



Effective reutilization of textile sludge from common effluent treatment plant with mineral admixture as a partial replacement for cement in mortar mixes

Somya Agarwal¹ · Ajit Pratap Singh¹

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Abstract

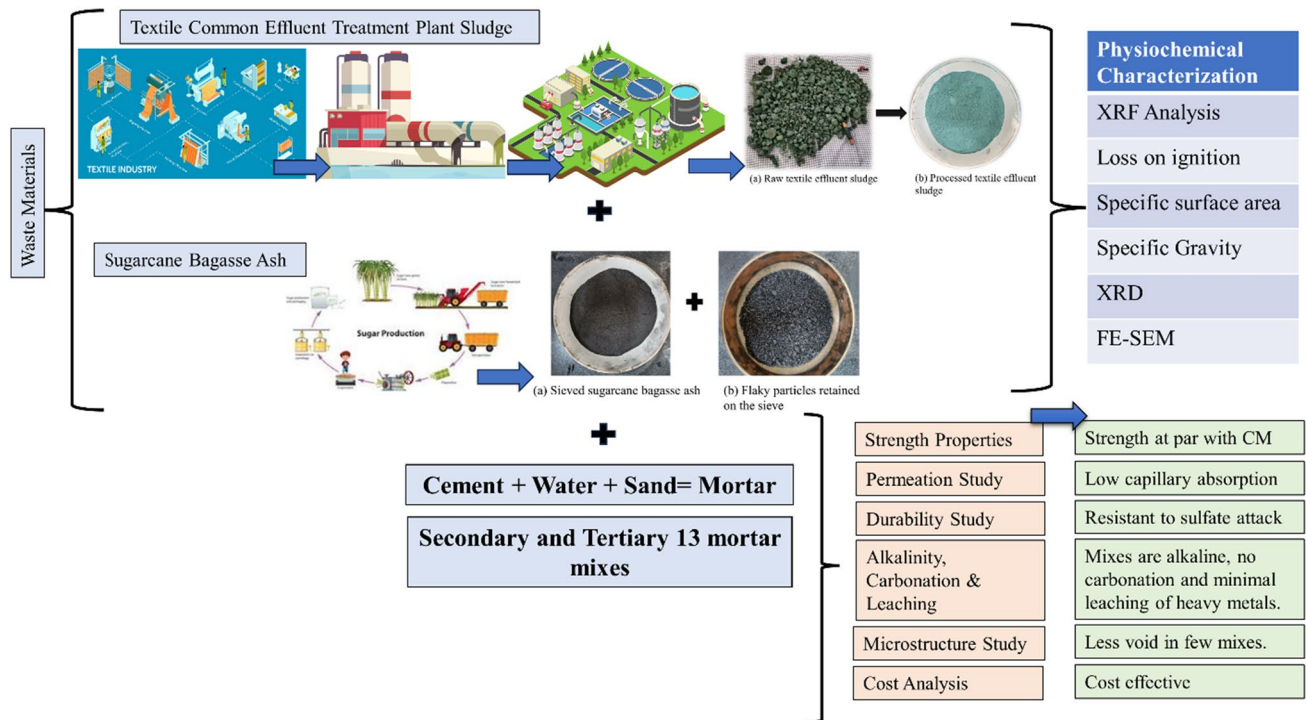
Treatment of textile industry effluents produces hazardous sludge. The improper disposal of sludge causes secondary pollution due to the leaching of heavy metals from it. Therefore, the prerequisite for the disposal of such hazardous sludge is its stabilization and solidification. The utilization of sludge as a resource for building materials is one of the sustainable solutions. The present study evaluates the feasibility of partially substituting cement with the textile common effluent treatment plant (TCETP) sludge and mineral admixture such as sugarcane bagasse ash (SBA) in cement mortar mixes. The 13 mortar mixes are prepared consisting of a control mix, four binary mixes with sludge (2.5, 5, 7.5, and 10%) and eight tertiary mixes with sludge (2.5, 5, 7.5, and 10%) and SBA (5, 10%) replacing cement by volume. Few binary and tertiary blended cement mortar mixes have demonstrated comparable strength, permeation, durability, and leaching properties that are on par with the control mix. The modified mortar mixes 2.5T, 5T, 2.5T5S, 5T5S, and 7.5T5S have improved strength compared to 7.5T, 10T, 10T5S, 2.5T10S, 5T10S, 7.5T10S, and 10T10S. Increased strength in mortar mixes is mainly attributable to the filler effect of sludge and SBA and the development of secondary CSH gel. The mortar mixes 7.5T, 10T, 10T5S, 2.5T10S, 5T10S, 7.5T10S, and 10T10S have increased sorptivity indices showing the presence of large-size pores. Durability results suggest a loss in strength due to sulfate attack. Carbonation is not observed in the mixes, and all the mixes are alkaline. However, the leaching study shows the presence of heavy metals in leachate solution above the permissible limit, mainly with mixes having 10% sludge and is within the permissible limit for all other mixes. The SEM image and XRD fingerprint analysis revealed the formation of porous structure and a reduction in CSH gel formation at higher replacement by sludge and SBA.

✉ Ajit Pratap Singh
aps@pilani.bits-pilani.ac.in

Somya Agarwal
p2015040@pilani.bits-pilani.ac.in

¹ Civil Engineering Department, Birla Institute of Technology and Science, Pilani 333031, India

Graphical abstract



Keywords Textile common effluent treatment plant sludge · Compressive strength · Hazardous sludge · Sustainable · Binary mixes · Tertiary mixes

Abbreviations

ASTM	American standard of testing and materials
BET	Brunauer–Emmett–Teller
BIS	Bureau of Indian Standards
CETP	Common effluent treatment plant
CH	Calcium hydroxide
CM	Control mix
CSH	Calcium silicate hydrate
E	Ettringite
FE-SEM	Field emission scanning electron microscopy
ICP-OES	Inductively coupled plasma-optical emission spectroscopy
IS	Indian Standard
OPC	Ordinary Portland cement
Q	Quartz
SBA	Sugarcane bagasse ash
SEM	Scanning electron microscope
TCETP	Textile Common Effluent Treatment Plant
TCLP	Toxicity characteristics leaching procedure
US EPA	United States Environment Protection Agency
V	Voids
XRD	X-ray diffraction
XRF	X-ray fluorescence

Introduction

Rapid urbanization and unprecedented infrastructure growth have driven increased demand for industrial goods, promoting large-scale industrial growth. This generates excessive anthropogenic waste and poses a huge challenge to human beings for its safe disposal. Landfilling and incineration are the two most used disposal techniques of solid waste. Landfilling hazardous industrial waste brings secondary challenges such as soil pollution, groundwater pollution through the leaching of heavy metals and air pollution. The incineration poses the risk of air pollution, and the ash generated must be disposed of safely. It is, therefore, imperative to find a sustainable use for industrial waste that stabilizes hazardous chemical compounds [1].

The textile industry in India is the second-largest employment sector in India [2]. This industry uses chemicals such as caustic soda, dyes, and pigments along with water, thereby generating chemical effluent. The industrial effluent before final discharge into the environment needs treatment so that the environment does not deteriorate further. Hence, the textile common effluent treatment plant (TCETP) collects and treats the effluent. The sludge is generated

as a residual during the physio-chemical treatment of the effluent at TCETPs. Heavy metals including chromium (Cr), zinc (Zn), lead (Pb), ammonium, and organic and inorganic materials are present in hazardous sludge [3]. The typical discarding methods, such as open dumping, landfilling, and incineration, are not recommended due to the hazardous nature of the textile sludge since they result in secondary pollution. Therefore, finding a distinct approach to immobilizing hazardous compounds through solidification and stabilization is crucial. One such approach is to use the TCETP sludge as a partial substitute for construction materials.

Several researchers have investigated the possibility of reusing TCETP sludge by partially replacing clay in bricks and fine aggregate, cement in mortar and concrete [4, 5]. As per the studies, 10–15% of clay is replaced by TCETP sludge in brick, which is found to be optimal [6, 7]. The optimum replacement percentage of TCETP sludge in cement–mortar mixes is comparatively low (5–10%) as, at a higher percentage, the strength decreases rapidly [8, 9]. The presence of heavy metals and organic debris retards the hydration process and has an undesirable effect on the strength attributes of cement-based mixtures [10].

