Utilization of textile denim sludge waste in high load-bearing structural applications

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Abstract: The paper presents the results of research on the utilization of textile denim sludge waste, which was used to obtain concrete and mortar. For this purpose, cement and sand were replaced with the powdered sludge in the amount of 5 and 10 wt. The sludge was initially analyzed by FTIR and XRD to identify functional groups and chemical composition, respectively. Replacing sand with sludge resulted in a significant reduction in the concrete compressive and flexural strength (> 50%). Significantly better results were obtained by replacing cement with 5 wt% of sludge. In this case, the concrete compressive strength decreased by only 14%, and the flexural strength by 15%.

Keywords: textile composite, textile effluent sludge, concrete, mortar.

Wykorzystanie odpadów szlamowych z dżinsu tekstylnego w zastosowaniach konstrukcyjnych o dużej nośności

Streszczenie: W pracy przedstawiono wyniki badań dotyczące utylizacji odpadów szlamowych z dżinsu tekstylnego, których użyto do otrzymywania betonu i zaprawy murarskiej. W tym celu cement i piasek zastąpiono sproszkowanym szlamem w ilości odpowiednio 5 i 15% mas. Osad poddano wstępnej analizie za pomocą FTIR i XRD w celu zidentyfikowania odpowiednio grup funkcyjnych i składu chemicznego osadu. Zastąpienie piasku szlamem spowodowało znaczne zmniejszenie wytrzymałości betonu na ściskanie i zginanie (> 50%). Znacznie lepsze wyniki uzyskano zastepując cement szlamem. W tym przypadku wytrzymałość betonu na ściskanie zmniejszyła się tylko o 14%, a wytrzymałość na zginanie o 15%.

Słowa kluczowe: kompozyt tekstylny, tekstylny osad ściekowy, beton, zaprawa.

Pakistan has an enormous and potent textile sector. The sizeable amount of indigenous cotton fiber provides the country an economic edge. Denim has a significant share in clothing which is the largest portion of the industrial production. Denim answers the trends, that millions of hearts from traditional users to designers crave. Denim attracts the youthful, the stars, the businessmen, and the politicians, which is truly a cross-cultural trend across the very structure of the society not only the industry.

The fourth-largest cotton producer, third-largest in the consumer base, textile is the biggest industry of the country. Textile products contribute 8.5% of the economy gross domestic product. Pakistan stands 12th in the global textile export rankings. The total export of the country consists of 56% of the textile products and 38% of the country's whole industrial workforce is employed in the textile sector alone. Pakistan's textile industry, having 40 composite and 477 spinning units stands with strong 517 textile units countrywide. [1]. Pakistan is one of the major global partners with 28,500 shuttleless looms and 375,000 conventional looms [2]. The industry showed remarkable resilience during the Covid 19 pandemic through out 2020–21 period. The sector shows even growth and the volume of the export reached an all-time the highest number of $15.4 billion. [3]. Thanks to the increased production of denim products manufactured by Pakistan, the country has become one of the denim industry leaders within less than a decade, producing goods for the most famous and well-esTablished brands across the globe. This in turn is contributing significantly towards

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job opportunities and substantial export, leading to the investment of about PKR 100 billion in the denim sector. At present, Pakistan is one of the top three denim manufacturers in Asia. [4].

The monthly average of clothes exports from Pakistan was $565.60 million in 2021, which is expected to rise by 13.44% in 2022 to reach $641.60 million, according to a report. The US, the UK, Germany, Spain and France were the top importers of Pakistani clothes, accounted for approximately 68.27% of total clothes export of the country [5]. Generally, denim is heavy fabric made from 100% cotton and woven from coarse indigo dyed warp and grey un-dyed weft yarns. Traditionally produced denim is hard-wearing, high density fabric with high mass per unit area [4]. With an increase of 2% annually, Pakistan is currently producing 48.5 million tons of solid waste per year. The underdeveloped infrastructure and poorly constructed waste management system has lead to continuous rise in effluent in all forms. according to the estimation of Pakistani government, the solid waste amount generated by some of the metropolitan cities is approximately 87,000 tons per day. Karachi, being the busiest and largest metropolitan city of Pakistan, generates more than 13,500 tons of municipal waste daily. In this context, improper inner-city waste management, inadequate waste management equipment, lack of urban planning, bureaucracy and insufficient public awareness has led to the exponential increment in this problem [6].

