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An investigation on recycling potential of sulfur concrete

Muhammad Ahsan Gulzar [a,*], Abdur Rahim [a], Babar Ali [b], Ammad Hassan Khan [a]

[a] Department of Transportation Engineering and Management University of Engineering & Technology, Lahore, 54000, Pakistan
[b] Department of Civil Engineering, COMSATS University Islamabad, Sahiwal Campus, Sahiwal, 57000, Pakistan

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ABSTRACT

The rising concerns about the environment and sustainability are leading towards the need of recyclable materials to protect the environment and help in resource conservation. Sulfur concrete (SC) is a unique composite that does not utilize water and energy intensively produced cement. As a substitute to cement, molten sulfur firmly binds aggregates upon hardening. The sulfur concrete is recyclable and can be easily remolded in new applications. Moreover, sulfur is a by-product of the petroleum industry with almost zero carbon footprint. In this study, the mechanical (compressive strength and modulus of rupture) and durability properties (water absorption, salt attack-resistance, acid attack-resistance and alkali attack-resistance) of SC were evaluated after recasting without the addition of a new binder. The properties of fresh and recast SC were compared with those of the conventional Portland cement concrete (PCC) and sulfate resisting cement concrete (SRC). The results reveal that both the mechanical and durability properties of SC have significantly improved after the first recycling/recasting. However, there is a drastic decrease in strength and durability performance after the second recasting. Durability and mechanical performance of fresh and first recast SC is noticeably higher compared to PCC and SRC.

1. Introduction

The development of stringent environmental protection laws has increased the need to implement advanced methods for the consumption of sulfur from petroleum products. At the same time, the increasing demand for transportation and environmental regulations, consequently, has led to the production of large sulfur along with natural oil and gas [1]. According to the US Geological Survey [2], the worldwide production of sulfur is about 70 million metric ton. The chemical, physical and mechanical properties of sulfur offer potential for a wide range of its applications i.e. vulcanization of rubber, detergents, sulfuric acid, fertilizers, and construction. There is an increased focus on finding new applications to consume a large amount of sulfur waste in the construction industry.

The issues of global warming and sustainability are major concerns that have endangered the existence of life on earth. The main reason is the growth of industrial manufacturing and processing and one of such processes include cement concrete manufacturing. However, conventional cement concrete is not an environment friendly construction material [3]. It consumes large amounts of natural resources to meet its requirement of cement, coarse and fine aggregates. The most (about 82%) of the global warming emissions of concrete production comes from its main constituent cement [4]. Cement manufacturing burns high amounts of fuels to achieve the elevated temperature for the clinker production, also the calcination of limestone releases 40% of emissions of cement production. Cement industry also affects the nearby flora and fauna [5]. Dust from the cement industry can stunt the growth and productivity of plants and cash crops in the neighboring areas [6]. On the other hand, concrete wastes (both from construction and demolition practices) create social, environmental and waste management issues [7,8]. To save the environment and life, there is a grim need for sustainable, recyclable and eco-friendly composites to meet the demand for concrete in the construction industry.

SC is a unique construction material that can be used as a replacement of Portland cement concrete (PCC) in different applications such as road pavements, wastewater treatment plants, sewer and drainage pipes, hydraulic structures and retaining walls [9]. Recyclability, high chemical resistance, quick hardening are some of the special properties of SC that add to the uniqueness of this material. Also, SC produces very low emissions as compared to Portland cement [10]. The only heat involved in the manufacturing process of SC is of the mixing process (up to 120 °C), which is quite lower than the heat (up to 1450 °C) required for the clinker production in cement manufacturing.

* Corresponding author.
E-mail addresses: ahsan_gulzar975@yahoo.com (M.A. Gulzar), rahim@uet.edu.pk (A. Rahim), babar.ali@scetwah.edu.pk (B. Ali), chair-tem@uet.edu.pk (A.H. Khan).

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Table 1
Chemical composition of conventional cements.

Binder	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI (%)
Portland cement	21	5.04	3.24	61.7	2.56	1.56	1.83
Sulfate resisting cement	25	3.95	4.96	51	2.11	1.93	0.95

Table 2
Physical properties of conventional cements.

Binder	Specific Gravity	Specific Surface (cm^2/kg)	Initial setting time (min)	Final setting time (min)	Compressive Strength (MPa)
Portland cement	3.11	3719	101	609	41
Sulfate resisting cement	3.21	3142	80	240	38.2

SC is a thermoplastic concrete composed of filler, aggregates and sulfur mixed at 120 °C or higher temperature to form a homogenous mixture. The absence of water and hydration reaction contributes to the good durability properties of sulfur concrete. In sulfur concrete, elemental sulfur when heated at mixing temperatures (at 120 °C), changes to monoclinic crystallized sulfur (S_α). The temperature below 115 °C, S_α changes to more stable crystallized form known as orthorhombic sulfur (S_β). This limit the use of sulfur concrete in areas where the service temperatures are well below the melting point of sulfur [11]. SC achieves 90% of its mechanical strength in 24 h after casting, this can reduce the time for dry curing and expedite the construction process. Moreover, SC shows excellent resistance against saline and corrosive environments [12]. The further advantages of sulfur concrete over conventional concrete include [13]; a) improved mechanical strength and durability such as low water absorption, high resistance against acid and salt attacks, b) economic benefits of rapid setting, c) eco-friendliness and low carbon footprint due to absence of calcination and clinker temperatures (850–1400 °C), elimination of water, and recyclability. Additionally, these properties make SC a preferred choice for extraterrestrial construction with a low possibility of water such as Mars [13] and Moon [14]. SC can be re-casted after crushing the used SC into grains and then re-melting it [15]. Moreover, SC recycling does not require an additional binder and supplementary aids like conventional cement concrete.

