with identical composition.

A notable aspect of this approach is the utilization of the inherent moisture content within the sulfur sludge (approximately 0.48 wt.%) to facilitate blending with cement, eliminating the need for additional water, and the pH is neutral due to the addition of limestone during the sulfur melting and filtration process. In contrast to conventional concrete formulations. Among the different compositions, the mix designated SSC50, containing equal parts cement and sulfur sludge, demonstrated a promising compromise between mechanical strength and environmental considerations. Increasing the sulfur sludge content beyond 50% tends to reduce compressive strength, likely due to insufficient cement content to ensure cohesive bonding and structural integrity.

On the other hand, increasing the cement content above 50% may lead to inadequate mixing due to limited moisture, resulting in inhomogeneous distribution and cement agglomeration.

The fabrication procedure is illustrated schematically in Figure 1. After achieving homogeneity, the mixture is cast into standard molds with dimensions of 50 mm × 50 mm × 50 mm. Curing is conducted under ambient laboratory conditions. A significant advantage of this modified sulfur-based concrete is its rapid demolding time, allowing removal within approximately 60 minutes. In contrast to conventional concrete, it does not require extended curing periods or controlled environments to gain early strength. The data presented in Table 1 and Figure 1 highlight the feasibility of incorporating sulfur sludge at varying levels. Mixtures containing between 40% and 50% sulfur sludge appear to offer a favorable balance of compressive strength, durability, water resistance, and environmental performance. Ongoing

Ibrahem, et al.

evaluation of mechanical and physical properties—including compressive strength and water absorption—is essential to optimize the formulation for specific engineering applications. Given its potential chemical resistance and rapid setting properties, this type of concrete may be particularly suitable for use in aggressive environments such as wastewater facilities, underground pipelines, and marine or coastal structures. Continued research is warranted to validate and expand the applicability of this material.

Samples were prepared in the central laboratories of El Nasr Company for Intermediate Chemicals.

Table 1. Different components of sulfur sludge are used to make sulfur concrete models.

SAMPLE	PORTLAND CEMENT (PC) (KILOGRAM)	SULFUR SLUDGE (SS) (KILOGRAM)	WATER (LITER)	CODE
1	1	0	0.3	PC
2	0.7	0.3	0	SSC30
3	0.6	0.4	0	SSC40
4	0.5	0.5	0	SSC50
5	0.4	0.6	0	SSC60
6	0.3	0.7	0	SSC70

Note: The sulfur concrete was prepared without the addition of water, as it was mixed with molten sulfur sludge. The process relied on the inherent moisture content present within the sulfur sludge.

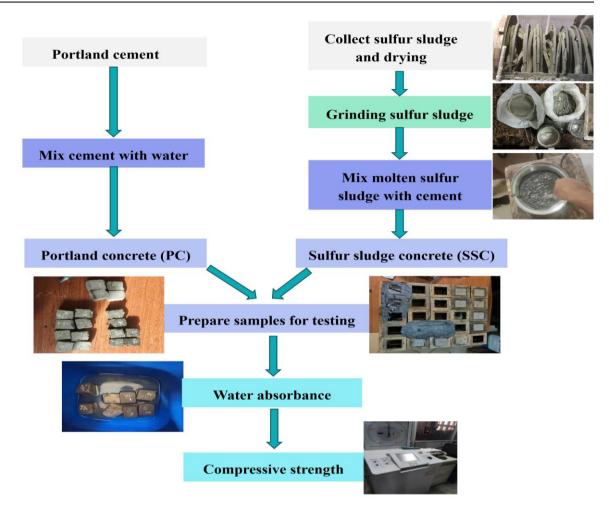


Figure 1. Schematic of the preparation of cement concrete and sulfur concrete

RESULTS

All tests were conducted at the Faculty of Engineering, Cairo University, and the analyses were carried out at the Central Laboratories of the Armed Forces.

The XRF

The XRF analysis data in **Table 2** indicate that the primary component of sulfur sludge is elemental sulfur, accompanied by several oxides, including silica, iron, and aluminum oxides. Additionally, the analysis reveals the presence of heavy metals at low concentrations, which do not exceed the established limit and guidance values. X-ray fluorescence (XRF) analysis showed that the sulfur sludge is composed primarily of elemental sulfur (97.56%), with minor

amounts of calcium (2.38%) and silicon (0.23%)(Chubarov et al., 2016; Hrdlička et al., 2018)

Table 2. Chemical composition of sulfur sludge.

ELEMENT	S	CA	SI	MG	FE	AL	CD	P	RU	CU
Concentration (wt%)	97.56	2.38	0.23	0.22	0.16	0.08	0.08	0.06	0.02	0.01

The sulfur percentage is above 97% and depends on the quality of sulfur during purchase and storage. In the worst cases, it may reach 55%.

