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CIRCULAR ECONOMY IN PRACTICE: PRODUCTION OF SUSTAINABLE COMPOSITES FROM WASTE

An element of the circular economy strategy is processing waste into useful products. This study aimed to produce materials incorporating hazardous waste contaminated with heavy metals. Sulfur polymer cement (SPC) modified with natural tung oil was used as the binder. Compared to Portland cement, sulfur-based binders not only reduce CO_2 emissions but also enable the utilization of sulfur waste, such as byproducts from the petrochemical industry. Samples were prepared using an SPC modified with different amounts of tung oil (2–8 wt % S) and two filler mixtures containing 6 and 12% waste (mixes A and B). The composites were tested for water absorption by immersion, compressive strength, and metal leaching toxicity (TCLP test and EN 12457-4). The water absorption of all samples was below 1.6 wt %. Worse results were observed for monoliths with higher waste content (mix B). The highest mechanical strength was observed for samples where the sulfur binder was modified with 4 wt % tung oil. The compressive strength of composites based on mixtures A and B was 8.0 and 11.5 MPa, respectively. Analysis of the samples showed an uneven coating of waste particles by the sulfur binder. The TCLP test indicated a potential risk of heavy metal leaching in an acidic environment (pH ca. 4). Conversely, the EN 12457-4 test demonstrated low heavy metal leaching for all mixture A composites.

1. INTRODUCTION

Proper industrial waste management is crucial for environmental protection and sustainable development. Many types of industrial waste have significant raw material potential. Their use as alternative materials is consistent with the principles of the circular economy. However, despite advancements in recycling technologies and rising landfill costs, a substantial portion of industrial waste ends up in landfills. For example, over 40% of industrial waste is landfilled in Poland. Improper waste management not only

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results in the loss of valuable resources but also can lead to uncontrolled pollutant emissions into the environment. Achieving circular economy goals such as minimizing the consumption of primary raw materials, reducing waste generation, and optimizing production processes through renewable materials use requires cross-sectoral collaboration [1]. The construction industry presents a high-potential sector for the alternative applications of industrial waste such as metallurgical slags, sulfur, and basalt screenings [2].

One of the current challenges is the management of sulfur, a byproduct of the petrochemical industry. Global sulfur production is estimated at approximately 70 million tons annually [3], while its market demand remains limited. Furthermore, forecasts suggest that sulfur production will continue to increase with economic growth. Sulfur overproduction leads to its storage, which imposes environmental risks and generates additional costs. Developing technologies for producing durable and eco-friendly sulfur-based materials presents a viable solution for its utilization. One approach consistent with the principles of the circular economy is using sulfur in construction binders as an alternative to Portland cement, whose manufacturing process is highly energy-intensive and emission-heavy. Producing Portland cement requires temperatures of up to 1400 °C, consumes significant amounts of natural resources, and is responsible for 8–10% of global carbon dioxide emissions. Most of the CO_2 emissions from cement production are classified as process emissions, primarily resulting from the decomposition of calcium carbonate. Moreover, the environmental impact of raw material extraction for cement production is also significant [4–7].

Sulfur binders are characterized by a low production temperature of 140–160 °C and do not require water [8]. These factors contribute to a reduction in CO_2 emissions during production, making sulfur binders an attractive alternative to conventional binding materials. However, an important condition is to obtain a durable product with good mechanical properties. In sulfur-based systems, preventing sulfur transformation to the orthorhombic form during cooling is essential. Uncontrolled shrinkage and internal stresses negatively affect mechanical performance. To mitigate phase transitions, modifiers that enhance the cross-linking of the sulfur cement matrix, such as dicyclopentadiene or styrene, are commonly used [4, 9]. However, the use of toxic organic solvents is not in line with the principles of sustainable development. They can be replaced with neutral, bio-based alternatives [7, 10].

Based on modified sulfur binders and mineral waste such as slags or fly ash, it is possible to produce materials with properties at least comparable to traditional products [2, 11, 12]. Additionally, their hydrophobic properties and resistance to acids and salts make them suitable for specialized applications [2]. Sulfur binders can also be used for waste encapsulation [8]. Incorporation of waste into sulfur-polymer mixtures helps reduce the consumption of primary raw materials, lower air pollutant emissions, and decrease the quantity of waste landfilled [12]. Composites based on mineral waste and sulfur binders can be used as road subbase materials [2, 13].