Literature survey of SC indicates that it had not been fully investigated for its recycling potential. A study by Joshua [16] reported that the compressive strength of recast SC was 216–230% greater than fresh cast SC. Another study conducted by Wan et al. [13] investigated the performance of fresh and recast SC with Martian soil simulant. Their research indicates that the compressive strength of recast SC is slightly better than fresh cast SC. The understanding of the behavior of recast SC is crucial from the redesign or reclamation viewpoint. To the best knowledge of the authors, a lack of understanding exists in the available information/literature about the strength of recycled/recast SC [17] and no information is available on the durability properties of recast SC.

Therefore, the main objective of this study is to evaluate the properties of SC after two cycles of recasting. The mechanical performance of recast SC was evaluated based on the results of compression and flexural testing. Durability properties of recast SC were also investigated such as resistance against salt (NaCl), alkali (NaOH), and acid solution (H_2SO_4). Properties of fresh and recast SCs were also compared with those of the conventional cement concretes i.e., Portland cement concrete (PCC) and sulfate-resisting cement concrete (SRC).

2. MATERIALS and methods

2.1. Materials

The sulfur binder used in this research was 99% pure sulfur with 1% mineral impurities i.e. arsenic, sulfur acid, etc. The density and melting point of the binder was 2100 kg/m^3 and 120 °C, respectively. A general-purpose Portland cement of 53 grade (Type I as per ASTM C150 [18]), was used to manufacture Portland cement concrete (PCC). Additionally, sulfate resisting cement, Type II as per ASTM C150 [18], was used to produce sulfate resisting concrete (SRC). The properties of Portland cement and sulfate resistant cement are presented in Table 1 and Table 2, respectively.

The aggregate (locally known as Sargodha crush) passing through 4.75 mm and retaining on 0.075 mm sieve were used. The aggregate remained well within the upper and lower limits of ASTM C33 [19] pre-

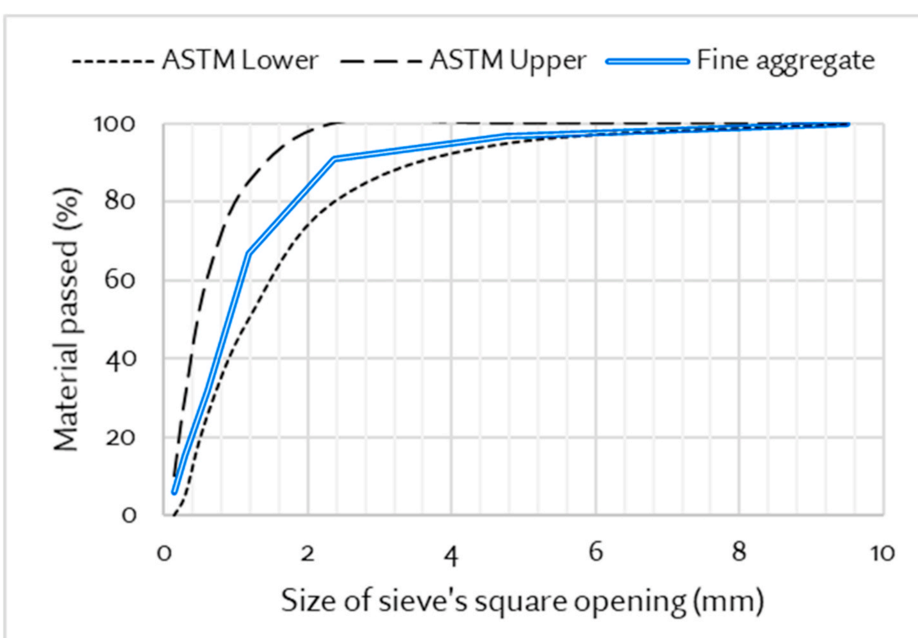


Fig. 1. Gradation of aggregate.

Table 3
Nomenclature, composition and fresh density of concrete mixtures.

Nomenclature	Type of binder	Binder (kg/m^3)	Water (kg/m^3)	Aggregate (kg/m^3)	Fresh density (kg/m^3)
PCC	Portland cement	450	200	1150	1831
SRC	Sulfate resisting cement	450	200	1150	1827
Fresh SC-25	Sulfur binder (25%)	450		1350	1837
Fresh SC-30	Sulfur binder (30%)	540		1260	1818
Fresh SC-35	Sulfur binder (35%)	630		1170	1795

ferred for concrete aggregate. The density of aggregate was about 1515 kg/m^3 and water absorption less than 1%. Gradation of aggregate is shown in Fig. 1. Mineralogical composition of this aggregate is dolomite-sandstone.

2.2. Composition of concrete mixtures

A total of five different mixes were investigated, see Table 3. First and second mix have Portland cement and sulfate resisting cement as binders, respectively. Portland cement concrete (PCC) and sulfate resisting cement (SRC) were produced using 1 part of cement and 2.5 parts of crushed stone-aggregate by weight. Water to cement ratio in conventional concretes is kept as 0.45. Properties of sulfur concrete (SC) were compared with these conventional mixes. Three types of SC were produced using different contents of sulfur i.e., 25%, 30%, and 35% by weight of concrete. SC-25 contained the same binder quantity as those of the conventional concretes. SC-30 and SC-35 had higher binder quantity compared to conventional concretes. Properties of SC-25, PCC and SRC were compared to understand the behavior sulfur binder compared to conventional binders. Whereas SC-30 and SC-35 helps to understand the effect of sulfur content on the properties of fresh SC and recast SC. The details and designations of all mixtures are provided in Table 3. The fresh density value of all mixes is also provided in Table 3. It is noticed that fresh density of all mixes is variable in the range of 1795–1837 kg/m^3. There is a no huge difference between the density of SC-25 and conventional mixes i.e., PCC and SRC. This is because the composite specific gravity of cement (3.11), water (1.00) and aggregate (2.67) in conventional concrete is about 2.58. Whereas the composite specific gravity of sulfur binder (2.2) and aggregate (2.67) in SC-25 is about 2.54. That is why for equivalent binder content, density of SC-25, PCC and SRC does not vary significantly. Moreover, density of SC decreases with the rise in sulfur content, because the increase in the sulfur content decreases the composite specific gravity of SC.