Patel and Pandey have replaced 30–70% of cement with sludge in blocks, and the compressive strength results show that the mixes cannot be used for structural purposes [3]. Zhan and Poon pretreated the sludge with lime to reduce the ammonia (present due to azo dye) from the sludge and used it to replace the fine aggregate (0–30%) by weight. The 10% substitution by lime-treated sludge

satisfies the minimum strength for a non-load-bearing structure [9]. Goyal et al. replaced the cement with textile sludge from 0 to 20% by weight and found no adverse impact on the structural properties of cement mortar till the 5% replacement level [8]. Rahman et al. have used the TCETP sludge to replace the cement/natural fine aggregate in mortar and concrete. The reduction in strength, along with increased water absorption and porosity, is observed in the mixes [11]. Zhan et al. have evaluated the effect of TCETP sludge (0–20%) on the strength, leaching, and microstructure of cement mortar mix. A decrease in 71% of compressive strength on 20% substitution of cement by sludge is reported due to the weak interface and development of macropores [12]. Goyal et al. treated the TCETP sludge with the low-grade MgO for stabilization and then used it to make the mortar specimen. Up to 10% replacement with the MgO stabilized TCETP sludge is reported to have no negative impact on the properties of mortar [1]. The summary of the previous literature is presented in Table 1.

The literature has reported that the lower amount of silica in textile sludges results in poor pozzolanic activity on addition in the cement mixes [8]. However, mineral admixture from the agricultural waste rich in silica, i.e., sugarcane bagasse ash (SBA), is added to improve the pozzolanic activity by providing silica needed for converting CH into secondary CSH in the later stages of mortar mixes. SBA is responsible for developing secondary CSH gel and improving the cement mixes' strength, permeation, and durability properties [13].

Table 1 Literature review summary

Reference	Product formed	Pre-treatment	Replacement %	Results
Patel and Pandey [3]	Cement sludge blocks	No	30–70% replacement of cement	Compressive Strength reduced from 60 to 90% of control mix
Zhan and Poon [9]	Concrete Blocks	Lime-based pretreatment	0–30% replacement of fine aggregate	10% replacement satisfies the minimum strength criteria for non-load bearing structure
Goyal et al. [8]	Mortar blocks	No	0–20% replacement of cement	No adverse impact on the strength of the mortar mixes up to 5%
Zhan et al. [12]	OPC-TES mortar blocks	No	0–20% replacement	Even at 5% replacement, 47.7% decrease in compressive strength was observed
Goyal et al. [8]	Mortar Blocks	Low-grade MgO stabilization	5–15% MgO stabilized sludge and 0–15% cement replacement	10% replacement with 15% MgO stabilized sludge resulted in no adverse impact on compressive strength of mortar mixes
Present Study	Mortar Blocks	Sludge and Sugarcane bagasse ash (SBA)	Sludge (2.5–10%) and SBA (5%, 10%) partial replacement of cement	Mixes with 7.5% sludge and 5% SBA have achieved 95.66% of control mix strength

The sugarcane bagasse is the waste residue produced by the sugar manufacturing units. This biomass is used in cogeneration boilers for electricity production. The SBA is produced as waste in boilers and is disposed of in landfills [14]. The uncontrolled disposal of SBA leads to environmental problems such as air pollution, and the particulate matter present in SBA is hazardous to human health. The SBA combines flaky, tubular, irregular-shaped, lightweight porous particles rich in silica and alumina [15, 16]. Gupta et al. observed the maximum compressive strength for 5% substitution of cement with SBA [17]. Batool et al. have partially substituted the cement with SBA in concrete, and 10% is the optimal replacement for higher compressive strength [15]. Chi has also indicated 10% as the optimal replacement of binder material with SBA for mortar [18]. Mohan et al. have suggested 15% optimal cement replacement in mortar mixes using the high temperature (750–800 °C) cogeneration plant bagasse [19]. Nassar et al. have reported that 10% is the optimal cement replacement by SBA [20]. Queduo et al. have inferred that the concrete mixes with 10% binder material replaced with SBA have shown positive performance [21]. Therefore, considering the above-reported studies, 5 and 10% substitution are considered in this study, along with the TCETP sludge.

The use of TCETP sludge and SBA as the building material might benefit society in several ways; (1) the stabilization and safe disposal of hazardous textile effluent sludge and conversion into a stable product; (2) the use of agricultural waste SBA as a mineral admixture in mortar mixes will reduce the landfill issue of the SBA; (3) the study will curb the exploitation of natural resources as the construction material using the sludge and SBA as a replacement for conventional building material; (4) the majority of earlier research has been on weight replacement of building materials with TCETP sludge and SBA, despite the difference in the specific gravity of cement and sludge, SBA.

According to the literature, researchers over the recent years have attempted to use either sludge or SBA to substitute cement partially. The current work seeks to assess and compare the performance of mortar mixes including both sludge and SBA by strength tests, durability, permeation, carbonation, alkalinity, and leaching test. The study also analyzes the micro-structural properties of the mixes using FE-SEM and XRD techniques. The study is divided into four sections, namely, the introduction that explains the need for stabilization of sludge and past literature on the use sludge and SBA in mortar mixes, followed by the section of methodology. Methodology section explains the various experiments designed and procedures used as per the standards by Bureau of Indian Standard (BIS) and American Society of Testing and Material (ASTM). This is followed by results and discussion of the outcome of numerous experimental studies while the final section, i.e., conclusion incorporates inferences drawn from the findings of experimental results.

Methodology

Material

The study uses Ordinary Portland Cement (OPC) of 43-Grade, a commercially accessible binder material for cement mortar mixtures as per IS 8112:2013 [22]. Textile sludge was obtained from the CETP of Balotra and is blue. Sludge preprocessing includes air drying followed by over-drying for 24 h at 105 °C to remove excess moisture and ground using the tumbling mill. It is sieved through 90 µm [8]. The raw and processed TCETP sludge is presented in Fig. 1. SBA was obtained from Awadh Sugar Mills Ltd. company in Uttar Pradesh. The SBA was also oven dried for 24 h at 105°C and sieved through 90 µm, as depicted in Fig. 2 [23]. Dried sludge, cement, and SBA samples are investigated for their chemical composition using X-ray

Fig. 1 TCETP sludge: **a** raw, **b** processed

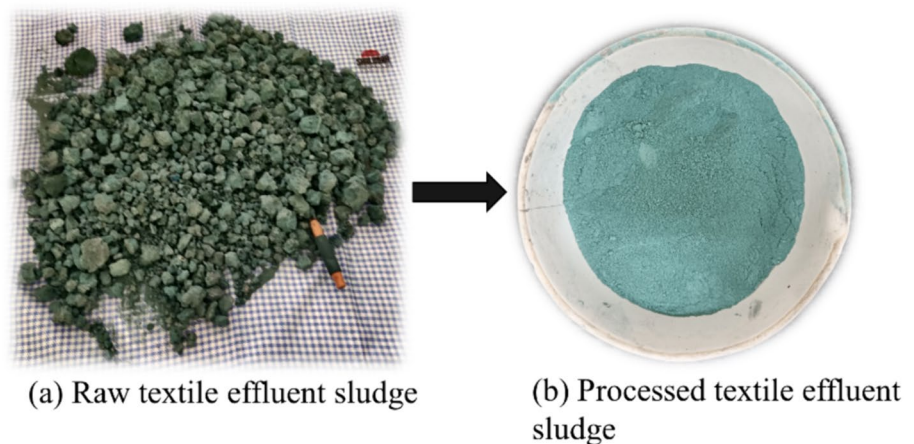
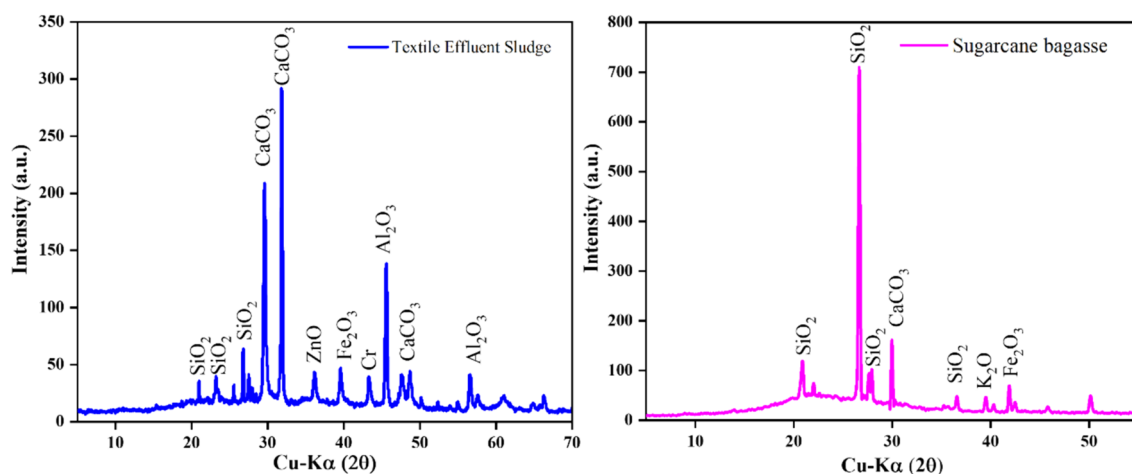


Fig. 2 Sugarcane bagasse ash: **a** sieved, **b** flaky particles**Table 2** Physicochemical properties of binder materials (cement, TCETP sludge, and SBA)