Table 3. Comparison of chemical compositions between sulfur sludge from Abu Zaabal and NCIC (wt.%).

Sulfur sludge	e Concentration (wt%)									
type	S	SiO ₂	Fe ₂ O ₃	Al_2O_3	MgO	CaO	Na ₂ O	K ₂ O	P	CdO
Abu Zaabal	56	32.7	4	2.5	0.41	0.50	0.30	0.18	0	0
NCIC	97.5	0.23	0.16	0.08	0.22	2.38	0	0	0.06	0.08

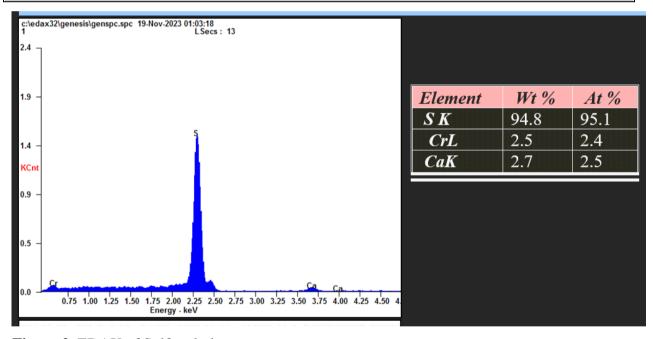


Figure 2. EDAX of Sulfur sludge.

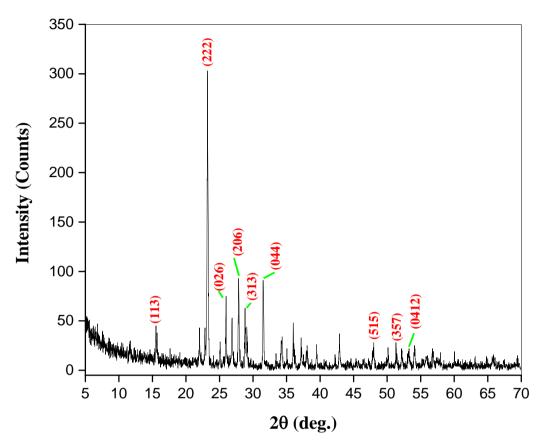
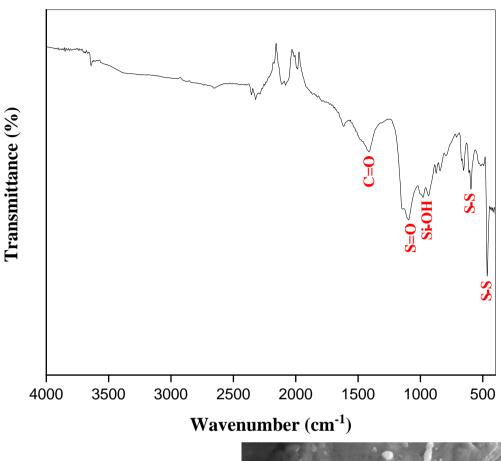


Figure 3. XRD of Sulfur sludge.



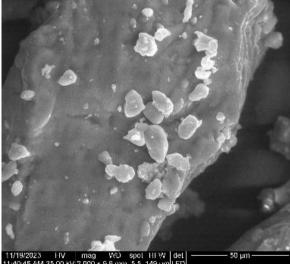


Figure 4. FTIR spectrum of Sulfur sludge. 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2023 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11/19/2025 11

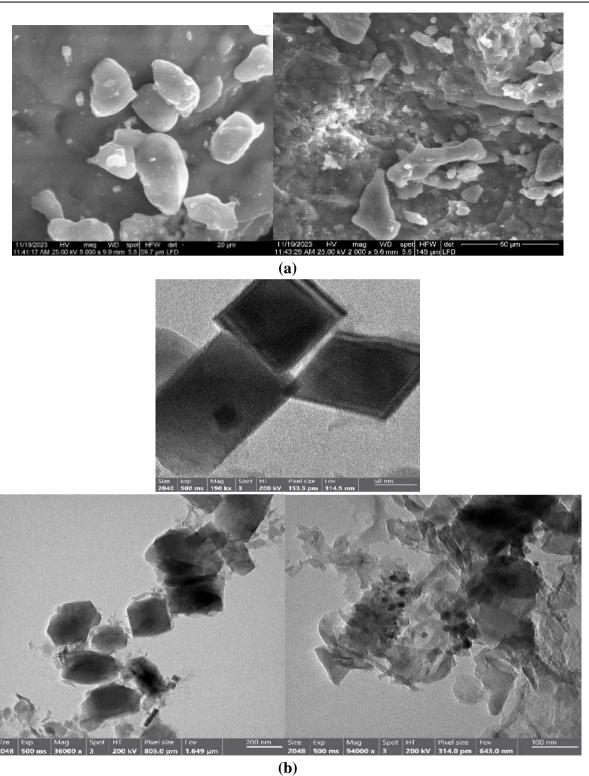


Figure 5. Morphology of sulfur sludge determined by (a) SEM, (b) TEM.