2.3. Mixing, casting, and recycling

The fresh specimens of PCC and SRC were prepared manually by mixing dry aggregates and cement for 4 min. Then a required amount of water was added, and concrete was further mixed for 6 min. After thorough mixing, specimens were cast in steel molds at normal temperature.

In the case of SC, firstly, aggregates were heated at the temperature of 120 °C for not less than 3 h in a convection oven. Then molten sulfur (heated at 120 °C) in a separate container was thoroughly mixed with aggregates manually. The concrete mixture was then added in cast-iron molds (pre-heated 120 °C for 1 h). The compaction was done with a manual tamping rod.

In the recycling of SC, four replicate batches (batch I, II, III and IV) of freshly cast SC were prepared as shown in Fig. 2. The batch-I used in the testing the properties of fresh cast SC. After 48-h, Batch-II, III and IV of fresh cast SC were crushed to the passing size 4.75 mm. The batch II, III and IV were heated at 130 °C for 10 min and thoroughly mixed and re-cast in preheated molds. One of the three batches of 1st recast SC was used in the mechanical and durability testing to evaluate the influence of 1st recycling/recasting on the properties of SC. The remaining two batches of 1st recast SC were used to produce and test one complete 2nd recast-Batch with some losses due to recycling. The composition of fresh, 1st and 2nd recast SC at all binder contents was not varied unless there is some loss of binder content in heating.

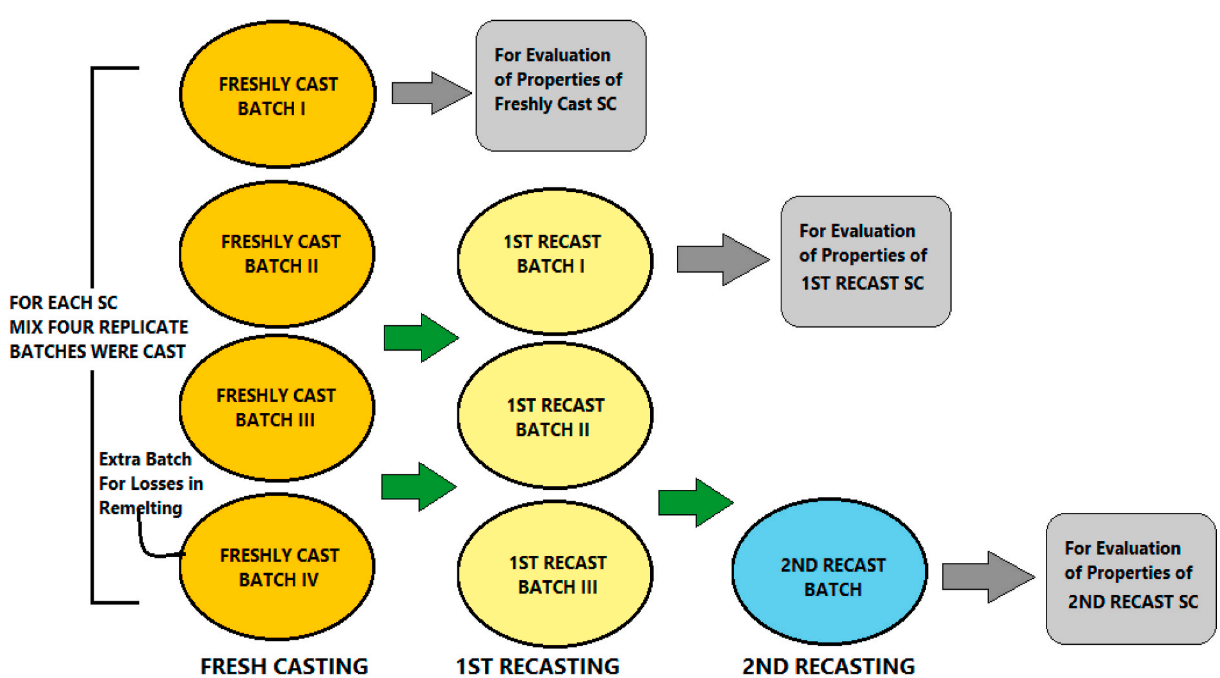
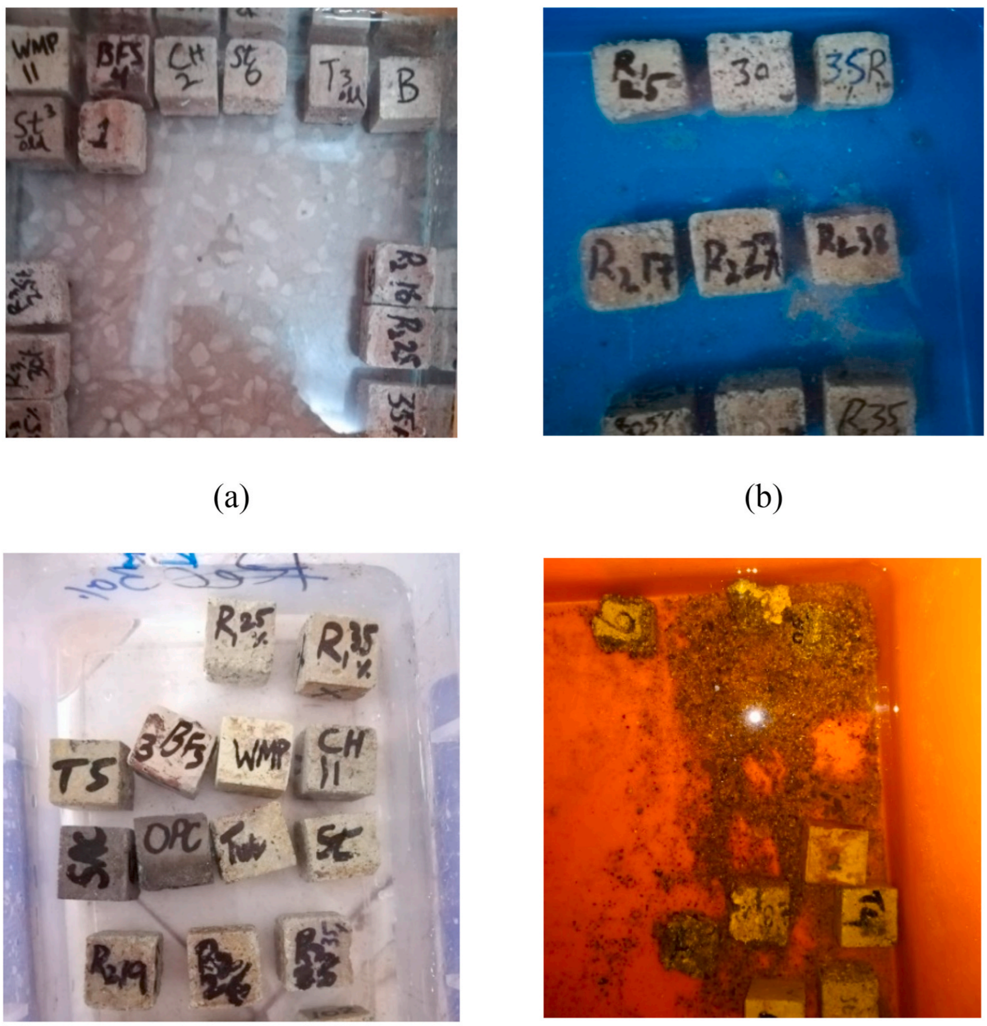