This paper examines the possibilities of producing sustainable composites based on industrial waste as an example of implementing circular economy principles. The presented work has characterized and compared the mechanical and physicochemical properties of the obtained sulfur-waste materials.

2. MATERIALS AND METHODS

Sulfur cement. Sulfur polymer cement (SPC) was used as the matrix to prepare composites with mineral waste. The primary component of the binder was elemental sulfur (CAS: 7704-34-9, Chempur). To stabilize the sulfur phase transformation from monoclinic to orthorhombic and reduce binder brittleness, an environmentally friendly modifier – tung oil (CAS: 8001-20-5, Sigma-Aldrich) – was used as a modifier. Replacing conventional modifiers (e.g., dicyclopentadiene) with a renewable, biodegradable raw material makes the process less harmful to the environment [10]. To preparation of SPC involved heating a specified amount of sulfur to 145–150 °C. Then, the tung oil was added to the molten sulfur under continuous stirring to form a homogeneous mixture. Five modifier doses were tested, constituting 2, 4, 6, and 8 wt % sulfur (wt % S). After the modifier addition, the mixing process was carried out for approximately 40 minutes. Next, fine aggregate and mineral waste were added to the SPC.

Mineral waste and aggregate. The filler materials in the prepared sulfur-mineral composites consisted of fine aggregate and mineral waste. Based on aggregate particle-size distribution analysis, the substitutive diameters (D_{10}, D_{30}, and D_{60}), the uniformity coefficient (C_U), and the coefficient of gradation (C_C) were calculated. The applied fine aggregate ($\varnothing < 2$ mm) was homogeneous, with a dominant sand fraction accounting for more than 98 wt % (Table 1, Fig. 1).

Table 1

Characteristics of fine aggregate

Grain size characteristics	Size or gradation metric	Value
Stone fraction	> 63 mm	–
Gravel fraction	2–63 mm	–
Sand fraction	0.063–2 mm	98.8%
Particulate fraction	0.002–0.063 mm	1.2%
Clay fraction	< 0.002 mm	
Alternate diameters	$D_{10}/D_{30}/D_{60}$	0.16/0.26/0.42 mm
Coefficient of gradation	C_C	1.01
Uniformity coefficient	C_U	2.63

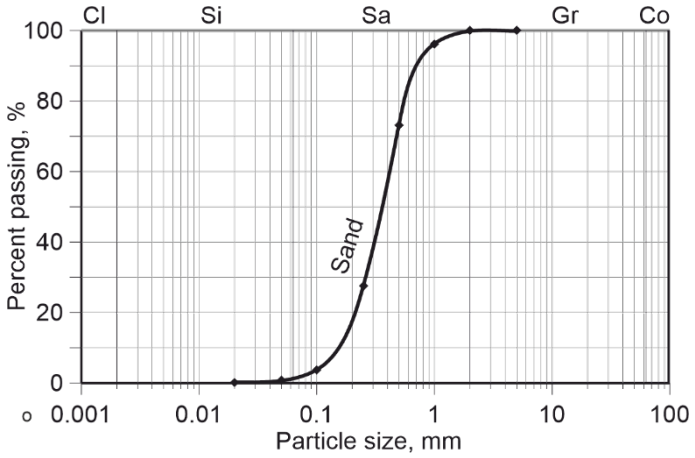

Fig. 1 Fine aggregate particle size distribution:
Cl – clay, Si – silt, Sa – sand, Gr – gravel, Co – cobble

The waste was characterized by 7% moisture and an organic matter content of 0.07% (as loss-on-ignition). Based on particle size distribution (sieves with 5, 2, 1, 0.5, 0.25, 0.1, and 0.05 mm meshes were used), the percentage content of fractions was determined. The results of the sieve test are presented in Table 2 and Fig. 2. The waste exhibited a high contribution rate of fractions > 2 mm, including fraction larger than 5 mm (> 40 wt %).