The high amounts of various types of chemicals usage is a usual practice in order to impart specific properties and functionalities of textile materials such as softness, brightness, color, etc. This leads to the generation of huge amounts of hazardous waste water containing sand, grit, lint, volatile organic compounds, metals, oil and grease and several other types of physical and chemicals products. Denim production requires a large amount of water. Apart from growing cotton, most of the used water is discharged as wastewater after industrial processes such as indigo dyeing and fabric finishing [7]. Several phases are followed in order to carry out the treatment of textile wastewater. Initially, the particles found in wastewater are agglomerated using micro-emulsion by the process known as coagulation and flocculation, which is then followed by biological treatment of water. This process results in the formation of sludge where particles are made to increase their size. The treatment process that is based on ferric salts, bentonite clay and/or aluminum salts produces a lot of sludge. The produced sludge in addition to high amounts of chemical coagulant, has high amounts of pigments.

Nearly 5% of all the landfill space worldwide consists of textile, including denim as indicated by Environmental Protection Agency (EPA). Following this concern, the need of efficient recycling and upcycling technologies are necessary to recover the generated waste. These technologies enable to open new accesses and methodologies to engineer products and processes. According to recent estimates, around 2900 literes of water along with large amounts of utilities and chemicals are consumed to produce a pair of jeans. Using this information, we can get a clear idea of the alarming scenario created to meet the denim requirements throughout the world which contributes to let out massive amounts of wastewater and harmful gases to the environment by denim industry. The dyestuff consumed for denim is mostly the indigo dye which is an organic colorant, also used to dye leather, plastic and paper for various applications such as photochemical production, cosmetics, etc. The inclusion of these dyes to wastewater makes it extremely toxic and harmful if consumed by animals and humans which ultimately brings about disturbance of food chain and ecosystem. Because of the fact that most of the denim warp dyeing uses sulphur an indigo dyes, the environmental impacts of denim processing can be broadly classified in three main categories: air pollution as a result of cotton, abrasives, dust and volatile chemicals found in air; water pollution as a result of pretreatment, dyeing and finishing of textiles and solid waste (sludge).

It is a well known fact that denim washing is a process which is used to impart various properties and functionalities to denim, such as comfort, softness and most importantly a range of different looks such as abraded and worn-out looks. One method of denim washing includes the usage of pumice stone, where the stone gets abraded and ultimately transforms into the powdered form, a part of which remains in the washing liquid. Remaining part of this pumice stone gets stuck on the garment. A large amount of water is required to thoroughly wash off the remains of pumice stone deposited on denim. Another technique of garment washing, namely micro-sanding, also pollutes the environment. In case of chemical washing, various chemicals such as sodium hypochlorite and potassium permanganate make the effluent water having chlorinated organic substances, causing serious damages to environment and human body and health. Acid washing is the process which uses both pumice stone and oxidizing chemicals, which although requires less amount of water increases the presence of chemicals and stone particles in effluent. [8].

Material circularity is a concept which makes use of the wastes as raw materials for manufacturing new products which aids in shifting the conventional take-make-dispose linear economic model of material flow to a more viable environmentally friendly circular economy model. In recent times, the assessment and limitations of circular economy is gaining interest worldwide in the science, industry and government sectors. Minimization of raw materials and efficient utilization of energy have enforced the changes brought about in laws and policies of wastes management within the closed loop of production, consumption and disposal [9-11]. Energy from semi-solid sludge is biologically recovered by anaerobic digestion and nutrients such as phosphorus and nitrogen can be used as fertilizers [12,13]. In addition to this, the semi-solid sludge can be transformed into dry powdered form. This ash end product is produced after drying, carbonization, incineration or composting depending on the available wastewater treatment facility. This ash end product can be used as a raw material for various other applications [14,15].