Fig. 2. Recasting schedule of SC.

(a) (b)

Fig. 3. Mechanical tests a) compression and b) flexural.



(a) (b) (c) (d)

Fig. 4. Conditioning a) water b) salt (15% NaCl) c) acid (5% H_2SO_4) and d) base (5% NaOH).

3. Methods

For mechanical testing of SC and conventional binders, ASTM standards developed for cement mortars were employed. 25.4 mm cubical specimens of each mix were tested to determine the compressive strength according to ASTM C109 [20] as shown in Fig. 3. The PCC and SRC specimens were tested after 28-days of curing in tap water. Whereas, SC specimens were tested after 48 h of casting. For the evaluation of flexural strength of mixes (Fig. 3(b)), 25.4 mm × 25.4 mm x 102 mm prisms were cast and tested according to ASTM C348 [21].

The water absorption test was conducted on 25.4 mm specimens by noting the difference in dry and saturated weight of the specimen [22]. The SC specimens were, then, soaked in water for 24 h to measure the saturated surface dry weight of the specimen. In case of both PCC and SRC, dry weight of specimens was measured after oven drying at 80 °C for 2-days. After determining the dry weights, PCC and SRC specimens

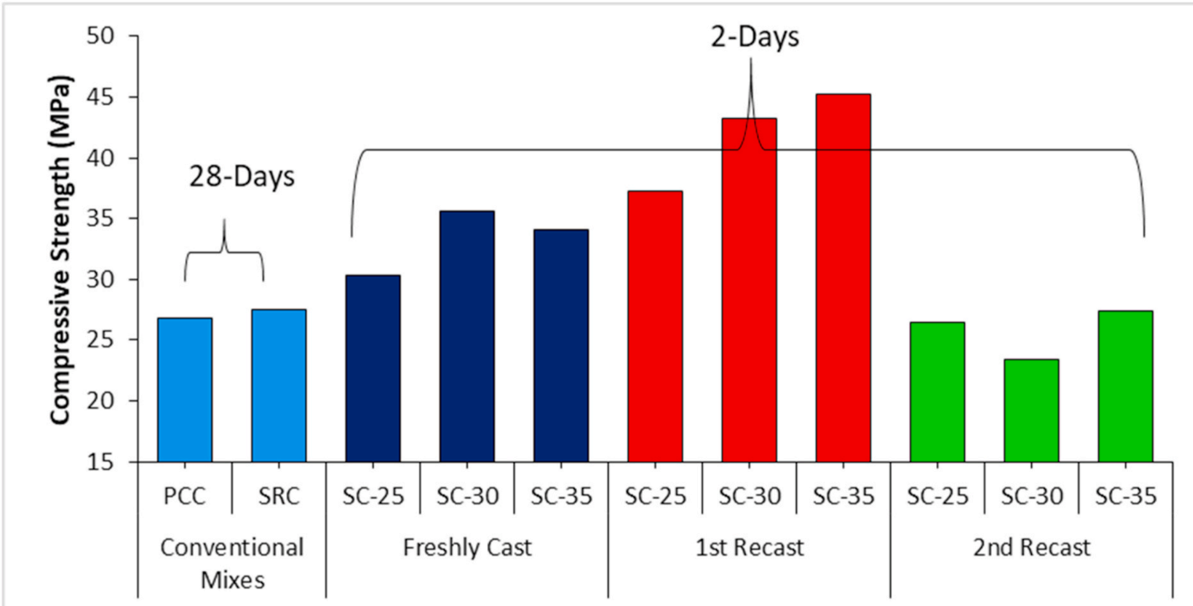


Fig. 5. Compressive strength of each concrete mix.