Table 2

Distribution of different size fractions in waste

Size, mm	> 5	2–5	1–2	0.5–1	0.25–5	0.1–0.25	0.05–0.1	0.02–0.05	< 0.02
Content, %	41.1	21.9	9.7	7.3	5.4	4.8	3.3	4.0	2.5

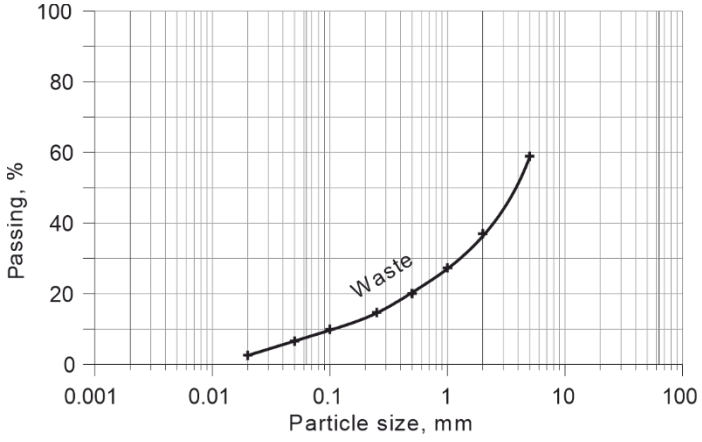

Fig. 2. Waste particle size distribution curve

Due to the variability in particle size distribution and friability, the waste was crushed. The final grain size of the processed waste was < 1 mm. The bulk density of the processed waste was approximately four times lower than that of the fine aggregate. Two filler material mixtures were used, with the following proportions: mix A: 6 wt % waste (W) and 94 wt % fine aggregate; mix B: 12 wt % W and 88 wt % fine aggregate. The waste accounted for 20% and 34% of the total filler material volume, respectively. The contents of Cu, Pb, Cr, Ni, and Cd in the waste were analyzed. The material was characterized by high concentrations of Cd, Pb, and Cr. The results are presented in Table 3.

Table 3

Metal concentrations in waste and leachates

Metal/pH	Dry waste	Leachate		
		TCLP test	EN test	EN test (dry weight)
Cu	10 500	17.4	0.95	8.87
Pb	11 010	1.23	0.01	0.11
Cr	226	0.14	0.1	0.96
Ni	869	6.95	not detected	not detected
Cd	25 061	437	2.67	25.0
pH	–	6.26	8.28	8.28

Preparation of SPC-waste composites. The sulfur composites were obtained by combining SPC with a different modifier content with mixture A or B. The filler material (waste and aggregate) was dried, homogenized, and preheated to 160 °C. Such prepared material was added to the molten SPC. The temperature was continuously monitored to maintain the mixture's proper workability during the addition of filler material. All components were mixed for approximately 20 minutes.

Table 4

Material composition of obtained samples [wt %]

Sample	Sulfur (S)	Tunog oil (TO)	Fine aggregate (Fa)	Waste (W)
S-TO(2)+A	47.8	1.0	48.1	3.1
S-TO(4)+A	47.4	1.9	47.7	3.0
S-TO(6)+A	46.9	2.8	47.2	3.0
S-TO(8)+A	46.5	3.7	46.8	3.0
S-TO(2)+B	46.5	0.9	46.3	6.3
S-TO(4)+B	46.1	1.8	45.8	6.2
S-TO(6)+B	45.7	2.7	45.4	6.2
S-TO(8)+B	45.2	3.6	45.0	6.1

The filler mixtures A and B accounted for 107 and 113 wt % of sulfur, respectively. For each mixture, four composite samples (cylindrical specimens with a diameter of 56 mm and a height of 50 mm) were produced, each with a different modifier dose (2, 4, 6, and 8 wt % of sulfur). The prepared composites were compacted and cooled at room temperature. The composition of prepared samples is presented in Table 4.