Out of various types of building materials used worldwide, concrete hold the visibly high proportion because of its availability, ease of fabrication and versatile thermal, chemical, physical and mechanical properties. Concrete is a composite containing cement and aggregates (coarse and fine), which makes it very durable and able to endure various environmental impacts, with little or no maintenance costs [16,17]. Modern concretes are complex composite materials containing new components such as cement, aggregates (usually fine and coarse aggregates), water and chemical mixtures or mineral additions. In concrete structures, different types of waste are used as concrete additives to increase its durability, strength and fracture toughness. [18-21]. Because of the growing demand for concrete, research on supplementary cementitious materials (SCMs) has increased attention [22–24].

The compressive strength was reported to be dropped from 17 MPa to 7 MPa with the increasing amount of water-treatment plant sludge addition from 5% to 30% at 1000°C sintering temperature [25]. Another study showed the decrease in compressive strength of concrete when cement was replaced with dried sewage sludge [26]. Incorporating the waste polysilicon sludge in concrete up to 20% replacement level resulted in the increase in compressive strength after 28 days of curing period [27]. The amounts of both dry and wet waste water sludge can be used up to 15% as an additive without causing considerable reduction in concrete compressive strength, because of the fact that sludge acts as filler materials responsible for improving the mechanical properties of concrete mix [28]. For mortar cementous mixes, compressive strength of the modified mortar remains unchanged with fine sludge content up to 15%. [29]. 15% of textile sludge could be added in order to manufacture first class bricks, whereas 30% of textile sludge could be used to produce second class bricks as reported by Begum et al [30]. Sustainability of bricks using different quantities of textile sludge for structural and non-structural applications was further studied by Balasubramanium [31].

It had been stated that for load-bearing walls, the bricks were able to incorporate 10% textile sludge, whereas the higher amounts of inclusion of sludge (up to 30%) Chen and Wu concluded that approximately 20% of raw materials could use textile sludge on the attempt to produce the bricks using textile sludge, coal ash and ground soil [32]. Studies and experiments performed by Rehman et al. Revealed that the inclusion of textile sludge was limited to 5% and 20% for first grade and third grade bricks respectively, with the supplementary component of waste glass. For this purpose, no harmful implications were indicated by leaching tests in usage of textile sludge for engineering bricks [33]. The behavior of concrete made with the addition of ETP sludge in place of cement was also investigated by Arul et al [34]. This study showed that 20% replacement of cement with textile sludge indicated lower water absorption of concrete. Another research conducted to incorporate medical waste incineration ash indicated the decrease in compressive strength of concrete by 11.3MPa (29.7%) when cement was replaced with 50% of ash [35]. Considering its growing market, there is need of research in this area which should be extensively and dedicatedly conducted. The aim of this research is to identify the chemical characterization of Denim-based sludge, its identification with respect to structural applications and determination of powdered sludge optimum amount which can be added and partially replace the sand or cement and produce product with enhanced performance.

The study focuses on re-utilizing of denim hazardous sludge in structural application, *i.e.* concrete and mortar, which can prove to be a breakthrough in achievement of sustainability paradigm by circular economy. A leading denim industry is taken as a source of sludge which would be utilized for the aforesaid purposes. This sludge can be formed either by biological or chemical treatment. The chemical composition and characteristics would be different for types of sludge prepared with each respective process. In biological treatment, the sludge is treated with bacteria through aeration. In chemical treatment the sludge is treated with different chemicals and then dewatered through screw press. Combined sludge was obtained from the combined chemical and biological treatment of the denim-based waste in the effluent treatment plant. The aim of this study is to utilize the denim sludge to replace cement and fine aggregate in concrete and mortar at different percentages (5% and 10%). Compressive strength and flexural strength were evaluated accordingly to measure the performance of mix design incorporating the sludge in concrete and mortar.