Ibrahem, et al.

Table 4. Compressive strength of the prepared cement mortar after 28 days.

SAMPLE	FAILURE LOAD KN	COMPRESSIVE STRENGTH MPA (N/MM²)
PC	51.33	20.53
SSC30	25	10
SSC40	54.5	21.8
SSC50	58.5	23.4
SSC60	34	13.6
SSC70	26	10.4

Compressive strength measurements were performed on both conventional concrete and modified sulfur concrete, with all specimens subjected to identical curing conditions, specifically, ambient air exposure under normal ventilation for an equal duration. The results revealed comparable strength values between the two types in the absence of any external stressors or environmental influences. These findings provide evidence supporting the durability and structural reliability of the modified sulfur concrete under standard curing environments.

It is worth noting that both units—kN and MPa (N/mm²)—have been included in the table to facilitate comparison with findings from other studies on the same subject. This dual-unit presentation also ensures ease of comparison for future research involving similar experimental parameters.

It is important to note that increasing the sulfur sludge content beyond 50% leads to a noticeable reduction in compressive strength, particularly evident at a concentration of 55% during testing. As a result, a 50% sulfur sludge content was selected as the optimal ratio. Conversely, increasing the cement content beyond 50% results in poor homogeneity due to the low moisture content in the sulfur sludge, which leads to sample clumping and failure to form a cohesive mixture.

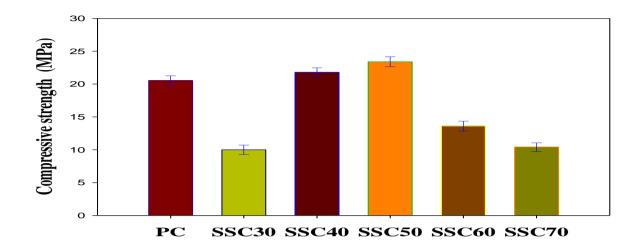


Figure 6. Comparison of Compressive strength for the prepared cement mortar after 28 days.

Analysis of Standard Deviation Calculation:

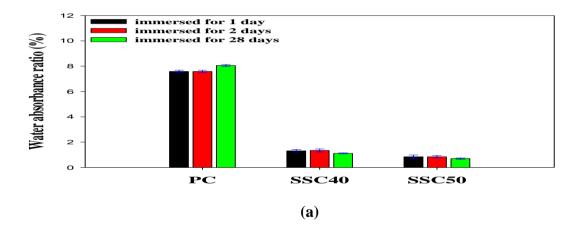
The experimental results demonstrate a clear influence of sulfur content on the compressive strength of sulfur-modified concrete. Samples with moderate sulfur-to-cement ratios (such as SSC40 and SSC50) exhibited higher compressive strength values (21.8 MPa and 23.4 MPa respectively), while those with either very low (SSC30) or very high (SSC70) sulfur content showed significantly reduced strength (10.0 MPa and 10.4 MPa respectively).

The compressive strength ranged from 10.0 MPa to 23.4 MPa, with a mean value of 16.6 MPa and a standard deviation of 5.98 MPa, indicating moderate variability among the samples.

These findings suggest that while sulfur sludge can be utilized as a partial replacement in cementitious composites, there exists an optimal sulfur content range beyond which mechanical performance diminishes. The results are consistent with previously published data, confirming the importance of optimizing the mix proportion in sulfur-based concrete.

Table 5. Water absorption ratio and Compressive strength for the PC, SSC40, and SSC50 cement mortars after immersion in water for 1,2,28 days.

SAMPLE	DAYS	SPECIMENS WEIGHT		WATER ABSORBANCE	FAILURE LOAD	COMPRESSIVE STRENGTH	COMPRESSIVE STRENGTH
		before	after	RATIO (%)	(KN)	MPA (N/MM ²)	(KG/CM^2)
PC	1	323	347.5	7.58	16	6.4	64
SSC40	1	340.5	345	1.32	41	16.4	164
SSC50	1	351	354	0.85	46	18.4	185
PC	2	310	333.5	7.58	26.5	10.6	106
SSC40	2	332	336.5	1.35	45	18	180
SSC50	2	352	355	0.85	47.5	19	190
PC	28	322.5	348.5	8.06	51	20.4	204
SSC40	28	342.5	346.5	1.12	53	21.2	212
SSC50	28	356	358.5	0.70	57.5	23	230



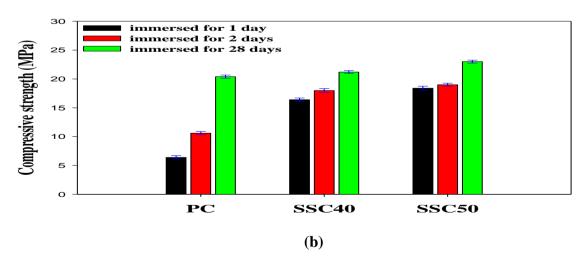


Figure 7. Comparison of **(a)** Water absorbance ratio, **(b)** Compressive strength for the prepared cement mortar after immersed in water for 1, 2, and 28 days.