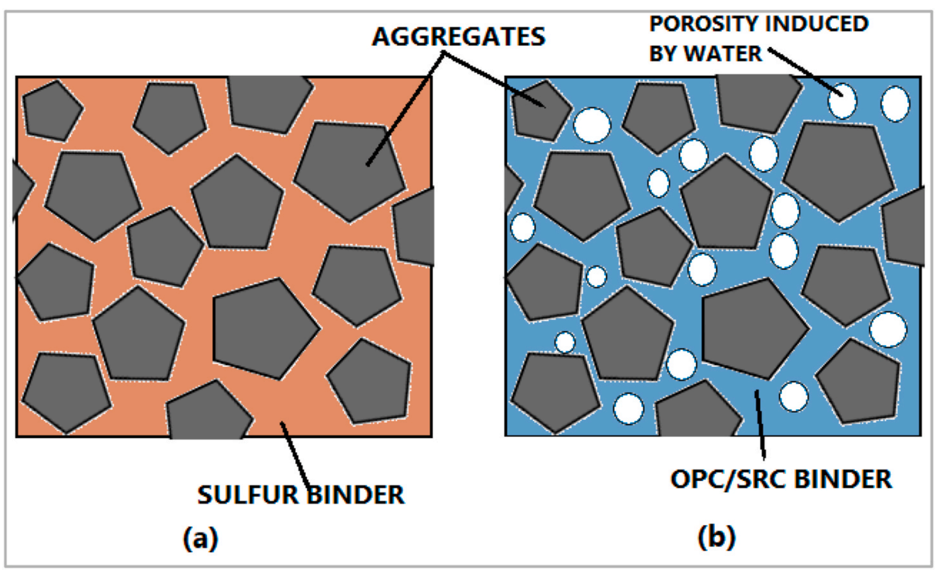


Fig. 6. Binder matrices of sulfur concrete SC and both PCC and SRC with equivalent aggregate concrete.

were air-cooled for one day. Subsequently, these specimens were dipped in the water for 24 h to determine the saturated surface dry weight. Water absorption of each specimen was calculated using $WA\ (\%) = \frac{W_S - W_D}{W_D} \times 100$, where, WA is water absorption; W_S indicates saturated surface dry weight (grams) and W_D is oven-dry weight of specimen (grams).

To assess the durability of each mix in aggressive environments, the resistance of each mixture was observed in three different chemical solutions i.e. 15% sodium chloride, 5% sulfuric acid and 5% sodium hydroxide. The resistance of each mixture was measured for loss in mass due to the action of chemicals as shown in Fig. 4. The loss in mass was recorded by noticing the difference between mass of specimen before and after the exposure in chemical environment.

4. RESULTS and discussion

4.1. Compressive strength

The results show that the compressive strength of freshly cast SC is higher than conventional concrete mixes i.e., PCC and SRC (Fig. 5). This behavior is due to the low porosity of SC in the absence of water. The PCC and SRC mixes contain a high quantity of water for hydration of cement, this cause generation of voids leading to the low mechanical strength as shown in Fig. 6. This strength difference is also due to high binder content per unit volume of SC mixes as compared to PCC and SRC. Also, the voids of SC are small and discontinuous, whereas, in case of PCC and SRC, large interconnected voids act as sites for stress relief at lower loads and cause mechanical failure of material [11]. Additionally, there is a slight difference (about 3%) between the compressive strength of PCC and that of the SRC (Fig. 5). The findings of this research are in line with the existing literature on fresh cast SC. Vlahovic et al. [23] compared the strength of fresh cast SC and PCC having aggre-

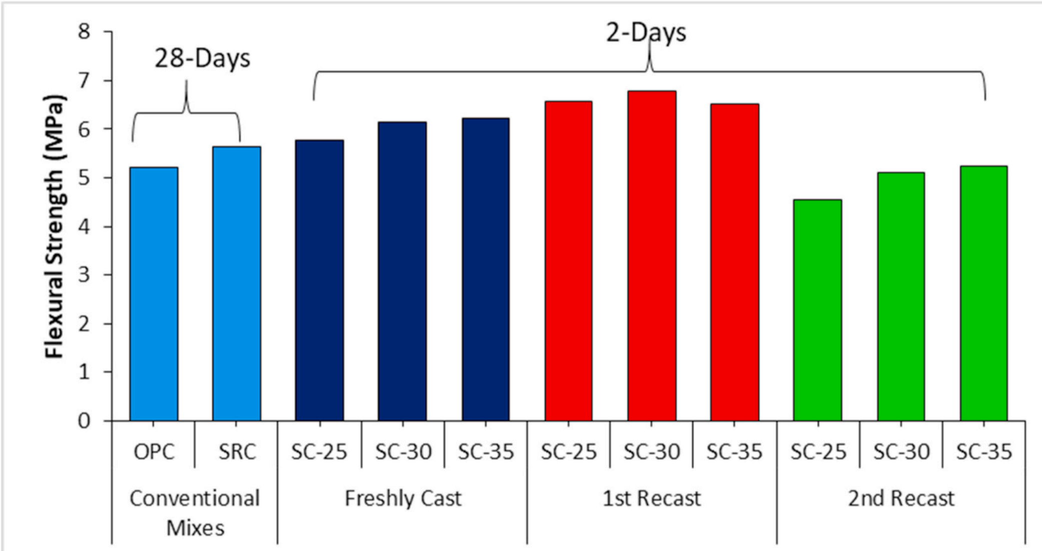


Fig. 7. Flexural strength of each concrete mix.