Formula	Cement	Textile effluent sludge	SBA
CaO	64.91	52.222	6.829
SiO ₂	20.62	13.403	70.529
Al ₂ O ₃	4.794	5.590	3.025
Fe ₂ O ₃	3.409	3.222	4.424
K ₂ O	1.025	0.534	8.338
MgO	3.6	–	–
Na ₂ O	1.04	–	1.482
P ₂ O ₅	0.10	–	3.939
TiO ₂	0.21	0.217	0.479
Loss on ignition	2.18	29.74	13.32
Physical properties			
Specific surface area (BET) (m ² /g)	0.3291	22.89	2.14
Specific gravity	3.17	2.29	1.78
Color	Grey	Blue–green	Dark grey

fluorescence (XRF) analysis. The physicochemical characteristics of the three binder materials (cement, TCETP sludge, and SBA) are given in Table 2. Mineralogical constituents of TCETP sludge, and SBA, determined by the X-ray diffraction (XRD), are given in Fig. 3. The XRD peak analysis of the sludge shows the presence of calcium carbonate, silica oxide, zinc oxide, ferric oxide, chromium, and aluminium oxide. The fingerprint of the SBA shows that silica oxide is the main component. Figure 4 depicts the scanning electron microscopy (SEM) analysis of TCETP sludge and the SBA. The SEM images show the irregular-shaped particles for the sludge and the porous, flaky particles for the SBA. The specific surface area of the cement, TCETP sludge, and SBA is measured by the Brunauer–Emmett–Teller (BET) analyzer. The specific gravity was measured per IS 4031 (part 11):1988 [24]. Results indicate that sludge has low specific gravity and high specific surface area.

Coarse aggregate, mainly sand comprising three grades of particles passing through a 2, 1 mm, and 500 µm sieve and retained on a 1 mm, 500, and 90 µm sieve, respectively,

**Fig. 3** XRD fingerprints of **a** TCETP sludge and **b** SBA

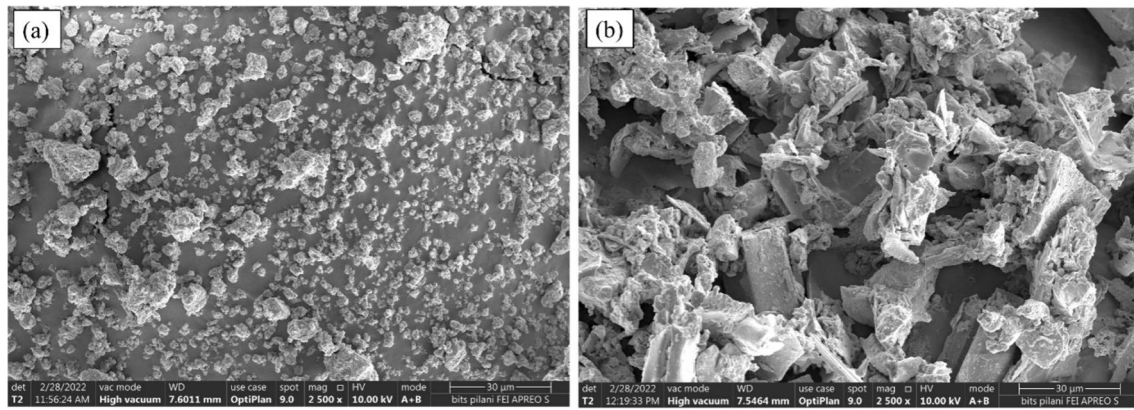


Fig. 4 SEM images of **a** TCETP sludge and **b** SBA

are used in the study to prepare the mortar mixes as per IS 650:1991 [25]. All these three sizes of sand are mixed in equal proportion for each mortar mix. The mortar mixtures use potable water as per IS 456:2000 [26].

Mix proportions

Thirteen mortar mixes are prepared to study the feasibility of partially replacing cement with TCETP sludge and SBA in mortar mixes. The control mix is designated as CM. The cement was replaced by sludge at 2.5, 5, 7.5, and 10%, whereas SBA at 5 and 10% by volume. The mortar sample preparation, mixing, moulding, and curing were performed as per IS 4031 (Part 8):2005 [27]. The prepared mortar specimens were investigated for their strength, permeation, durability, and microstructure properties. The water-to-cement (w/c) ratio for the 13 mortar mixes is maintained at 0.44, 0.46, and 0.48 based on the standard consistency test

performed on the cement–sludge–bagasse pastes under IS 4031 (Part 4):1988 [28]. During the preliminary study, the sludge and SBA absorb the water due to the high specific surface area as given in Table 2, reducing the consistency of the mixes. Therefore, excess water is required for adequate standard consistency of the mixes at high replacement. The binder to the sand ratio (b/s) is maintained constant at 1: 3 following the IS 4031 (Part 6):1988 [29]. The details of the considered mixes are shown in Table 3.

Test procedures

The strength, permeation, durability, alkalinity, carbonation, and leaching properties of the 13 modified mortar mixes are evaluated. The following describes the test methods used to investigate each property:

Table 3 Mix proportion of mortar mixes

Mix notations	% of cement replaced by		Mix proportion (g)				
	Textile sludge	Sugarcane bagasse ash	Cement	Sand	Textile sludge	Sugarcane bagasse ash	water/binder
CM	0	0	200	600	0	0	0.44
2.5T	2.5	0	195	600	3.61	0	0.44
5T	5	0	190	600	7.22	0	0.44
7.5T	7.5	0	185	600	10.83	0	0.46
10T	10	0	180	600	14.44	0	0.46
2.5T5S	2.5	5	185	600	3.61	5.61	0.46
5T5S	5	5	180	600	7.22	5.61	0.46
7.5T5S	7.5	5	175	600	10.83	5.61	0.46
10T5S	10	5	170	600	14.44	5.61	0.46
2.5T10S	2.5	10	175	600	3.61	11.23	0.48
5T10S	5	10	170	600	7.22	11.23	0.48
7.5T10S	7.5	10	165	600	10.83	11.23	0.48
10T10S	10	10	160	600	14.44	11.23	0.48

Strength testing

The performance of modified mortar mixes is examined for compressive, split tensile, and flexural strength tests at 7, 28, and 90 days of immersed water curing. For compressive strength, nine cubes of 70.6 mm were casted for each mix and tested per the procedure mentioned in IS 4031 (Part 6):1988 [29]. The split tensile strength is alternate method to measure the tensile strength and the flexural strength is the measure of tensile strength during bending. The prismatic specimens (beams) of size 160 mm × 40 mm × 40 mm are prepared and tested for flexural strength as per ASTM C78:2019, and 100 mm × 50 mm (diameter × height) cylindrical samples are casted and tested for split tensile strength test [30, 31]. It is worth mentioning that an average of three specimens is considered for strength testing. Figure 5 depicts the experimental setups for the strength testing. A total of 351 specimens consisting of 117 specimens each of 70.6 mm cubes, beams, and cylinders were prepared for the strength test.

Permeation study

The permeation characteristics of the mortar mixes are assessed using the sorptivity test and performed as per ASTM C1585:2013 [32]. For sorptivity tests, two cylindrical specimens of 100 mm × 50 mm (diameter × height) are tested for sorptivity test for each mix. The experimental setup for the sorptivity test is represented in Fig. 6.

For the sorptivity test, the specimens are kept in the environment chamber (temperature of 50 ± 2 °C, relative humidity of $80 \pm 3\%$) for 3 days. Then the samples are placed in the sealable container for the remaining time and the sample age is 28 days at the time of testing. The circumference of the specimens is covered with aluminum tape, and the top surface is covered with silicon sealant so that capillary water absorption can take place from only one surface, which is in contact with the water.

Fig. 5 Strength test setup: **a** compressive, **b** tensile, and **c** flexural

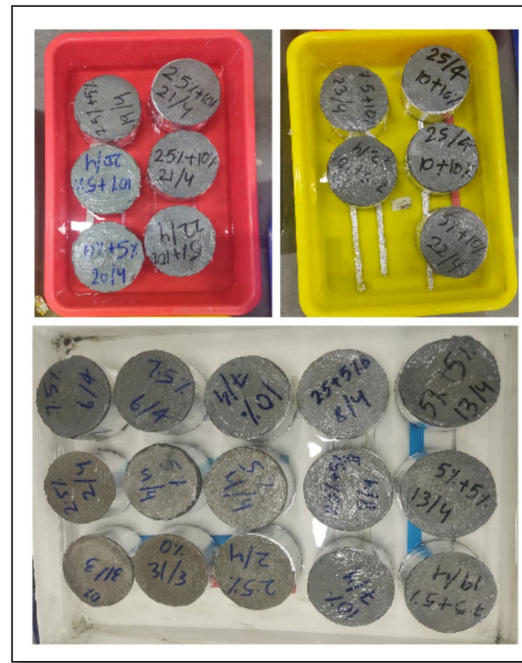
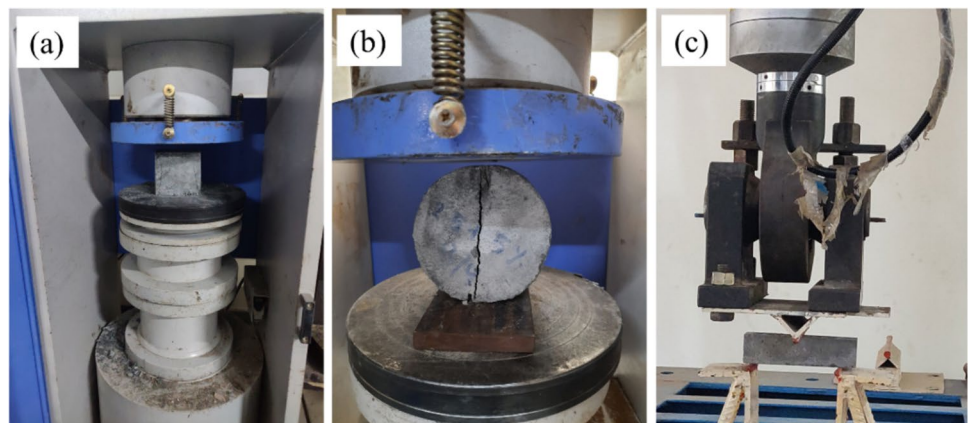