EXPERIMENTAL PART

Materials

For the purpose of manufacturing the structured samples for mechanical testing, cement, sand, dried powdered sludge and aggregates were evaluated and utilized.

Cement

53-Grade Ordinary Portland Cement (OPC) confirming to IS Code – IS 12269: 1987, was used in the study. The physical properties of cement are shown in Table 1. In brick manufacturing, the identification of important mineral/crystalline phases in the raw material and the final product represents an important aspect. In this case, X-ray diffraction (XRD) tests can provide accurate infor-

Table 1. Physical properties of ordinary portland cement

Properties	Values
Average compressive strength, MPa (28 days setting time)	33
Fineness, m^2/kg.min	225
Durability (by Le Chatelier's method)	10
Durability by autoclave method	0.8
Initial setting time, min	60
Final setting time, min	500
Specific gravity, g/cm^3	3.15

Table 2. Qualitative analysis of denim-based sludge and cement through XRD

Compounds/elements identified in sludge	Compounds/elements identified in cement
Ferric oxide (Fe_2O_3)	Dicalcium Silicate
Aluminum oxide (Al_2O_3)	Tricalcium Silicate
Silicon dioxide (SiO_2)	Gypsum
Calcium oxide (CaO)	Silicon dioxide (SiO_2)
Magnesium oxide (MgO)	Magnesium oxide (MgO)
Titanium dioxide (TiO_2)	Calcium oxide (CaO)

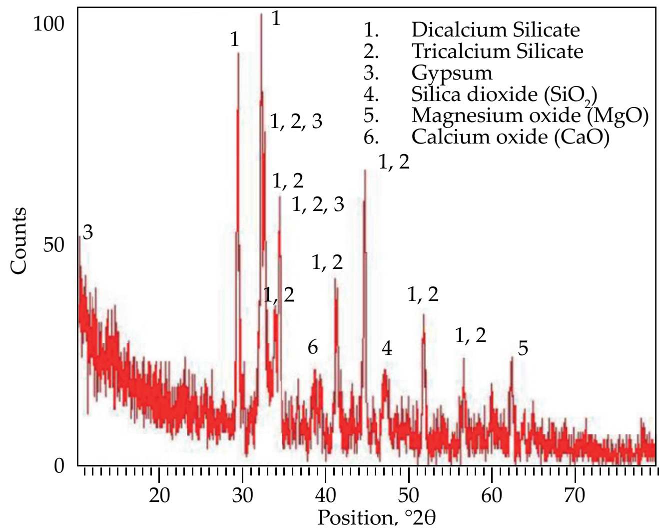

Fig. 1. XRD of cement

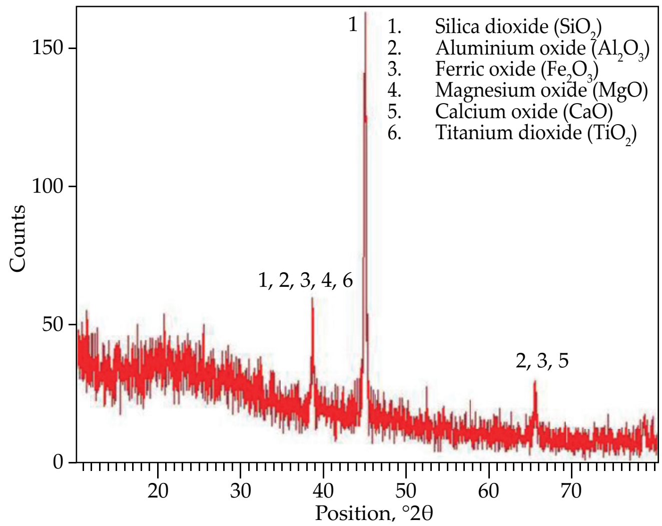

Fig. 2. XRD of denim sludge

mation about the phases of the crystal and the actual raw materials of the bricks. The XRD pattern and qualitative analysis of compounds found in cement along with their composition are shown in Fig. 1 and Table 2, respectively.