Water absorption by immersion. The durability of the produced composites was assessed, among other factors, by water absorption capacity. Mass water absorption is the ratio of the weight of water absorbed by a sample to the weight of the dry sample. The test was carried out through a gradual immersion of samples in water. In the first stage, the samples were submerged in water up to 1/4 of their height. After 2 hours, the bath was filled with water to 1/2 of the samples' height. The samples were kept in that state for another 3 hours. Subsequently, the water was filled to 3/4 of the samples' height, and they were left for a further 19 hours. Finally, water was added to a level about 20 mm above the upper surface of the samples. From that point onward, the samples were removed from the bath and weighed every 24 hours. The measurements were repeated until the difference between two consecutive weightings did not exceed 0.2 g.

Unconfined compressive strength. The mechanical properties of the produced composites determine their suitability for practical applications. Ensuring the structure stability is particularly important for composites with waste. Damage to the composite may lead to exposure to weathering and substantially increase heavy metal leaching. After 24 hours of curing, the composite samples were demolded and subjected to compressive strength testing. The tests were conducted on non-standard cylindrical specimens with a diameter of 56 mm and a height of 50 mm.

Leaching tests. Sulfur-polymer composites were tested for the leaching of heavy metals present in the waste used as one of the filler components. The samples were subjected to two tests following EN 12457:2002 and the TCLP (Toxicity Characteristic Leaching Procedure) [14, 15]. For this purpose, the composites were crushed to a particle size of <10 mm. Then, the samples were extracted for 24 hours with distilled water (EN test) and for 18 hours with acetic acid solution at pH 2.88 (TCLP test). The liquid-to-solid phase ratio was 10 dm^3/kg for the EN test and 20 dm^3/kg for the TCLP test. The leachates were filtered and analyzed for heavy metal concentrations.

3. RESULTS AND DISCUSSION

3.1. PHYSICAL AND MECHANICAL PROPERTIES OF COMPOSITES

The density difference between waste and other components (aggregate and SPC) caused problems with uniformly covering the surface of the A and B mixture particles

by the SPC. During the mixing, the uneven distribution of mineral components was observed. As a result, the prepared composites were characterized by a non-uniform structure (Fig. 3). The poor workability of the mixture also affected their incomplete degassing, which resulted in the formation of voids within the composites. The problem with the samples' degassing was also observed in research with galvanic waste [10]. Additionally, in composites where large doses of tung oil were used, its accumulation on the surface of the mixture was observed. An excess dosage of natural modifiers could reduce the adhesion between the filler materials and SPC, which may reduce both the liquid resistance and compressive strength.

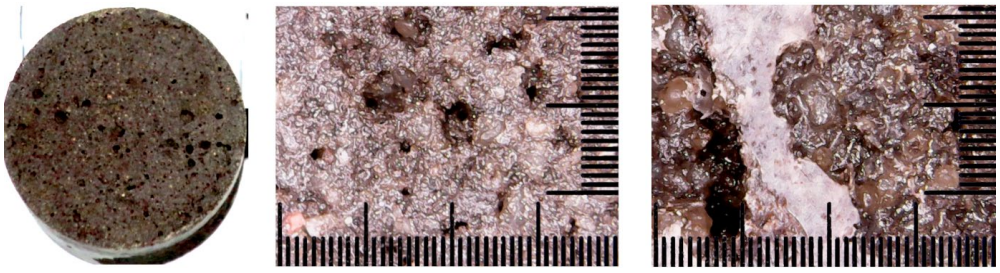

Fig. 3. Surface structure of the S-TO(6)+A sample

The water absorption test was carried out to assess the durability of the produced composites. This is an important parameter, especially for materials that may be exposed to chemical attacks or freeze-thaw cycles. The test was conducted for 5 days. The water absorption level of composites based on mixture A ranged from 0.4 to 0.9 wt % (Table 5). Composites based on mixture B exhibited higher water absorption capacities. The weight of water absorbed ranged from 0.5 to 1.5 wt %. The increase was due to the higher waste content and its hydrophilic properties. Additionally, the hydrophobicity of SPC was reduced due to structural defects in the composites. For instance, the sample S-TO(6)+A, presented in Fig. 3, exhibited a non-uniform structure. The visible voids were related to incomplete mixture degassing and were the main sites of water entry. From the samples based on mixture A, the highest value of absorbed water (0.86 wt %) was recorded for sample S-TO(6)+A.