Ibrahem, et al.

Table 6. Comparison of compressive strength for sulfur concrete made with sludge versus raw sulfur (the obtained data was compared with that of (Eri, 2022)).

SOURCE OF SULFUR	AGE (DAYS)	AVERAGE OF STRONG PRESS (KG/CM ²)
Sulfur sludge 50% (SSC50)	1	185
Sulfur raw 30%		223
Sulfur sludge 50% (SSC50)	2	190
Sulfur raw 30%		226,5
Sulfur sludge 50% (SSC50)	28	230
Sulfur raw 30%		272

This comparison was made against a study utilizing sulfur concrete produced with pure elemental sulfur. Although the compressive strength results were slightly higher for raw sulfur-based concrete, the cost efficiency of using sulfur sludge is considerably more advantageous. As a by-product, sulfur sludge provides a significant economic benefit, making it a more sustainable and practical alternative—especially given that the performance difference remains marginal.

DISCUSSION

X-ray fluorescence (XRF)

The XRF analysis data in **Table 2** indicate that the primary component of sulfur sludge is elemental sulfur, accompanied by several oxides, including silica, iron, and aluminum oxides. Additionally, the analysis reveals the presence of heavy metals at low concentrations, which do not exceed the established limit and guidance values. X-ray fluorescence (XRF) analysis showed that the sulfur sludge is composed primarily of elemental sulfur (97.56%), with minor amounts of calcium (2.38%) and silicon (0.23%)(Chubarov et al., 2016; Hrdlička et al., 2018). This high sulfur content indicates that the sludge is a viable source of sulfur for concrete production. Calcium and silicon, likely in oxide forms, suggest some contamination from soil or dust particles. The trace amounts of other elements detected, including magnesium, iron, aluminum, cadmium, phosphorus, ruthenium, and copper, are typical impurities in industrial sulfur waste streams.(Gazulla et al., 2009). Their low concentrations (all under 0.25%) indicate effective sulfur purification during processing. The XRF results provide crucial compositional

Ibrahem, et al.

data to assess the suitability of this sludge for reuse in construction materials. (Uhlig et al., 2016).

Energy Dispersive X-ray Spectroscopy (EDAX)

The EDAX data in **Figure 2** demonstrate that the sulfur sludge contains an exceptionally high sulfur content, reaching nearly 95%. Energy Dispersive X-ray Spectroscopy (EDX) results corroborated the XRF findings, showing an unusually high sulfur content of almost 95%. The EDX technique provides elemental composition data from a smaller sample volume than XRF, which explains the slight difference in sulfur percentage. The detection of trace amounts of chromium and calcium by EDX, which were not prominent in the XRF results, highlights the complementary nature of these analytical techniques. Chromium, if present as a chromate compound, could potentially impact the setting behavior of sulfur concrete (Singh & Srivastava, 2022). The high sulfur purity confirmed by EDX supports using this sludge as an alternative to raw sulfur in sulfur concrete production, potentially offering economic and environmental benefits.

X-ray diffraction (XRD)

X-ray diffraction (XRD) analysis identified that the crystalline form of sulfur in the sludge is primarily cyclooctasulfur (S8). Distinct diffraction peaks were observed at 2θ values of 15.4°, 23.1°, 25.8°, 27.8°, 28.7°, 31.4°, 47.75°, 51.2°, and 53.06°, corresponding to the crystal planes of S8, confirming its orthorhombic structure(Khavari et al., 2023; Sarapajevaite & Baltakys, 2021). Cyclooctasulfur (S8) is the most stable allotrope of sulfur at room temperature. It is advantageous for its use in sulfur concrete applications due to its low melting point (~115°C), facilitating easy processing and molding. The sharpness and intensity of the XRD peaks indicate a high degree of crystallinity, further suggesting that the sulfur is in a stable and pure form.(Ongarbayev et al., 2024). No significant peaks corresponding to the minor elements detected in the XRF analysis (such as calcium or silicon) were observed, which implies that these elements are either amorphous or present in quantities too low for XRD detection. This high crystallinity is desirable in construction materials, as it ensures predictable behavior under thermal conditions and during the solidification process in concrete applications.(Shankar et al., 2018; Singh & Srivastava, 2022). The XRD results reinforce the 3520

Vol. (54); No. (12); Dec. 2025 Print ISSN 1110-0826 Online ISSN 2636 - 3178

Ibrahem, et al.

potential for this sulfur sludge to be effectively utilized in sulfur concrete production. Since sulfur is the predominant component of the sulfur sludge, only sulfur was detected in the XRD spectrum, while other compounds were identified in the XRF analysis (**Table 2**) (Shankar et al., 2018; Singh & Srivastava, 2022).