Aggregate

The aggregates used in this study were crushed stone and sand. The specific gravity of used sand is 2.65 and water absorption is 0.50%. The crushed stone was ranging from 3/8 and 1.5 inches in diameter, with specific gravity of 2.7 and water absorption of 1.17%. Sieve sizes of #4 and #200 were used for coarse and fine aggregate respectively. The percentage retained and passed is specified in Table 3.

Sludge

The sludge was collected from a mass-production leading textile denim industry of Pakistan. Effluent Treatment Plant of this industry located in Karachi, has 2273m^3 storing machine to convert it into fine, powdered form for the usage in concrete and mortar samples. The study used the ground sludge passing Mesh no. 25 (aperture of 600 μm). XRD analysis was carried out to reveal the chemical composition of the sludge. Fig. 2 below demonstrates the results.

Characterization

The X-ray diffraction pattern of the powdered-based sludge samples was recorded using an X-ray diffractometer. The XRD analysis of the samples was performed using a scan speed of 1° min^{-1} with a step difference of 0.02″ in the 2θ range between 10° and 60°. XRD patterns reveal the presence of main compounds found the sludge sample which are Fe_2O_3, Al_2O_3, SiO_2, CaO, TiO_2, MnO and some heavy metals. These elements and compounds are, to some extent, similar in XRD pattern of cement. This provided a sign of possibility for using sludge in replacement with cement for constructional samples (concrete

Table 3. Sieve size designation for aggregates

Aggregates	Sieve designation	% weight retained	% weight passed
Coarse Aggregate	#4	7.2	92.8
Fine Aggregate	#200	99.3	0.1