Fig. 6 Experimental setups for the sorptivity test

Durability study

The durability study is mainly performed to study the resistance to adverse scenarios to which the mortar may be exposed during its service life. The cement mortar mixes could deteriorate when in contact with sulfate present in water or soil. The durability study for sulfate resistance is done by immersing the three cubes of 70.6 mm in a 5% magnesium sulfate (MgSO_4) solution for 90 days, including 28 days of moist curing per ASTM C1012:2004 [33].

Alkalinity, carbonation, and leaching

Measuring the pH of mortar mixtures is crucial because their acidic nature may degrade the passive layer surrounding the steel rebars in reinforced concrete. The pH of the mortar mixes is tested by mixing 30 gm of crushed samples from the 28 days compressive strength (passing through a 300 μm sieve) in 100 ml of Millipore water [1]. The pH is measured using the pH electrode by Hanna instruments. The carbonation of the mortar mixes is checked by spraying the phenolphthalein indicator (1% solution) on the freshly split beam surfaces after the flexural test at 28 and 90 days [34]. The phenolphthalein indicator changes color in the pH range of 8.2–10.

The toxicity characteristics leaching procedure (TCLP), in compliance with US EPA 1311, was performed to study the dissolution of the heavy metals from modified mortar mixtures [35]. As per the US EPA 1311, for the sample with initial pH > 5, the extraction fluid is made by diluting 5.7 ml of glacial acetic acid with 1 l of Millipore water. The extracted fluid should be preserved at pH of 2.88 ± 0.5 . The 5 g of mortar specimens from each mix from the 28-day compressive strength test are crushed and sieved through the 300 μm standard sieve. The crushed sample to extraction fluid ratio for each batch experiment is kept at 1:20. The batch experiments are performed by mixing 5 g of the sample with 100 ml of extraction fluid in a 250 ml Erlenmeyer flask. Each flask is kept in the orbital shaker for horizontal shaking at 180 r.p.m and 18 h at 25 °C. After shaking, the mixture is filtered through a 0.45 μm filter paper followed by 0.22 μm syringe filtration. The filtrate is preserved in 50 ml centrifuge tubes by acidification using the HNO_3 to a pH of <2. The concentration of heavy metals in the filtered leachate samples is tested using inductively coupled plasma- optical emission spectroscopy (ICP-OES), and data are compared to WHO and Indian Standard Drinking Water Specification as per BIS 10500:2012 [36].

Microstructure of the mixes

The microstructure of the mortar mixes was studied on the broken specimens of the 28-day compressive strength test. These broken samples are stored in bottles containing acetone. The SEM analysis assists in studying the modification in the morphology of mortar mixes with the addition of TCETP sludge and SBA. Image J software is used to analyze SEM images. And pore area is calculated for the mixes. Similarly, XRD fingerprinting for the mortar mixes is also performed. After the 28-day compressive strength test, the mortar mixes are ground using mortar n pestle and sieved through a 75 μm sieve before testing for XRD.

Result and discussion

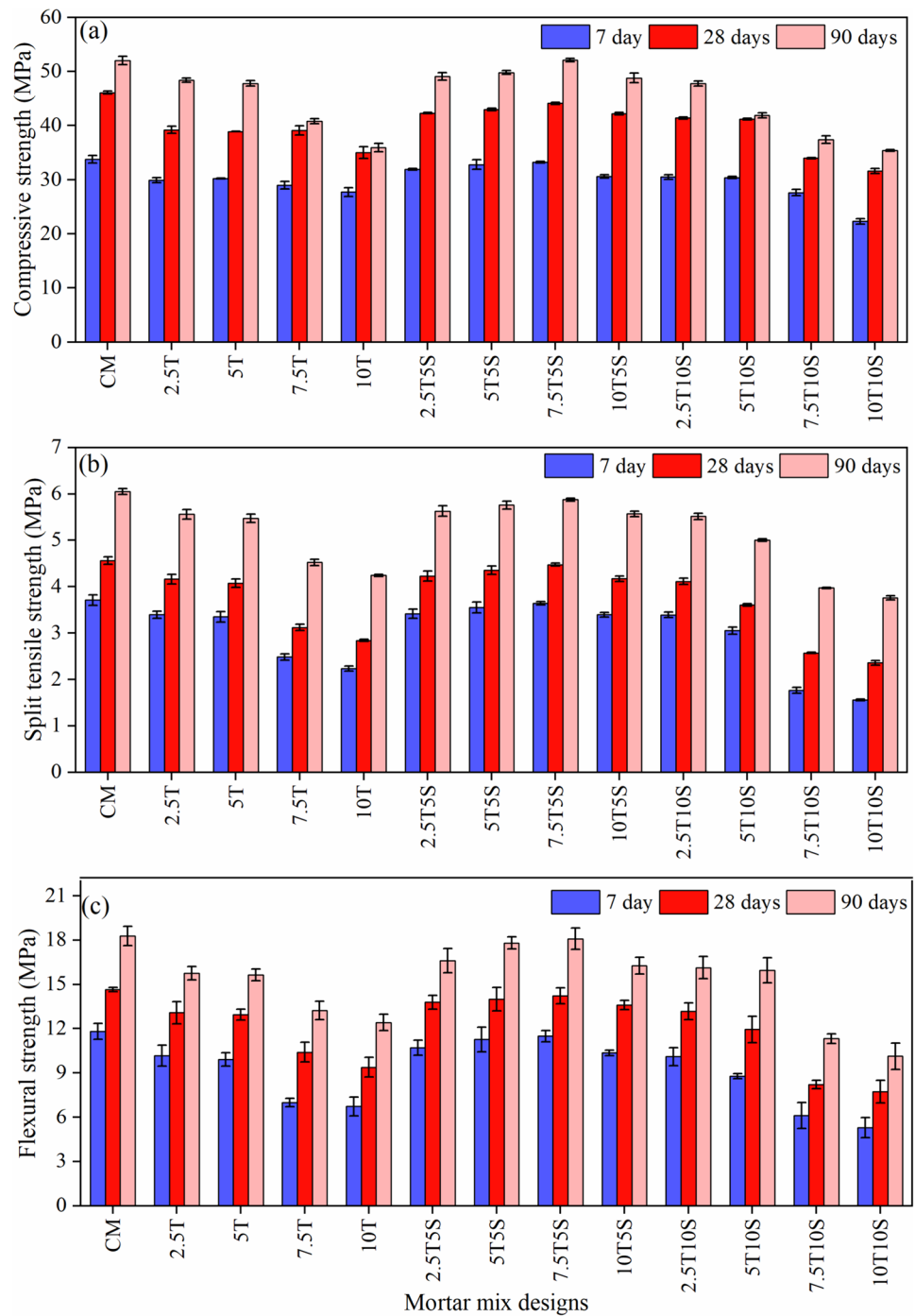
Strength properties

The strength of modified mortar mixes at 7, 28, and 90 days of moist curing is depicted in Fig. 7. As seen from Fig. 7, there was an increase in the strength of the mortar specimen with age; though, there was a decrease in strength for all the modified mortar mixes. The 28-day stipulated compressive strength for the cement mortar mixes using the OPC 43 cement is 43 MPa as per IS 456: 2000 [26]. Mixes 5T5S and 7.5T5S have achieved 93.27 and 95.66% of the characteristic strength of 43 MPa of the control mix strength. The binary mixes 2.5T, 5T, 7.5T, and 10T with only the sludge as cement replacement material have gained compressive strength of 85.03, 84.38, 84.81, and 75.92% of the control mix. The tertiary mixes 2.5T5S, 5T5S, 7.5T5S, and 10T5S have achieved strength of 91.75, 93.27, 95.66, and 91.5% of the control mix, respectively. The tertiary mixes 2.5T10S, 5T10S, 7.5T10S, and 10T10S have a compressive strength of 89.80, 89.37, 73.75, and 68.54% of the control mix, respectively. The addition of 5% of SBA with sludge has improved the strength of the mixes, and a significant increase is observed in long-term strength (90 days).