Samples based on mixture B exhibited a higher number of surface defects. Te highest water absorption (1.51 wt %) was observed for sample S-TO(8)+B. For comparison, the sample based on mixture A with the same modifier dose (S-TO(8)+A) showed more than twice the lower value, 0.61 wt %. Compression strength tests showed that composites based on mixture B for all analyzed modifier dosages exhibited higher strength than those based on mixture A (Table 5). Despite the uneven distribution of filler materials, composites with mixture B were characterized by more efficient sample volume filling. The greatest differences were observed for samples with a 2 wt % S of modifier dose. The

sample based on mixture B achieved more than 2.8 times higher compressive strength than the corresponding sample with mixture A.

Table 5

Physical and mechanical properties of composites

Modifier dose, % of the sulfur mass	Water absorption capacity [%]		Compressive strength [MPa]	
	Composite based on			
	Mix A	Mix B	Mix A	Mix B
2	0.51	0.45	3.98	11.15
4	0.42	0.82	7.97	11.55
6	0.86	1.26	7.17	9.16
8	0.61	1.51	7.17	9.56

The impact of the modifier dose was also observed. The highest compressive strength values were obtained for composites with 4 wt % S of tung oil, reaching 7.97 MPa for sample S-TO(4)+A and 11.55 MPa for sample S-TO(4)+B. Further increase in the modifier dose resulted in decreased workability and compressive strength of samples, regardless of the applied filler mixture. This deteriorating effect was related to increased viscosity and incomplete degassing of samples. As a result, composites were characterized by an uneven distribution of filler materials. For example, the compressive strength of composites based on mixture A and SPC with 6 wt % S of tung oil (S-TO(6)+A) was approximately 10% lower when compared to the sample based on SPC containing 4 wt % of modifier (S-TO(4)+A). In the case of composites with mixture B, the strength decrease was 21% for S-TO(6)+B and 17% for S-TO(8)+B in comparison to sample S-TO(4)+B. The results showed that material based on SPC modified with 4 wt % S of tung oil and mixture B exhibit application potential, e.g., in road construction.

3.2. CHEMICAL PROPERTIES OF THE COMPOSITES

The waste used as a filler contained heavy metals such as Cu, Pb, Cd, Cr, and Ni. Particularly high concentrations of Cd, Pb, and Cu were found (Table 3). The TCLP test [14] results showed an exceeded limit of cadmium (1 mg/dm^3) defined in federal regulations (40 CFR 261.24). The concentrations of Cr and Pb were below the regulatory limit (5 mg/dm^3). For the remaining analyzed metals (Cu and Ni), the federal regulations do not establish concentration limits that determine waste as hazardous.

In the case of the EN [15] test, the possibility of raw waste disposal in landfills was verified. The concentration of Cd in leachate exceeded the limit (5 mg/kg dry wt) defined for waste permitted for disposal in hazardous waste landfills [16]. Due to the impossibility of landfilling the waste, it was decided to use it as fillers in SPC composites.

The risk of heavy metal release from the produced composites was also assessed based on the metal concentrations in the leachates from the EN and TCLP tests. Due to the uneven distribution of filler components, the degree of reduction of the grain size of the composites was increased and the samples' homogeneity was improved.

pH of EN test leachates ranged from 7.91 to 8.54 for composites based on filler mixture A and 8.11 to 8.38 for composites based on mixture B. pH value affects Cr leaching, which exhibits an amphoteric nature. The highest detected concentrations of Cr (0.21 mg/dm^3), Pb (0.03 mg/dm^3), and Cu (0.04 mg/dm^3) were below the limit values applied for waste acceptable at landfills for non-hazardous waste (Table 6). All composites fulfilled the EU permissible limits for Cr (1 mg/dm^3), Pb (1 mg/dm^3), Cu (5 mg/dm^3), and Ni (1 mg/dm^3) [16]. In the case of Cd, the limit (0.1 mg/dm^3) was met by all composites based on mixture A and by one sample based on mixture B (S-TO(8)+B). The results for composites with mixture A showed a beneficial effect of increasing the tung oil dosage from 2 to 4 wt % S. For the analyzed metals, their concentrations in the EN leachates were lower.