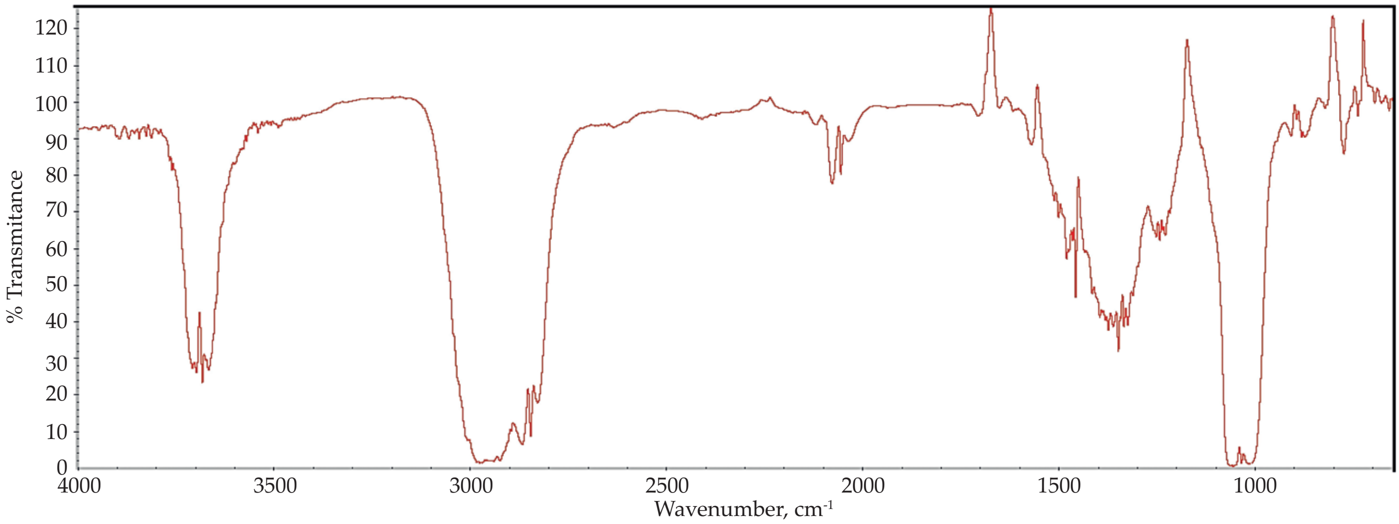

Fig. 3. FTIR of sludge

and mortar); however, additional work was done to replace fine aggregate with sludge in concrete.

In addition to the above, FTIR of sludge was performed to identify the presence of functional groups on the surface of the materials, which permitted further observations and understanding about surface features of the raw material. Fig. 3 represents the spectrum of the sludge presenting its peak at 3650 cm^{-1}, which could be assigned to hydroxyl groups present in smectite and bentonite. Also, the peak at 1437 cm^{-1} could be attributed to the symmetrical and asymmetric modes of the $(CO_3)^{-2}$ vibration. The results confirm the presence of carbonates that is in agreement with the previous XRD analysis. The peak at the region between 1000 and 909 cm^{-1} could be assigned to the SiO_2 stretching modes that come from the aluminosilicates in sludge sample [36].

Physical properties

The pH of textile denim-based sludge is found to be slightly alkaline with value of 8. Vicat test was done to determine the consistency and settling time of sludge, to decide further whether it can be used as replacement of cement. Results show that with the increasing percentage of sludge, the consistency of paste increases due to the hygroscopic nature of sludge. The results of test are shown in Tables 4 and 5.

Methods and sample preparation

The mechanical properties of concrete having sludge as a substitute of sand were evaluated by performing tests on cylindrical specimens and prisms. For compressive and split testing, cylinders having a diameter of 4 inches and height of 8 inches were used. For flexural testing, prisms having cross-section of 3 × 3 inches and length of 12 inches were used. Cylindrical specimens for compressive strength in Fig. 4 and prism specimens for flexural strength displayed in Fig. 5 were manufactured according to ASTM standards ASTM C293-02 and ASTM C109/C109M-20, respectively. Cube specimens for mortar testing were manufactured according to ASTM C109/C109M-20, as shown in Fig. 6, 3 samples of all different forms were manufactured and tested accordingly which is also depicted in Fig. 7.

Concrete

The materials used to manufacture concrete specimen for mechanical testing included the powdered sludge,

T a b l e 4. Comparison of sludge replaced samples consistency with standard

Sample	Water requirement, %	Plunger penetration, mm
Standard	27	10
5% sludge	32	10
10% sludge	37	9

T a b l e 5. Comparison of standard settling time with 5% and 10% sludge replacement

Sample	Initial time	Final time	Total
Standard	10:25 am	12:40 pm	135 minutes
5% sludge	11:00 am	12:25 pm	85 minutes
10% sludge	11:00 am	11:30 am	30 minutes