The modified mortar mixes are tested for split tensile and flexural strength, as depicted in Fig. 7b, c and are compared with the 28-day strength of the control mixes. The binary mixes 2.5T, 5T, 7.5T, and 10T have achieved 91.15, 89.2, 68.368, and 62.234%, tertiary mixes 2.5T5S, 5T5S, 7.5T5S, and 10T5S have 92.64, 95.44, 98.05, and 91.347%, and the mixes 2.5T10S, 5T10S, 7.5T10S, and 10T10S have 90.142, 78.97, 56.364, and 51.632% of the control mix split tensile strength (4.565 MPa), respectively. The maximum and minimum split tensile strength of 4.556 MPa and 2.357 MPa is achieved by the 7.5T5S and 10T10S mortar mixes, respectively. The binary mixes 2.5T, 5T, 7.5T, and 10T have achieved 89.314, 88.4, 71.02, and 64.074%, tertiary mixes 2.5T5S, 5T5S, and 7.5T5S have 94.11, 95.469, and 97.073%, respectively and the mixes 2.5T10S, 5T10S, 7.5T10S, and 10T10S have 89.98, 81.49, 56.076, and 52.849%, of the control mix flexural strength (14.655 MPa). The results of split tensile and flexural strength test exhibit similar patterns to those of compressive strength test.

The compressive strength of binary mixes containing 2.5 and 5% of sludge is at par with the CM due to the filler effect of the sludge. Further increasing sludge content has resulted in a decrease in compressive strength of mortar mixes which indicates slow hydration reaction with the incorporation of sludge compared to cement [37]. TCETP sludge has less lime and silica oxide than cement as observed from XRF results in Table 2. This results in less hydration products and insufficient pozzolanic

Fig. 7 Strength behavior of the different mortar mix design: **a** compressive strength, **b** split tensile strength, and **c** flexural strength



activity. Therefore, with the increasing sludge content, CH formation may occur but will not get converted to CSH gel due to the absence of silica in sludge. The high LOI content of the sludge also results in a reduction of strength. Organic matter decomposes during the mixing which impedes the further hydration of cement [11, 12]. Further, the presence of heavy metals in sludge retards the hydration process and thus, reduces the compressive strength [1, 3]. Therefore, supplementary cementitious

material (SBA) is added in 5 and 10% to enhance the hydration and conversion of CH into CSH.

In the case of tertiary mixes with 5% SBA and 2.5–10% sludge, presence of additional silica may have improved the pozzolanic activity and have compressive strength at par with the CM. The presence of free lime in cement and sludge may have resulted in formation of additional CSH gel, thus improving the strength of the mortar mixes [17]. However, further increasing the SBA to 10% and sludge content to

7.5 and 10% results in reduced strength of mortar mixed. Sludge and SBA both contribute to slow hydration of the mortar mixes. The presence of organic matter in both SBA and sludge also retards the hydration of cement [38]. The sludge particles are finer than cement as act as the filler material and at 10% bagasse ash, the silica is available but free lime reduces due to decrease in cement content. Less availability of cement of hydration leads to less formation of CSH gel and reduced conversion of free lime, portlandite to secondary CSH gel. This leads to the formation of a highly porous matrix which is being studied using the sorptivity test. In addition, the existence of heavy metals (zinc, iron, chromium, and copper) in the TCETP sludge prevents the cement particles from hydration by forming a membrane of compounds such as calcium hydroxyl zincate ($\text{CaZn}_2(\text{OH})_6 \cdot 2\text{H}_2\text{O}$) around them [1, 12].

Permeability test

The modified mortar samples at 28 days of age are evaluated to comprehend the capillary suction behavior. The capillary suction through a surface in contact with the water determines the sorptivity, which controls the propensity of water absorption by the mortar mixtures. The pore shape and surface properties of the specimens significantly impact their capillary suction behavior [39]. The sorptivity indices for the 13 mortar mixtures are depicted in Fig. 8.

When compared to CM, the 2.5T and 5T specimens showed decreases in sorptivity index of 8.68, 13.79%, respectively. This shows that the sludge effectively filled the pores and reduced the size of the big pores. In the tertiary mixes 2.5T5S, 5T5S, and 7.5T5S, the sorptivity indices decreased by 23.56, 29.14, and 26.35%, respectively.

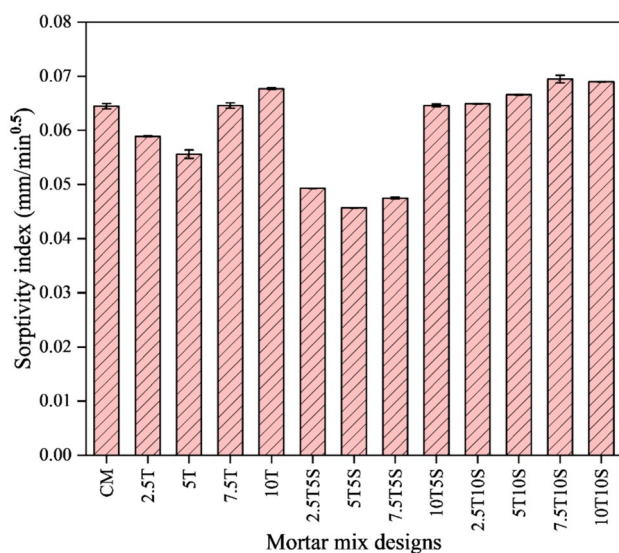


Fig. 8 Sorptivity index of the different mortar mix design

This is attributed to the filler effect and the potential for secondary CSH gel formation with the addition of SBA, which reduces large size pores and increases the denseness of the structure [8, 21]. However, the sorptivity indices were increased by 0.15 and 4.96% for the 7.5T and 10T mixes due to the lack of hydration of cement and increased size of pores. The sorptivity indices increased by 0.15, 0.62, 3.25, 7.75, and 6.97% for the 10T5S, 2.5T10S, 5T10S, 7.5T10S, and 10T10S mortar mixes, respectively. The increased capillary water absorption of the mixes is caused by the lack of cement hydration as a significant portion of cement is replaced by sludge and SBA, thus forming voids. Further, the mixes 2.5T10S, 5T10S, 7.5T10S, and 10T10S with 10% SBA require more water to form paste of standard consistency; thus, on drying, large size pores are formed which contributes to increase in sorptivity [17].

Durability study

The resistance to the sulfate ions attacks of the different mortar mixes is investigated by submerging the 28-day specimens in the 5% MgSO_4 for 90 days (including 28 days). The 5% MgSO_4 solution is revived every 2 weeks, and after the completion of 90 days, samples are washed in clean water. The compressive strength of the sulfate attack specimens is tested and compared with the specimens of moist curing for 90 days as per IS: 4031 (Part 6) [29].

The reduction in compressive strength due to the sulfate attack is observed for the CM and the other specimens. It is demonstrated in Fig. 9 that salt crystallization is predominantly responsible for the sulfate attack. Gypsum and ettringite are produced when the sulfate ions interact with calcium

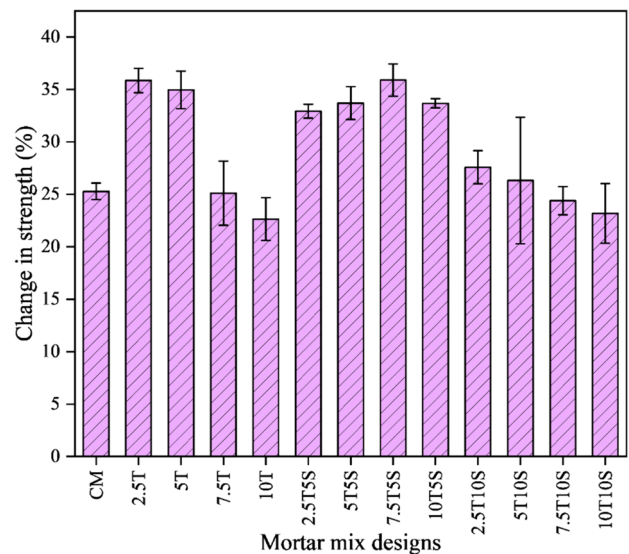


Fig. 9 Change in the strength (%) of the mortar mixes due to sulfate attack

hydroxide (CH) and calcium aluminum hydrate (C_3A). The ettringite is responsible for the volume expansion, and gypsum can cause softening and loss of mass. Initially, the formation of gypsum and ettringite in the micro-cracks and pores increases compressive strength. In the later stages, with the gradual increase in these products, the development of new cracks and fractures affects the compactness of the mixes [30]. However, there is 25% change in the strength of the CM, and 35.8, 34.969, 32.9, 33.7, and 35.9% for 2.5T, 5T, 2.5T5S, 5T5S, and 7.5T5S, respectively. The increased change in strength in the mixes is due to the filler action of sludge and the development of secondary CSH gel leading to a compact structure. It leads to the availability of fewer pores for the formation of ettringite and gypsum, thus reducing the compactness of mortar specimens. However, for the mixes 7.5T, 10T, 10T5S, 2.5T10S, 5T10S, 7.5T10S, and 10T10S, a change in the strength of 20–30% is observed. The lesser reduction in strength may be due to a large volume of pores and voids in these mixes leading to the greater availability of space for forming gypsum and ettringite. The pores and cracks are filled by these products, forming compact mortar.