FTIR-spectroscopic analysis

The Fourier Transform Infrared (FTIR) analysis reveals specific peaks in the spectra that provide essential insights into the functional groups present in the samples. In the FTIR spectra of sulfur sludge shown in **Figure 3**, a band at 1414 cm⁻¹ is associated with adsorbed CO₂ from the atmosphere during sample preparation(Taş et al., 2002). A prominent band observed in the 1130 to 1000 cm⁻¹ range, with a peak intensity at 1095 cm⁻¹, corresponds to the stretching of sulfoxide (S=O)(Oliveira et al., 2016). Similar bands associated with sulfate have been reported in the FTIR analysis of pyrite, a sulfur ore, within the range of 900 to 1200 cm⁻¹ (Shareef et al., 2023; Sharma et al., 2003). Additionally, a band at 653 cm⁻¹ is linked to Si–O stretching(Mureddu et al., 2014). Bands at 464 cm⁻¹ and 594 cm⁻¹ correspond to disulfide (S–S) bonds between adjacent thiol groups(Baruah et al., 2018; Vinayan et al., 2016). These findings are consistent with previous studies(Shareef et al., 2023; Sharma et al., 2003).

Morphology and microstructure analysis (SEM& TEM):

The surface morphology and microstructure of the sulfur sludge used to prepare sulfur concrete were examined using scanning electron microscopy (SEM) and transmission electron microscopy (TEM), as depicted in **Figure 5**. Scanning Electron Microscopy (SEM) analysis revealed that the sulfur sludge particles predominantly exhibit irregular spherical shapes. This morphology is primarily due to the high sulfur content (>95%) and minor impurities. The spherical nature of the particles can be attributed to the tendency of molten sulfur to form droplets upon cooling, which then solidify into roughly spherical shapes. The irregularities in these shapes likely result from impurities and variations in cooling rates during sludge formation. This particle morphology can significantly influence the packing density and flowability of the sludge when used in sulfur concrete mixtures. The irregular surfaces of the particles may also provide an increased surface area for bonding within the concrete matrix, potentially enhancing the strength of the final product. (Dong et al., 2023).

Ibrahem, et al.

Transmission Electron Microscopy (TEM) provided high-resolution imaging of the sulfur sludge particles, complementing the SEM analysis. The TEM images likely showed the sulfur particles' internal structure and crystalline nature, consistent with the XRD results indicating the presence of cyclooctasulfur (S8). The high magnification capabilities of TEM would have revealed any nanoscale features or inclusions within the particles that were not visible in SEM. This could include the distribution of impurities or the presence of amorphous regions within the crystalline sulfur matrix. (Yin et al., 2021). The TEM analysis is crucial for understanding the nanostructure of the sulfur sludge, which can influence its melting behavior, reactivity, and overall performance in sulfur concrete applications. The combination of SEM and TEM provides a comprehensive view of the sludge's morphology across multiple scales, from micron-sized particles to nanoscale features.

A comparative analysis of compressive strength for sulfur concrete made with sludge versus raw sulfur highlights significant differences from the results of Eri, 2022 who used sulfur as a raw material to produce sulfur concrete. Their study reported achieving maximum compressive strength at a given sulfur content. The data from comparison of the compressive strength of sulfur concrete made with 50% sulfur sludge (SSC50) with concrete made with 30% raw sulfur, as reported in a previous study show that at all ages tested (1, 2, and 28 days), concrete made with raw sulfur exhibits slightly higher compressive strength than SSC50. For example, after 28 days, the raw sulfur concrete reached a strength of 272 kg/cm², while the SSC50 reached 230 kg/cm². This slight difference in strength can be attributed to several factors, including the higher purity of raw sulfur compared to sulfur sludge, possible differences in mixing techniques (Portland cement was used with the sulfur sludge while aggregate was used with raw sulfur), and the significant cost advantage of sulfur sludge over raw sulfur. This comparison highlights the need for successful sulfur sludge-modified concrete mixes compared to conventional sulfur concrete with pure raw sulfur. The utilization of waste sulfur sludge contributes to environmental sustainability while reducing dependency on imported raw sulfur, which is not locally available in Egypt.

Ibrahem, et al.