Fig. 4. Cylinder samples for compressive strength test of concrete

Fig. 5. Prisms samples for flexural strength test of concrete

Fig. 6. Cube samples for compressive strength test of mortar

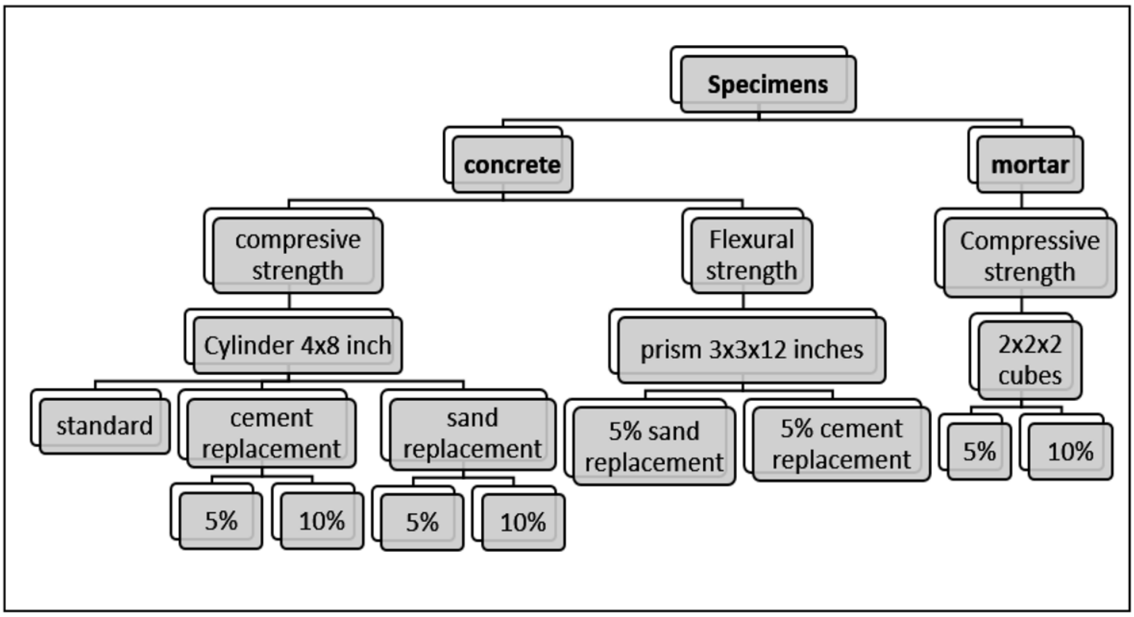

Fig. 7. Number and forms of specimens casted for testing

T a b l e 6. Mix proportions of concrete specimens

Building material type	% of cement/ clay substituted with sludge	Cement kg	Water/ cement ratio	Sludge kg	Coarse aggregate kg	Fine aggregate kg	Water kg
Cylinder	Standard						
	–	0.593	0.535:1	–	1.495	1.437	0.319
	Cement replacement						
	5	0.563	0.535:1	0.0296	1.495	1.437	0.319
	10	0.5333	0.535:1	0.0593	1.495	1.437	0.319
	Sand replacement						
	5	0.593	0.535:1	0.0718	1.495	1.365	0.319
	10	0.593	0.535:1	0.1437	1.495	1.2933	0.319
Prism	Standard						
	N/A	0.637	0.535:1	–	1.605	1.543	0.342
	Cement replacement						
	5	0.605	0.535:1	0.0318	1.605	1.543	0.342
	Sand replacement						
	5	0.637	0.535:1	0.077	1.605	1.465	0.342

Portland cement, water, coarse aggregate and fine aggregate. A concrete pan mixer was used to mix the sludge, cement, and aggregate such as sand or gravel, and water homogeneously to form concrete. Tamping rod type of vibrators were used to avoid the formation of air voids in the concrete specimen. Finally, the specimens were created in molds to achieve the desired shape and size. Trowels were used at the end to even out the samples from top of the mold.

As per IS456 code book, in mild exposure condition w/c ratio for normal mix of M20 concrete is about 0.55. The ratio between cement, Fine Aggregate and Coarse Aggregate was 1:2.4:2.5 respectively. For concrete, the method and proportions of ingredients chosen for casting samples are shown in Table 6.

Mortar

A type M mortar with water/cement/sand ratio of 1:2:5.5 was casted in this study. Table 7 represents the casting method and mix proportions of mortar by replacement of cement with 5 and 10% powdered sludge. Specimens were then cured for 28 days at room temperature and corresponding tests were conducted on samples accordingly.