Alkalinity, carbonation, and leaching tests

The pH of all the mortar mixes is determined and is reported in Table 5. The pH of the mortar mixes should lie between 12.0 and 13.0 to prevent corrosion of the reinforcement [40]. The pH of all the mixes under consideration is within the acceptable range. However, compared to CM, increase in pH is observed with addition of sludge and SBA in mixes.

The carbonation study is essential as the passive layer of the reinforcement deteriorates on the reaction of atmospheric CO_2 with the moisture present in the pores. As a result, the pH of pore moisture decreases due to the formation of carbonate and bicarbonate ions. After the flexural strength test, the phenolphthalein indicator is sprayed on the freshly split specimens. The sprayed surface turns pink, indicating the absence of carbonation [41]. All the mix designs, including the control mix, were observed to have no carbonation. Figure 10 represents the carbonation test performed on the beams.

In the present work, the heavy metal analysis for the four primary heavy metals (Cu, Cr, Fe, Zn) found in the XRF results of the sludge was analyzed for the 13 mortar mixes using the TCLP test. The amount of heavy metal in the sludge is determined by the digestion using concentrated HNO_3 [1]. Table 4 shows the XRF analysis of heavy metals in sludge, results of heavy metals in digested sludges, and the TCLP test analysis of the leaching of heavy metals from the sludge. Table 5 displays the TCLP test results for the various mortar mixtures. It is observed that the heavy metal content in the leachate of mortar mixes with 2.5, 5, and 7.5% of the sludge is under the permissible limit. The leaching of

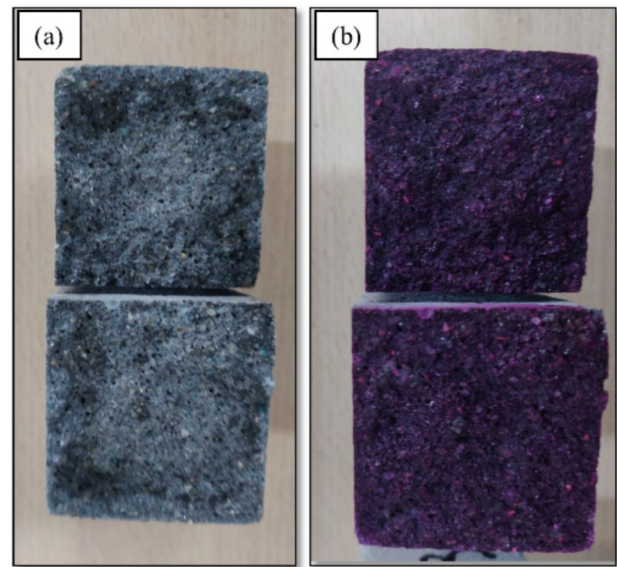


Fig. 10 Carbonation test: **a** the freshly broken beams, **b** specimen after the phenolphthalein indicator is sprayed

Table 4 Heavy metal determination in the raw sludge using XRF, after digestion, and TCLP leaching test

Heavy metals	XRF (TES) %	ICP-OES digested sludge (mg/kg)	ICP-OES (TCLP test) (mg/l)
Fe	3.616	442.4	3.4
Cu	0.495	68.8	1.6
Zn	0.686	225.9	11.8
Cr	0.090	11.2	0.4

zinc is under the permissible limit indicating the formation of the compound (calcium hydroxyl zincate) with cement. However, the leaching of heavy metal from the mortar mixes rises with an increase in sludge content. The heavy metals present in textile effluent treatment plant sludge (Cu, Zn, Cr) react with calcium to form hydrated compounds, thereby decreasing the content of CH in the mortar specimens. This leads to the formation of porous microstructure, which could be a reason for higher leaching of heavy metals at higher sludge content [12].

Microstructure study

XRD and SEM analyses are performed to analyse the components formed and the microstructure of the different mortar mixes. SEM images help study the microstructure changes in the mortar by adding sludge and SBA. Figures 11, 12, 13, and 14 depict the SEM and processed images for the CM, 5T, 7.5T5S, and 7.5T10S mortar mixes. The significant compounds observed in SEM analysis are

Table 5 Observed pH values and the heavy metal concentration of the mortar mixes

Mortar mix designs	Observed pH values	Cu (mg/l)	Cr (mg/l)	Fe (mg/l)	Zn (mg/l)
CM	12.48	0.027	0.065	0.194	0.097
2.5T	12.46	0.029	0.083	0.267	0.089
5T	12.51	0.033	0.088	0.285	0.177
7.5T	12.55	0.046	0.084	0.228	0.151
10T	12.57	0.081	0.089	0.353	0.184
2.5T5S	12.59	0.038	0.076	0.283	0.109
5T5S	12.61	0.039	0.081	0.297	0.066
7.5T5S	12.68	0.047	0.075	0.298	0.088
10T5S	12.72	0.059	0.117	0.303	0.069
2.5T10S	12.77	0.041	0.091	0.264	0.059
5T10S	12.78	0.043	0.088	0.272	0.083
7.5T10S	12.85	0.045	0.095	0.267	0.057
10T10S	12.89	0.061	0.163	0.371	0.104
WHO standards	–	2	0.05	NA	NA
CPCB	–	0.05	0.1	0.3	5

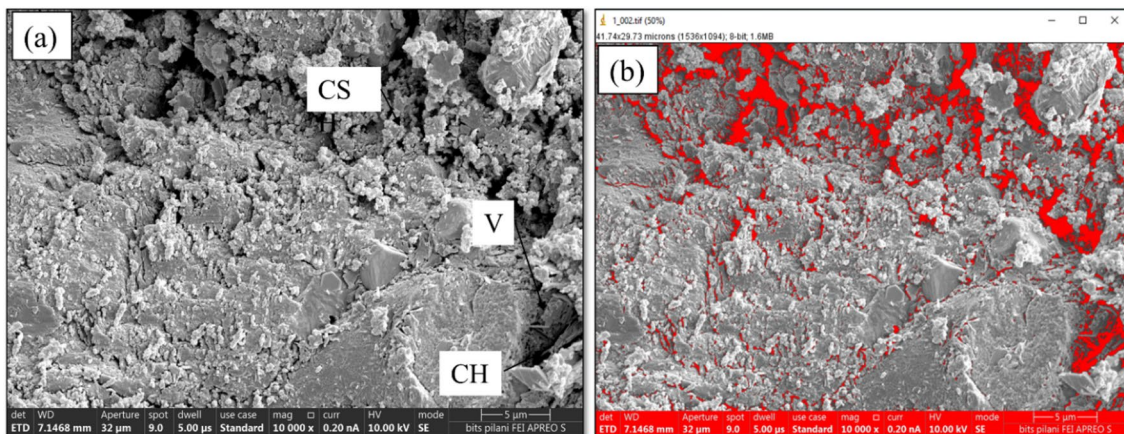
Bold values indicate that the leaching of heavy metals from the concerned mortar mixes has exceeded the prescribed permissible limit by CPCB, New Delhi

calcium hydroxide (CH), calcium silicate hydrate (CSH), voids (V), and ettringite (E).

It can be inferred from SEM analysis of mixes CM and 5T in Figs. 11 and 12 that the addition of sludge has led to the decrease in large-size pores due to its filler effect. In addition, as shown in Fig. 13, adding 5% SBA such as 7.5T5S promotes the formation of CSH which further reduces the pore area. It indicates the densification of the pore structure and, consequently, improved strength and permeation properties. However, increase in SBA to 10% and sludge to 7.5% in the mix such as 7.5T10S has led to the formation of large-size pores, as observed in Fig. 14. Therefore, the SEM analysis results corroborate well with the strength and permeation test performed in this study. The pore area for each

of the different mortar mixes is estimated using the Image J software and is represented in Fig. 15. The analysis of the pore area shows decreases in pore area with the addition of sludge and SBA but at the higher replacement amount, such as in the 7.5T10S mix, pore area significantly increases.

XRD analysis of the four mortar mixes CM, 5T, 7.5T5B, and 7.5T10B is presented in Fig. 16. The major compounds observed in the XRD fingerprint analysis are tricalcium silicate, dicalcium silicate, quartz, calcium hydroxide, calcium silicate hydrate, and ettringite. It is observed from the XRD analysis that the fingerprints of the mixes containing cement only, cement with sludge, and cement with sludge and SBA have not changed much. However, significant changes in the intensities of the compounds are observed for the mixes.