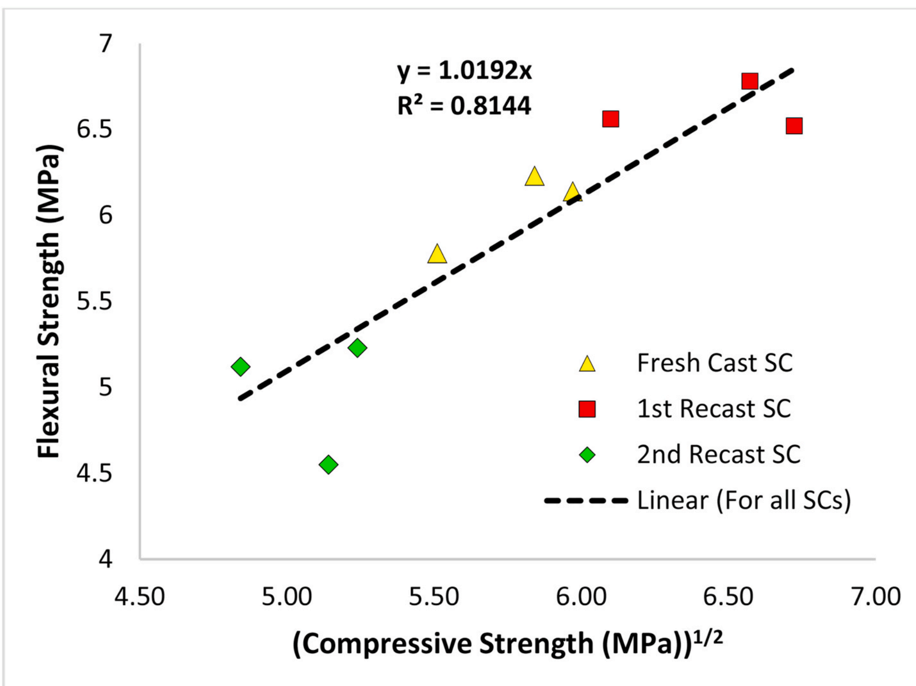


Fig. 8. Correlation between compressive and flexural strength of SC.

gate to binder ratios similar to those used in the present study. They reported fresh cast SC-30 had compressive strength 9% higher than PCC. Toutanji et al. [24] showed that SC-35 showed 29% higher compressive strength than PCC. Whereas, in the present research SC-30 and SC-35 respectively shows 37% and 33% higher compressive strength than the PCC.

The 1st recasting of SC shows significant improvement in the compressive strength over freshly cast SC. There is an improvement of about 30% in the compressive strength of SC after 1st recasting. The improvement in strength behavior of SC after recasting has been attributed [13] to the compact bonds of sulfur after recasting. Improved mixing, and application of external pressure to recast specimens helps in achieving high strength. Making a compact mix, produces a compact sulfur bond that reduces the number and size of pores in recast product. Karunaratne et al. [25,26] experimentally showed that sulfur-based polymers possess a good thermal-healability and modified sulfur polymers can be recycled several times with minor losses in mechanical strength. According to Jacobs [17] the strength improvement after first recasting can be credited to the loss in moisture from SC after remelting. This behavior needs further investigation for the better understanding of mechanical properties of recycled SC and establishment of evidence based on scanning electron microscopic results.

The compressive strength of 2nd recast SC mixes is significantly lower than that of the 1st recast and freshly cast SC mixes. The significant loss of binder content due to alternative heating might have lowered the strength of SC after 2nd recasting. The 1st recast-SC has given better results than fresh cast SC and 2nd recast-SC. This might be because fresh SC has a high binder content and 2nd recast-SC has lower binder content than the optimum binder content. At optimum binder content better aggregate-binder-aggregate contacts can efficiently dis-

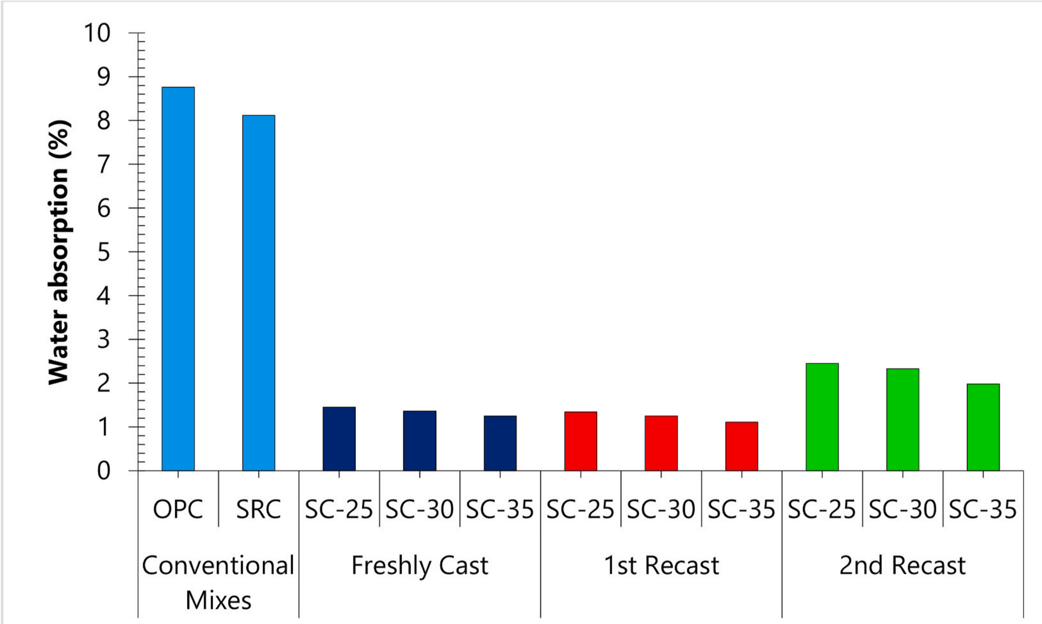


Fig. 9. Water absorption capacity of each mixture.