Table 6

Heavy metal concentrations in the EN test leachates [mg/dm^3]

Sample	pH	Metal				
		Cd	Cu	Pb	Cr	Ni
S-TO(2)+A	8.36	0.12	0.02	0.03	0.21	not detected
S-TO(4)+A	8.54	0.01	0.01	0.01	0.14	
S-TO(6)+A	7.91	0.01	0.01	0.01	ND	
S-TO(8)+A	8.20	0.01	0.01	0.01	0.02	
S-TO(2)+B	8.38	0.11	0.02	ND	0.08	
S-TO(4)+B	8.34	0.19	0.03	0.02	0.14	
S-TO(6)+B	8.11	0.26	0.04	0.02	0.08	
S-TO(8)+B	8.24	0.08	0.01	0.01	0.13	

pH of the TCLP test leachates ranged from 3.76 to 4.15 for composites based on filler mixture A and from 3.87 to 4.13 for those based on mixture B (Table 7). The acidic nature of the leaching solution affected the mobility of the analyzed heavy metals. For all composites, higher concentrations of metals were observed in the TCLP leachates compared to those from the EN test. Moreover, the Cd concentration exceeded the permissible 1 mg/dm^3 limit. In the case of composites with mixture B, its concentration was very high, above 38 mg/dm^3. The main factors affecting Cd mobility were its high concentration in the raw waste and SPC's uneven coating of waste particles. The raw waste particles were released during the sample size reduction for leaching tests. This allowed their direct contact with the leaching solution. In the case of Cr and Pb, their concentrations were below the 5 mg/dm^3 limit value. Nevertheless, the greater mobility of Cr and Pb was interpreted by the amphoteric properties of their compounds. Other analyzed metals (Cu and Ni) are not regulated by the federal Resource Conservation

and Recovery Act (40 CFR 261.24). The TCLP test confirmed that any structural defects may lead to the uncontrolled leaching of metal ions into the environment.

Table 7

Heavy metal concentrations in the TCLP test leachates [mg/dm^3]

Sample	pH	Metal				
		Cd	Cu	Pb	Cr	Ni
S-TO(2)+A	4.15	32.35	0.17	0.04	0.24	0.95
S-TO(4)+A	3.96	4.75	0.03	1.22	0.26	0.84
S-TO(6)+A	3.76	1.92	0.03	0.56	0.22	0.56
S-TO(8)+A	3.87	3.73	0.03	1.08	0.28	0.69
S-TO(2)+B	4.13	38.72	0.23	0.12	0.21	2.09
S-TO(4)+B	4.12	48.88	0.68	0.39	0.22	1.95
S-TO(6)+B	4.01	45.70	2.74	1.64	0.21	1.59
S-TO(8)+B	4.06	38.48	0.62	0.96	0.23	1.59

4. SUMMARY

This study evaluated the possibility of producing alternative materials based on hazardous waste and sulfur binders modified with natural tung oil. The process efficiency was assessed by analyzing water absorption by immersion, compressive strength, and heavy metal concentrations in the leachates from the EN 12457:2002 and TCLP tests. The results showed problems in homogenizing SPC with filler components. Analysis of the sample structure confirmed the unevenly coated waste particles. Moreover, samples based on SPC modified with greater doses of tung oil exhibited higher surface defects. This phenomenon adversely affected both the composites' water absorption and compressive strength.

Higher water absorption was observed in composites with a larger waste content (based on mixture B). Nevertheless, a more efficient distribution of filler components in the whole volume of the prepared composites had a beneficial influence on their strength. All samples based on mixture B were characterized by higher compressive strength.

Leaching tests showed greater mobility of heavy metals in an acidic environment (approximately pH 4). The indicated structural defects may significantly influence the degree of their leaching. The tests did not reveal a significant effect of the modifier dosage on the efficiency of heavy metal immobilization.

The results confirm that sulfur composites may represent a promising alternative to conventional building materials. Their advantages include limited consumption of primary raw materials and reduced CO_2 emissions during production. Nevertheless, further studies are needed to improve the structural integrity of composites to limit the mobility of heavy metals in acidic environments.

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