CONCLUSION

This study investigates the use of sulfur sludge—an environmentally harmful by-product of sulfuric acid and sulfur concrete production—as a partial or full replacement for conventional sulfur in modified sulfur concrete. This approach supports sustainable waste management, reduces dependency on imported sulfur, and contributes to lowering CO₂ emissions by decreasing reliance on Portland cement. The resulting mixtures demonstrated enhanced mechanical performance and durability, indicating strong potential for industrialscale applications The material was initially characterized using a range of analytical techniques, including XRF, EDS, XRD, FTIR, SEM, and TEM. The results revealed that the sulfur sludge mainly consisted of 97.56% sulfur, 2.38% calcium, and 0.23% silicon, and the sulfur sludge particles predominantly exhibit irregular spherical shapes. The compressive strength and water absorption of Portland concrete (PC) and sulfur concrete (SC) were evaluated after 1, 2, and 28 days. It was found that the optimal proportions for preparing sulfur concrete involved mixing the sulfur sludge by-product at 40% and 50% (designated as SSC40 and SSC50) with Portland cement. This mixture demonstrated improved compressive strengths of 21.4 MPa (SSC40) and 23.4 MPa (SSC50), outperforming standard Portland concrete with a compressive strength of 20.4 MPa. Additionally, the experiments showed that the water absorption ratio for sulfur sludge concrete ranged from 0.7% to 1.35%, while Portland concrete absorbed between 7.58% and 8.06% during the same period. After 1 day of immersion in water, the compressive strength was recorded at 6.4 MPa for Portland concrete (PC), 16.4 MPa for SSC40, and 18.4 MPa for SSC50. After 28 days of immersion, these values increased to 20.4 MPa, 21.2 MPa, and 23.0 MPa, respectively, Further research should focus on long-term durability assessments and microstructural analysis to better understand phase transformations and bonding behavior within the modified sulfur matrix.

REFERENCES

- Al-Otaibi, S., Al-Aibani, A., Al-Bahar, S., Abdulsalam, M., & Al-Fadala, S. (2019). Potential for producing concrete blocks using sulphur polymeric concrete in Kuwait. *Journal of King Saud University-Engineering Sciences*, 31(4), 327-331.
- Baruah, B., Tiwari, P., Thakur, P., & Kataki, R. (2018). TGA-FTIR analysis of Upper Assam oil shale, optimization of lab-scale pyrolysis process parameters using RSM. *Journal of Analytical and Applied Pyrolysis*, 135, 397-405.
- Chubarov, V., Amosova, A., & Finkelshtein, A. (2016). X- ray fluorescence determination of sulfur chemical state in sulfide ores. *X- Ray Spectrometry*, 45(6), 352-356.
- Dehestani, M., Teimortashlu, E., Molaei, M., Ghomian, M., Firoozi, S., & Aghili, S. (2017). Experimental data on compressive strength and durability of sulfur concrete modified by styrene and bitumen. *Data in brief*, *13*, 137-144.
- Dong, H., Sun, Y.-L., Sun, Q., Zhang, X.-N., Wang, H.-C., Wang, A.-J., & Cheng, H.-Y. (2023). Effect of sulfur particle morphology on the performance of element sulfur-based denitrification packed-bed reactor. *Bioresource technology*, *367*, 128238.
- Drack, J. M. E., & Vázquez, D. P. (2018). Morphological response of a cactus to cement dust pollution. *Ecotoxicology and environmental safety*, 148, 571-577.
- Elliott, K. S. (2019). Precast concrete structures. Crc Press.
- Eri, I. E. S. R. (2022). Jurnal: analysis of compressive strength of sulfur concrete. *Journal of Mechanical, Civil and Industrial Engineering*, 3(2), 7-16.
- Fediuk, R., Mugahed Amran, Y., Mosaberpanah, M. A., Danish, A., El-Zeadani, M., Klyuev, S. V., & Vatin, N. J. M. (2020). A critical review on the properties and applications of sulfur-based concrete. *13*(21), 4712.
- Gazulla, M., Gomez, M., Orduna, M., & Rodrigo, M. (2009). New methodology for sulfur analysis in geological samples by WD- XRF spectrometry. *X- Ray Spectrometry: An International Journal*, *38*(1), 3-8.
- Hrdlička, A., Hegrová, J., Novotný, K., Kanický, V., Prochazka, D., Novotný, J.,...Kaiser, J. (2018). Sulfur determination in concrete samples using laser-induced breakdown spectroscopy and limestone standards. *Spectrochimica Acta Part B: Atomic Spectroscopy*, 142, 8-13.
- Khalil, M., Abdel Razek, T., El-Fiki, M., & Youssef, M. (2018). Utilization of sulfur sludge results from the manufacturing of sulfuric acid in a filter aid production. *Journal of Environmental Sciences*, 3, 19-34.
- Khavari, B. C., Shekarriz, M., Aminnejad, B., Lork, A., & Vahdani, S. (2023). Laboratory evaluation and optimization of mechanical properties of sulfur concrete reinforced with micro and macro steel fibers via response surface methodology. *Construction and Building Materials*, 384, 131434.
- Kim, T. H., & Chae, C. U. (2016). Environmental impact analysis of acidification and eutrophication due to emissions from the production of concrete. *Sustainability*, 8(6), 578.