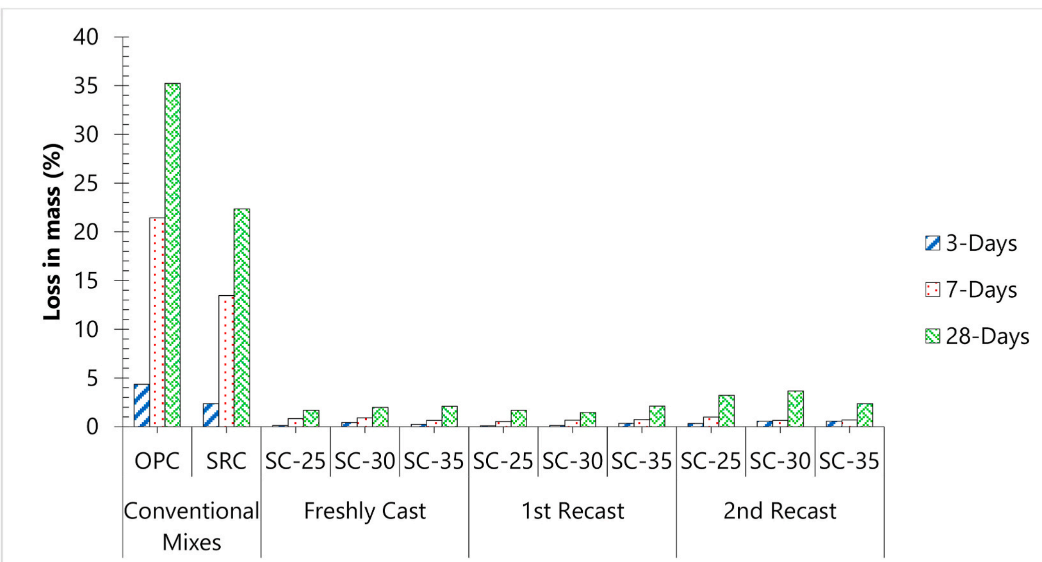


Fig. 10. Mass loss in sulfuric acid solution (5% H_2SO_4).

tribute stresses in the material. After 2nd recasting, the loss of binder (due to partial oxidization) may decrease the aggregate-binder-aggregate interface due to an increase in connectivity of pores that subsequently reduces the strength. Furthermore, alternative crushing of hardened SC to manufacture recast-SC may introduce a large number of fractures in aggregates and additional ITZs into recast-SC. The highest compressive strength after 2nd recasting was noticed in the mix with 35% sulfur content. This can be attributed to the higher residual binder content of SC-35 compared to that left in SC-30 and SC-25 after 2nd recasting.

4.2. Flexural strength

The flexural strength results, in Fig. 7, also show a similar trend as that of compressive strength. Freshly cast and 1st recast SCs shows very high flexural strength than conventional mixes. 1st recasting of SC improved the flexural strength by 5–10%. Overview of both strength results indicate that 1st recasting of SC has significantly enhanced the strength of SC. Whereas, the 2nd recast SC shows strength properties comparable to that of the conventional concrete mixes. These test results showed the positive influence of SC recycling on mechanical properties. However, there is a further need to evaluate the durability of recast sulfur concrete.

Using the half-power model, the correlation between compressive and flexural strength is evaluated for all SCs excluding conventional mixes (Fig. 8). The correlation between compressive and flexural strength is constructed for both fresh and recast SC. Coefficient of determination (R^2) for this relationship is 0.81. This shows that the flexural strength of SC can be predicted from its compressive strength with good accuracy for both fresh and recast SCs. Fig. 7 shows the relationship between compressive strength and flexural strength of SC is $f_r = 1.0192\sqrt{f_C}$, where f_r is flexural strength (MPa) and f_c indicates compressive strength in MPa.

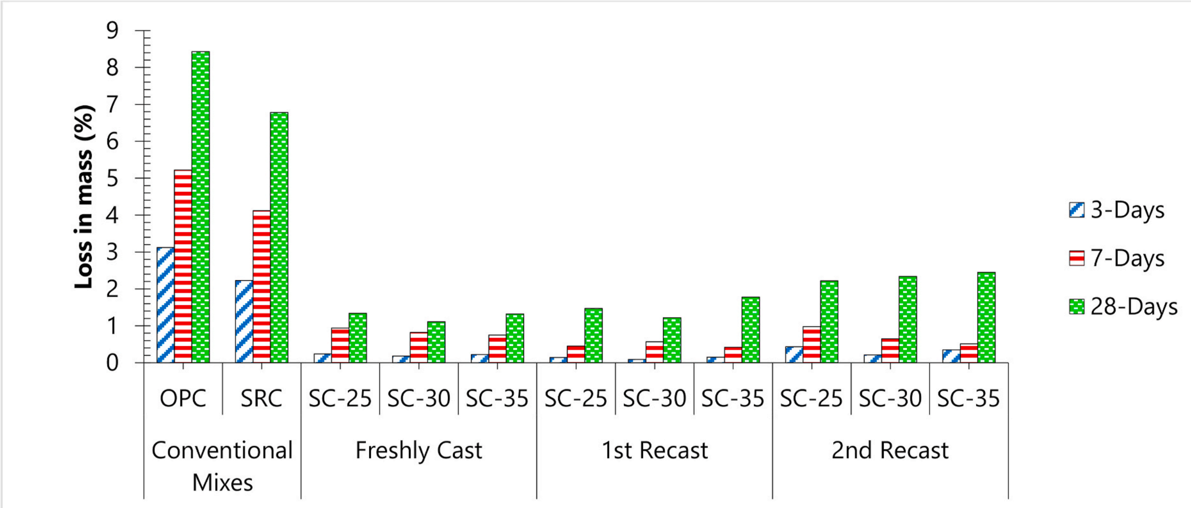


Fig. 11. Mas loss of each mix in a salt solution (15% NaCl solution).

Table 4
Mass loss in alkaline conditioning.

Type of mix	Type of binder	Loss in mass (%)		
		3-Days	7-Days	28-Days
Conventional Mixes	OPC	3.12	5.22	8.43
	SRC	2.23	4.12	6.78
Freshly Cast	SC-25	50	100	100
	SC-30	37	100	100
	SC-35	46	100	100
1st Recast	SC-25	31	100	100
	SC-30	66	100	100
	SC-35	54	100	100
2nd Recast	SC-25	55	100	100
	SC-30	21	100	100
	SC-35	36	100	100

4.3. Water absorption and porosity

The freshly cast SC mixtures have 6 times lower absorption than conventional mixes (PCC and SRC) as shown in Fig. 9. The low porosity of SC is due to the absence of need of hydration-water. Also, the sulfur binder is repellent to water. Compared to SRC and PCC, SC has lower connectivity of pores and micro-channels that promote water absorption. SCs have water absorption capacities of about 1% compared to 9% absorption capacity of PCC. Mohammed and Poornima [27] showed that SC had less than 1% water absorption compared to 7% absorption capacity of PCC. After 1st recasting, there is a further reduction in absorption of SC at all binder contents. The water absorption of SC increases after 2nd recasting; however, the absorption capacity of the recycled SC is significantly lower than that of the conventional mixes. The increased interface between the aggregate after the 1st-recast reduces the volume of voids leading to a slight reduction in water absorption compared to freshly cast SC. This affects the compressive strength of mixes. Moreover, the loss of binder content after the 2nd recast can increase the connectivity between pores, thus, providing easy access of water in the matrix of concrete.