**Fig. 11** a SEM analysis and b processed image of the CM mortar specimen

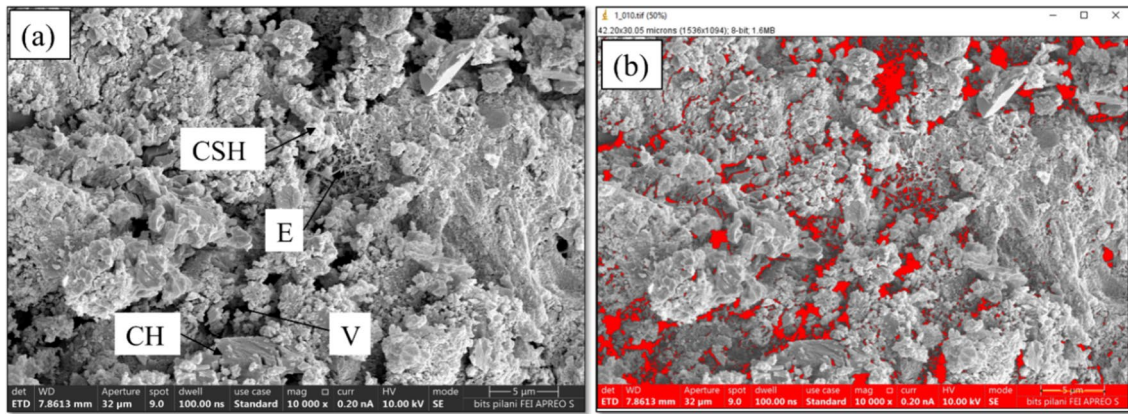


Fig. 12 **a** SEM analysis and **b** processed image of 5 T mortar mix

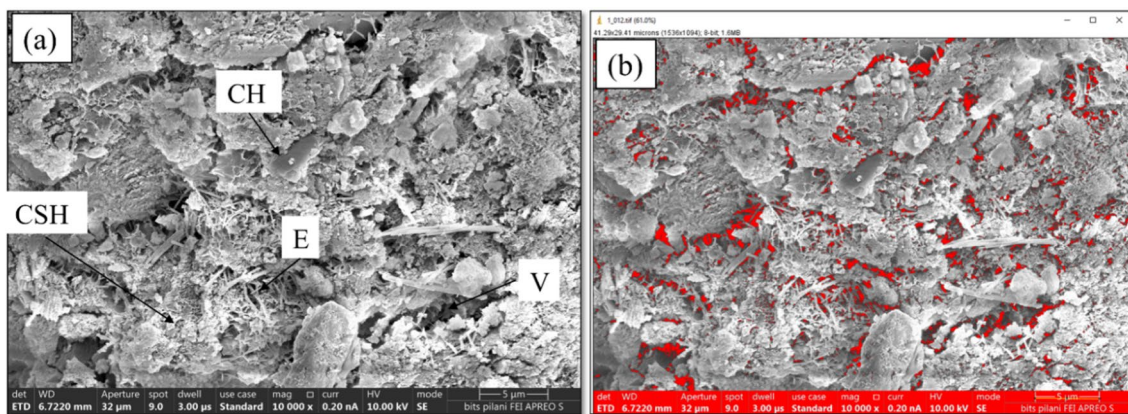


Fig. 13 **a** SEM analysis and **b** processed image of 7.5T5S mortar mix

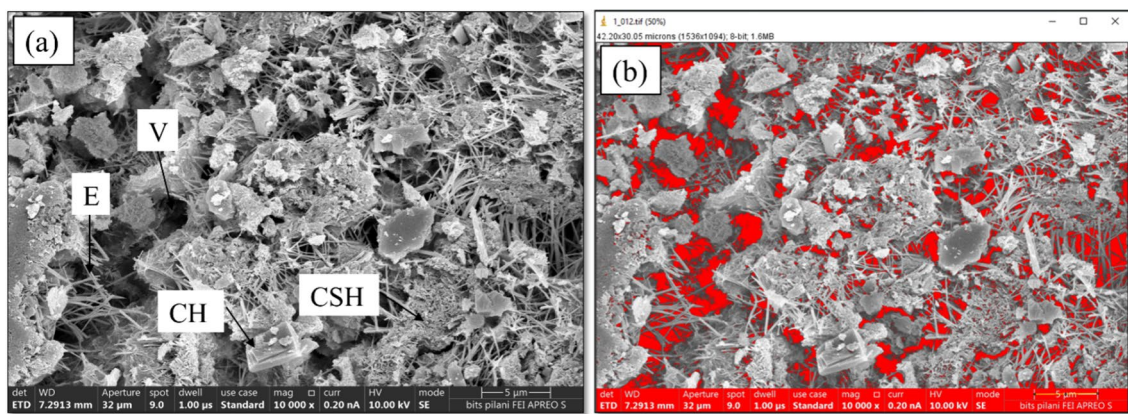


Fig. 14 **a** SEM analysis and **b** processed image of 7.5T10S mortar mix. *CH* calcium hydroxide, *CSH* calcium silicate hydrate, *V* voids, *E* ettringite

The addition of sludge shows the increased intensity of the peak of calcium carbonate and calcium silicate, as can be observed from Fig. 16a, b as compared to CM. However, the increased CSH and CH hydration products are observed

in the 5T and 7.5T5B mix. The reduction in the peak intensity of hydration products is found in the XRD fingerprint of the 7.5T10S mix in Fig. 16d as compared to CM. The

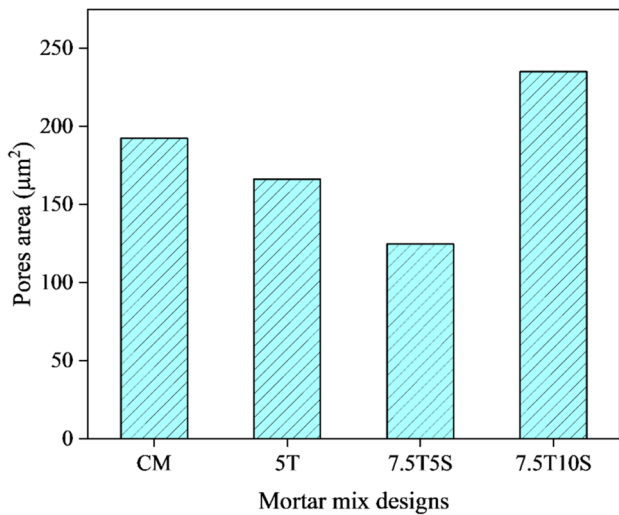


Fig. 15 The pore area of the mortar mixes based on SEM image analysis

replacement of cement with sludge and SBA in a higher considerable amount leads to lesser hydration products.

It is to be noted that the probable end use of the exhausted scrap materials such as sludge from water and wastewater treatment plants during reutilization processes are landfills, as sustainable materials in the construction industry, as activated carbons and energy recovery by various methods such as incineration, pyrolysis, gasification, and enhanced digestion using microbial fuel cell [42]. However, due to their hazardous nature, sludge, open dumping, incineration, and landfilling are not recommended for the textile industry [43]. The energy recovery is also difficult due to the presence of chemicals, heavy metals, and dyes in the sludge. The most sustainable method of stabilization of heavy metals from hazardous textile sludge is its use in the construction industry [8].