- Kurda, R., Silvestre, J. D., & de Brito, J. (2018). Life cycle assessment of concrete made with high volume of recycled concrete aggregates and fly ash. *Resources, conservation and recycling*, 139, 407-417.
- Kushnir, A. R., Heap, M. J., Griffiths, L., Wadsworth, F. B., Langella, A., Baud, P.,...Utley, J. E. (2021). The fire resistance of high-strength concrete containing natural zeolites. *Cement and Concrete Composites*, 116, 103897.
- Kurad, R., Silvestre, J. D., de Brito, J., & Ahmed, H. (2017). Effect of incorporation of high volume of recycled concrete aggregates and fly ash on the strength and global warming potential of concrete. *Journal of Cleaner Production*, 166, 485-502.
- Miller, S. A. (2020). The role of cement service-life on the efficient use of resources. *Environmental Research Letters*, 15(2), 024004.
- Mureddu, M., Ferino, I., Musinu, A., Ardu, A., Rombi, E., Cutrufello, M. G.,...Cannas, C. (2014). MeO x/SBA-15 (Me= Zn, Fe): highly efficient nanosorbents for midtemperature H 2 S removal. *Journal of Materials Chemistry A*, 2(45), 19396-19406.
- Oliveira, C., Machado, C., Duarte, G., & Peterson, M. (2016). Beneficiation of pyrite from coal mining. *Journal of Cleaner Production*, 139, 821-827.
- Ongarbayev, Y., Tileuberdi, Y., Baimagambetova, A., Imanbayev, Y., Kanzharkan, Y., Zhambolova, A.,...Kydyrali, A. (2024). Modification of Sulfur Cake—Waste from Sulfuric Acid Production. *Processes*, 12(9), 2048.
- Rai, P. K. (2016). Impacts of particulate matter pollution on plants: Implications for environmental biomonitoring. *Ecotoxicology and environmental safety*, 129, 120-136.
- Rasheed, M. F., Rahim, A., Irfan-ul-Hassan, M., Ali, B., & Ali, N. (2022). Sulfur concrete made with waste marble and slag powders: 100% recycled and waterless concrete. *Environmental Science and Pollution Research*, 29(43), 65655-65669.
- Sabour, M., Dezvareh, G., & Bazzazzadeh, R. (2019). Corrosion prediction using the weight loss model in the sewer pipes made from sulfur and cement concretes and Response Surface Methodology (RSM). *Construction and Building Materials*, 199, 40-49.
- Sarapajevaite, G., & Baltakys, K. (2021). Purification of sulfur waste under hydrothermal conditions. *Waste and Biomass Valorization*, *12*, 3407-3416.
- Shankar, S., Pangeni, R., Park, J. W., & Rhim, J.-W. (2018). Preparation of sulfur nanoparticles and their antibacterial activity and cytotoxic effect. *Materials Science and Engineering:* C, 92, 508-517.
- Shareef, S. S., Thaeir, A., & Motea, O. (2023). Concentration and study of the carbon-sulfur residues resulting from the thermal purification of Fracsh sulfur in the Al-Mishraq sulfur mine. *Journal of Survey in Fisheries Sciences*, 10(1S), 5355-5367.
- Sharma, P., Das, A., Rao, K. H., & Forssberg, K. (2003). Surface characterization of Acidithiobacillus ferrooxidans cells grown under different conditions. *Hydrometallurgy*, 71(1-2), 285-292.
- Singh, V., & Srivastava, V. C. (2022). Insight into the thermal kinetics and thermodynamics of sulfuric acid plant sludge for efficient recovery of sulfur. *Waste management*, 140, 233-244.

- Taş, A. C., Majewski, P. J., & Aldinger, F. (2002). Synthesis of gallium oxide hydroxide crystals in aqueous solutions with or without urea and their calcination behavior. *Journal of the American Ceramic Society*, 85(6), 1421-1429.
- Toutanji, H. A., Evans, S., & Grugel, R. N. (2012). Performance of lunar sulfur concrete in lunar environments. *Construction and Building Materials*, 29, 444-448.
- Uhlig, S., Möckel, R., & Pleßow, A. (2016). Quantitative analysis of sulphides and sulphates by WD- XRF: Capability and constraints. *X- Ray Spectrometry*, 45(3), 133-137.
- Vinayan, B., Zhao-Karger, Z., Diemant, T., Chakravadhanula, V. S. K., Schwarzburger, N. I., Cambaz, M. A.,...Fichtner, M. (2016). Performance study of magnesium—sulfur battery using a graphene based sulfur composite cathode electrode and a non-nucleophilic Mg electrolyte. *Nanoscale*, 8(6), 3296-3306.
- Yang, C., Lv, X., Tian, X., Wang, Y., & Komarneni, S. (2014). An investigation on the use of electrolytic manganese residue as filler in sulfur concrete. *Construction and Building Materials*, 73, 305-310.
- Yin, S., Hou, Y., Chen, X., & Zhang, M. (2021). Mechanical, flowing and microstructural properties of cemented sulfur tailings backfill: Effects of fiber lengths and dosage. *Construction and Building Materials*, 309, 125058.