4.4. Durability in chemical (acidic, basic and saline) environments

The products of cement hydration are highly alkaline making them vulnerable to the acidic environment. The free calcium hydroxide aggressively reacts with acids, in a neutralization reaction, resulting in the formation of the salts. For example, $Ca(OH)_2$ reacts with H_2SO_4 and produces gypsum $CaSO_4 \cdot 2H_2O$ as a salt which causes deterioration of material by building up internal pressure. The resistance of a material to acid also depends upon the porosity of the material. High porosity allows faster penetration of harmful chemical into the material.

The resistance of mixes against acid is measured in terms of mass loss in 5% sulfuric acid solution. Fig. 10 shows drastic degradation of PCC and SRC due to decalcification ($Ca(OH)_2 + H_2SO_4 \rightarrow CaSO_4 + H_2O$)) in acid medium showing more than 35% mass loss. SRC, due to its sulfate resistance, indicates lower degradation as compared to PCC. This is because of sulfate resisting cement has low calcium oxide content and relatively high alumina content than Portland cement. The reduction in $Ca(OH)_2$ and an increase in the alumina content is known to improve the acid resistance of concrete [28]. SC has low absorption and high chemical resistance against H_2SO_4. Moreover, there is no vulnerable alkaline chemical compound in SC [29] to aggressively react with acid i.e. H_2SO_4. The reaction between sulfur and H_2SO_4 is only possible at boiling temperatures which leads to the formation of sulfur dioxide and water. The effect of recycling does not change the acid resistance of SC. The 1st recast SC shows better resistance than freshly cast SC. There is a slight decrease in the resistance of SC after 2nd recasting and this is attributed to increased water absorption of SC after 2nd recasting.

Salt and base resistance of each mix was also measured for change in mass of the specimens after exposure to chemical solutions. In Fig. 11, SRC and PCC mixes indicate more mass loss than SC mixes. Similarly, recast SC mixes are also more stable than conventional mixes. A similar finding was made by Mohammed and Poornima [27]. PCC and SRC are more susceptible to the saline environment because of the presence of free Ca^{+2} and OH^- ions. Hydroxyl ions are highly reactive with cations (Na^+ and Mg^{+2}) of salts. The reaction of cations and anions results in the formation ($Na^+ + Ca^{+2} + OH^- + 2Cl^- \leftrightarrow NaOH + CaCl_2$) of crystals of new salts such as $CaCl_2$, which can initiate the internal deterioration mechanism of salts. In case of SC, there is no such possibility of these reactions leading to microstructural degradation.

The results of the base conditioning for each mix, in Table 4, show that SC is susceptible to 5% NaOH solution, whereas, conventional mixes show better resistance in base solutions than SC mixes. Also, most of the SC mixes dissolved in the base solution. The reaction between sulfur and NaOH is possible at 600 °C to produce sulfite and sulfide of sodium. Since the base conditioning is performed at room temperature; therefore, there is no possibility of such a reaction. Since, Sargodha aggregate is a dolerite sandstone [30], it is suggested that the alkali-silica reaction ($SiO_2 + 2NaOH + H_2O \rightarrow Na_2SiO_3.2H_2O$) between siliceous aggregates and alkali solution might be the reason for the rapid destruction of SC in the base solution [31]. Alkali combines with the silica of aggregates to produces hygroscopic gel around the aggregate particle. This hygroscopic gel absorbs water and swells (building up huge pressure inside concrete). The bond between the binder matrix and aggregates becomes weak. The less deterioration of PCC and SRC compared to SC is possibly due to the high alkalinity of cement. The high alkalinity of cement due to the calcium hydroxide can lead to slower alkali-silica expansion caused by external Na^+.

5. Conclusions

Following conclusions are drawn from the present study:

1. SC shows better mechanical properties than normal strength conventional concretes i.e. SRC and PCC. Strength of SC undergoes improvement of 30% after first recycling. Drastic drop in strength properties of SC is noticed after 2nd recycling.
2. SC shows significantly lower water absorption than both PCC and SRC. The 1st recasting showed a positive influence on water absorption of SC. Moreover, 2nd recasting caused a marginal decrease in water absorption of SC, yet the absorption capacity of SC was lower than that of the conventional mixes.
3. SC is highly resistant to both acid and salt environment. Recasting did not show any negative influence on the acidic and saline resistance of SC. Acid resistance of freshly cast and 1st recast SC was 40 times higher than that of the PCC and 30 times higher than that of SRC.
4. Neither of freshly cast SC or recycled SC withstands highly alkaline environment. SC completely loses its strength in NaOH solution, whereas both PCC and SRC were resistant to alkali-conditioning.

Future research

Sulfur concrete (SC) should be studied for its structural suitability in the precast elements such as masonry blocks, paving tiles, precast slabs, etc. Furthermore, recycling potential of SC should be explored considering the microstructural characteristics of sulfur binder. Interdisciplinary collaborations between polymer-chemistry and concrete material experts will facilitate better understanding of the engineering properties of fresh and recycled SC.

Author statement

We confirm that the manuscript has been read and approved by all named authors and that there are no other persons who satisfied the criteria for authorship but are not listed. We further confirm that the order of authors listed in the manuscript has been approved by all of us.

We understand that the Corresponding Author is the sole contact for the Editorial process (including Editorial Manager and direct communications with the office). He/she is responsible for communicating with the other authors about progress, submissions of revisions and final approval of proofs.

Declaration of competing interest

Authors have no potential conflict of interest.

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