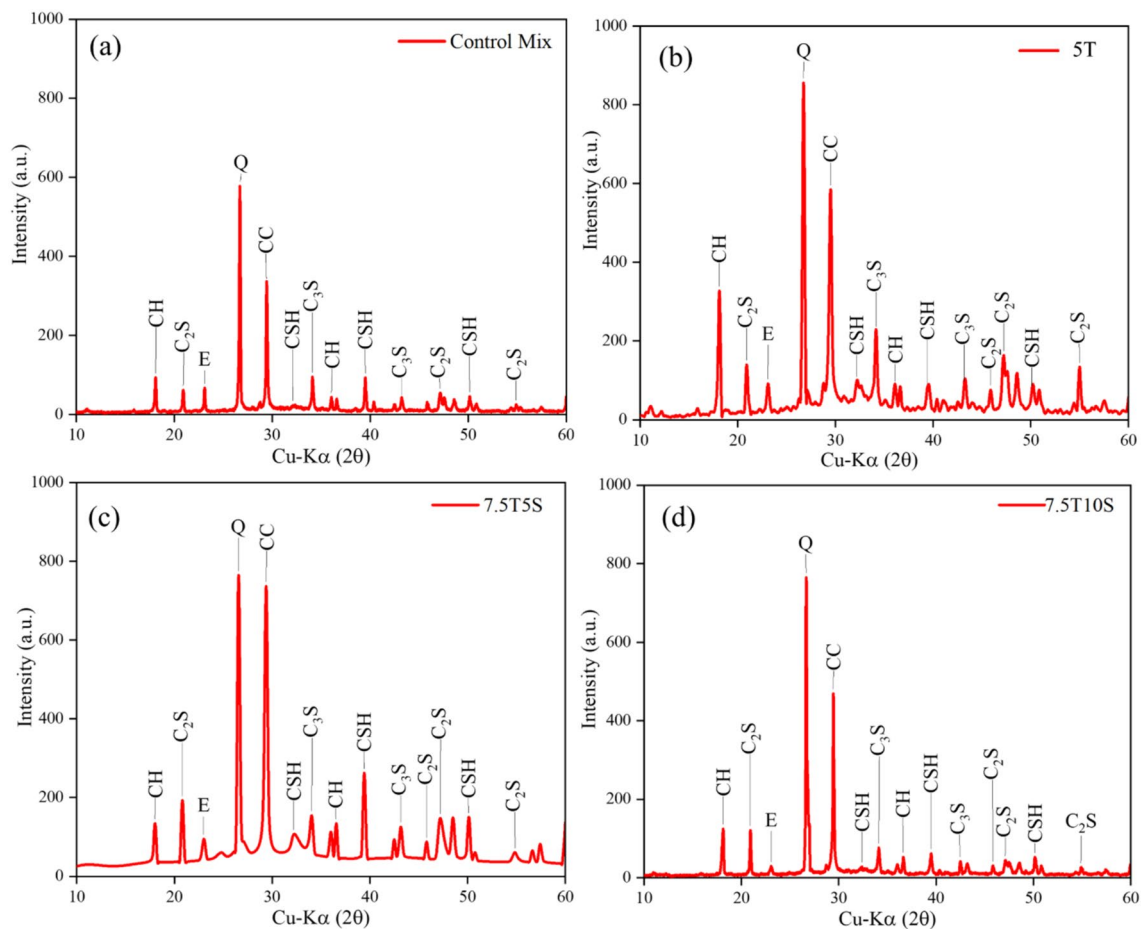
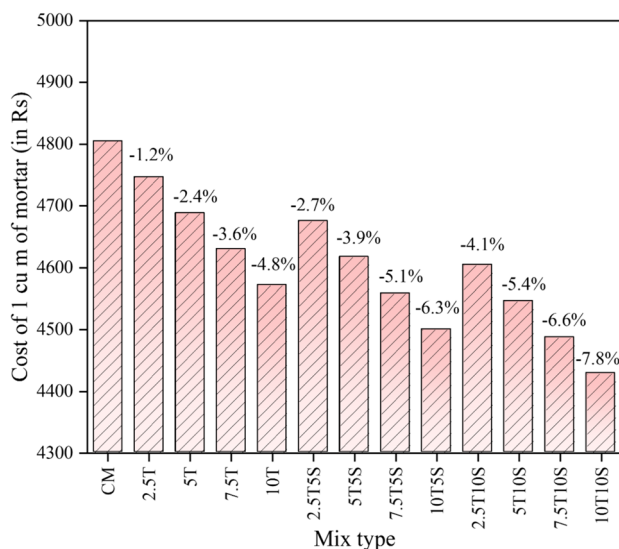


Fig. 16 XRD analysis of the mortar mixes **a** CM **b** 5T **c** 7.5T5S **d** 7.5T10S. CH calcium hydroxide, Q quartz, E ettringite, C_3S tricalcium silicate, C_2S dicalcium silicate, CSH calcium silicate hydrate

Table 6 Cost analysis of one cu m of mortar for the different mix combinations

Mix notations	Cement	Sand	TCETP sludge (transportation)	SBA (transportation)	Total cost of 1 cu m of mortar
CM	3978.45	826.26	0	0	4804.7
2.5T	3878.98	826.26	41.04	0	4746.3
5T	3779.51	826.26	82.12	0	4687.9
7.5T	3680.04	826.26	123.16	0	4629.5
10T	3580.5	826.26	164.2	0	4571
2.5T5S	3680.06	826.26	41.04	127.6	4675
5T5S	3580.57	826.26	82.12	127.6	4616.6
7.5T5S	3481.17	826.26	123.16	127.6	4558.2
10T5S	3381.63	826.26	164.2	127.6	4499.7
2.5T10S	3481.17	826.26	41.04	255.28	4603.8
5T10S	3381.7	826.26	82.12	255.28	4545.4
7.5T10S	3282.16	826.26	123.16	255.28	4486.9
10T10S	3182.76	826.26	164.2	255.28	4428.5

T Textile sludge, S SBA, c cement, w water, CM control mix

**Fig. 17** Cost reduction in percentage for the different modified mortar mixes

Cost estimation of the modified mortar mixes (economic viability)

The economic feasibility of incorporating TCETP sludge and SBA as a partial replacement for cement in mortar mixes is essential. The details of the cost estimate of all the modified mortar mixes and the control mix are presented in Table 6. The cost is calculated in (Rs) for the quantity of raw material required for making one cu m volume of mortar. The rates of each raw material have been taken from the scheduled rates followed in district Balotra, Rajasthan, India, for the year 2023. The transport distance for the TCETP sludge is considered 5 km, for SBA is 500 km, while cement,

sand, and water are considered to be locally available. The total expenditure for making one cu m for each modified mix is compared with the cost of the control mix.

From the cost analysis, the reduction in the cost of the modified mixes is observed with the partial replacement of cement by sludge and SBA in mortar mixes. In Fig. 17, relative reduction in the cost of modified mixes compared to control mixes is presented. The production cost for one cu m of modified mixes compared to the control mix has reduced by 1.2, 2.4, 3.6, and 4.8% for the binary mixes 2.5T, 5T, 7.5T, and 10T. For the tertiary mixes, the reduction in production cost of modified mixes is 2.7, 3.9, 5.1, 6.3, 4.1, 5.4, 6.6, and 7.8% for 2.5T5S, 5T5S, 7.5T5S, 10T5S, 2.5T10S, 5T10S, 7.5T10S, and 10T10S mixes, respectively.

Conclusion

Industrial waste management has become challenging for human beings. TCETP sludge and the SBA are industrial wastes and their stabilization and use as a resource could help in achieving sustainability goals. In the current study, TCETP sludge and SBA have partially replaced cement in mortar mixes and are investigated for strength, permeation, durability, carbonation, alkalinity, and leaching tests. 13 different mortar mixes are prepared for this study. Microstructural analysis is performed on the broken 28-day compressive test samples. The investigation reached the following specific conclusions:

- The strength of a few of the modified mortar mixes is comparable to the CM, and the later age strength (90 days) of the 2.5T, 5T, 5T5S, and 7.5T5S is equivalent to the CM. However, the compressive

strength of the mortar mixes declines with increasing sludge and SBA content such as in 7.5T10S, 10T10S mixes.

- The sorptivity test of the modified mortar mixes 2.5T5S, 5T5S, 7.5T5S, 2.5T, and 5T are less than the CM. It indicates the decline in capillary water absorption behavior and thus reduced pores.
- The durability test results using the MgSO_4 show contradictory results to compressive strength results as the mixes 2.5T, 5T, 2.5T5S, 5T5S, 7.5T5S, and 10T5S have higher loss of strength compared to other mixes.
- The pH of all the mortar mixes is between 12 and 13. The carbonation test shows that the mixes are carbonation free. The finding of the leaching test results indicates stabilization of sludge, and the presence of metals ions in the leachate for a few of the sludge and SBA blended mixes is below the admissible limit.
- SEM images demonstrate the densification of pores with the addition of sludge and SBA. However, the excessive formation of ettringite and large voids occurred at higher replacement levels. This also agrees with the conclusion drawn from the results of the compressive strength tests.
- The cost analysis indicates a maximum of 7.8% reduction in cost for the modified mix 10T10S.
- The suggested approach can be implemented in a real scenario even after considering administrative complexities. The Ministry of Environment, Forest and Climate Change, Government of India and the Central Pollution Control Board (CPCB), New Delhi could play a major role in the enforcement of policies such as the disposal of hazardous sludge at the Treatment, Storage, and Disposal Facilities (TSDFs). The government could also help strengthen the guidelines and develop systematic plans for the timely monitoring of effluent, sludge quantity, and quality. The government could keep checking on the expanding industrial clusters and illegal ways of disposal of sludge and effluent. Based on these efforts, alternative use of sludge, resource recovery (metal, dye recovery) could be planned.

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Authors' contributions SA: Conceptualization, Methodology, Data Collection, Analysis, Data Interpretation, Investigation and Modeling, Investigation, Analysis, Writing. APS: Conceptualization, Visualization, Modeling and Analysis, Supervision, Editing, Correspondence.

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Data availability All required data supporting the findings are available in the manuscript. If the readers require any additional data, the same would be shared electronically by the authors whenever required.

Declarations

Conflict of interest The authors declare that there are no personal relationships or known competing financial interests that could have influenced the work reported in this manuscript.

Consent for publication Not applicable.

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