الاستغادة من الحمأة الكبريتية الناتجة عن إنتاج حامض الكبريتيك للاستغادة من الحرسانة الكبريتية لتطبيقات البناء

معتز فتحي إبراهيم $^{(1)}$ أسامة عبدالغفور أحمد هدهد $^{(3)}$ محمد محمدي غباشي $^{(4)}$ طه عبد العظيم محمد عبد الرازق $^{(1)}$

1) كلية الدراسات العليا والبحوث البيئة، جامعة عين شمس 2) شركه النصر للكيماويات الوسيطة 3) قسم الهندسة الإنشائية، كلية الهندسة، جامعة القاهرة، الجيزة 4) قسم البوليمرات، المركز القومي لبحوث وتكنولوجيا الإشعاع، هيئة الطاقة الذربة، القاهرة.

* المؤلف المراسل: mizofito@gmail.com

المستخلص

يُنتِج التصنيع واسع النطاق لحمض الكبريتيك كميات كبيرة من النفايات الصناعية الصلبة، والتي غالباً ما ما يتم التخلص منها في مدافن النفايات ممايشكل مخاطربيئية كبيرة. تهدف هذه الدراسة إلى توصيف الحمأة الكبريتية واستكشاف إمكانية استخدامها كمادة بديلة في إنتاج الخرسانة الكبريتية. تم تحليل خصائص الحمأة باستخدام مجموعة من التقنيات المتقدمة تشمل: التحليل الفلوري بالأشعة السينية(XRF) ، التحليل الطيفي للطاقة المشتتة(EDAX) ، حيود الأشعة السينية (XRD) ، التحليل الطيفي للطاقة المشتة(SEM) ، والمجهر الإلكتروني النافذ (XRD) ، التحليل الطيفي للأشعة تحت الحمراء(FTIR) ، المجهر الإلكتروني الماسح(SEM) ، والمجهر الإلكتروني النافذ (TEM) .أظهرت النتائج أن التركيب الكيميائي الأساسي للحمأة يتكون من 97.56% كبريت، 2.38% كالسيوم، و 20.3% سيليكون. وفي سياق الدراسة، تم تقييم كل من مقاومة الضغط ونسبة امتصاص الماء لعينات من الخرسانة البورتلاندية (PC)والخرسانة الكبريتية (SC) خلال فترات زمنية تمتد إلى 1، 2، و28 يوماً. تم تحديد التركيبتين الأمثل للخرسانة الكبريتية عبر دمج 40% و 50% من مخلفات الحمأة مع الأسمنت البورتلاندي، وسُميت التركيبتان الناتجتان بـ 23.4 SSC40 و SSC40 و SSC40 ميجا باسكال لعينة SSC40 و 23.4 ميجا باسكال لعينة SSC40 و 23.4 ميجا باسكال لعينة SSC40 و 23.4 ميجا

Ibrahem, et al.

باسكال لعينة SSC50 ، متجاوزة الخرسانة البورتلاندية التقليدية التي سجلت مقاومة قدرها 20.4 ميجا باسكال. كما كشفت نتائج الامتصاص المائي انخفاضاً ملحوظاً في الخرسانة الكبريتية (بين 7.0% و 1.35%) مقارنة بالخرسانة البورتلاندية (بين 7.58% و 8.06%) خلال الفترة نفسها.عند اختبار مقاومة الضغط بعد يوم واحد من الغمر بالماء، تم تسجيل 6.4 ميجا باسكال للغرسانة PC ميجا باسكال لعينة SSC50 أما بعد 28 يوماً ميباً باسكال للخرسانة 6.4 ميجا باسكال لعينة 20.4 ميجا باسكال العينة 18.4 ميجا باسكال الغمر، فقد ارتفعت هذه القيم إلى 20.4، و21، و22 ميجا باسكال، على التوالي. يمكن الاستنتاج أن حمأة الكبريت الناتجة عن صناعة حامض الكبريتيك يمكن استخدامها كمادة مضافة في تصنيع الخرسانة.

الكلمات المفتاحية: الخرسانة الكبريتية؛ خبث الكبريت كمنتج ثانوي؛ قوة الأنضغاط؛ امتصاص الماء