RESULTS AND DISCUSSION

Mechanical properties

Compression testing on mortar specimen cubes of material with 2 inches on each side was carried out based on ASTM C109 standard as represented in Fig. 8. Fig. 9 shows compression testing performed on the concrete cylinder specimens, which was carried out based on ASTM C39 standard and Fig. 10 shows the flexural strength test performed on concrete specimen.

The compressive strength of mortar cubes was reduced with the increase in replacement ratio of cement with the TES. Average compressive strength of standard mortar cubes is 17.01 MPa. Average Compressive strength of mortar cubes at 5% replacement of cement by sludge is 10.98 MPa that is 35% reduction in compressive strength, by further increasing percentage replacement of cement with sludge to 10% average compressive strength of mortar cubes reduces to 9.63 Mpa, i.e. 43% reduction in compressive strength, as indicated graphically in Fig. 11.

Compressive tests which were performed on concrete specimens with 5% and 10% replacement of cement with sludge and fine aggregate (sand) with sludge revealed the

T a b l e 7. Mix proportions of mortar specimens

% of cement/clay substituted with sludge	Cement kg	Water/cement ratio	Sludge kg	Fine aggregate kg	Water kg
N/A	0.08230	0.5:1	N/A	0.226	0.03993
5	0.07819	0.5:1	0.00411	0.226	0.03993
10	0.07407	0.5:1	0.00823	0.226	0.03993

Fig. 8. Mortar cubes for compression testing

Fig. 9. Concrete cylinder for compression testing

Fig. 10. Concrete prism for flexural testing

average compressive strength of standard specimens to be 19.54 MPa, illustrated in Fig. 12.

The replacement of sand with sludge, demonstrates drastic reduction in strength i.e. by replacing 5% of sand with sludge the compressive strength reduces to 9.46 MPa that is 51% reduction in compressive strength. With further increasing the sludge content to 10%, the compressive strength additionally reduces to 5.55 MPa that is 71% reduction from standard compressive strength. Better results were observed by the replacement of cement as compared to those obtained by replacement of fine aggregate (sand). By replacing 5% of cement with sludge, the compressive strength reduces to 16.8 MPa that is 14% reduction of compressive strength with the standard value. At 10% sludge proportion, the strength reduces to 13.57 MPa that is 30% reduction in compressive strength with the standard specimen. Minimum reduction of compressive strength in concrete cylinders is achieved by 5% replacement of cement by sludge that is 14%, as compared to the strength of standard cylinders.

Flexural test on concrete (ASTM C293) results reveal huge reduction in strength of concrete prisms by 10% replacement of cement and sand with sludge, therefore, only 5% of cement and sand were replaced by sludge. Flexural strength of standard prisms was found to be 6.2 MPa. By replacing 5% of cement with sludge flexural strength reduces to 5.23 MPa that is 15% reduction, while by replacing 5% of sand with sludge flexural strength reduces to 3.23 MPa that is 48% reduction with the flexural strength of standard prism sample, presnted graphically in Fig. 13.

Cost analysis

The average sludge density is 1000 kg/m^3, therefore 1 m^3 of sludge contains appoximately 1000 kg of wet sludge. Theis concludes that 1000 kg of wet sludge has 10% of moisture content. Using this information, the summary

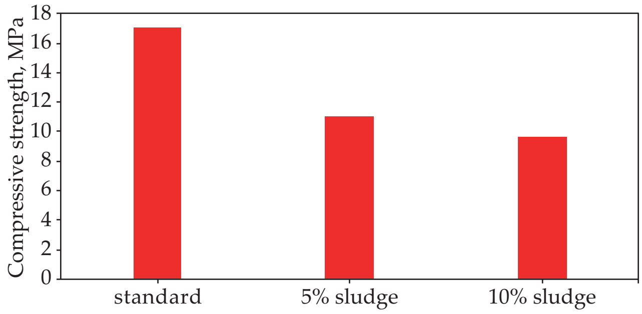

Fig. 11. Compressive strength of mortar

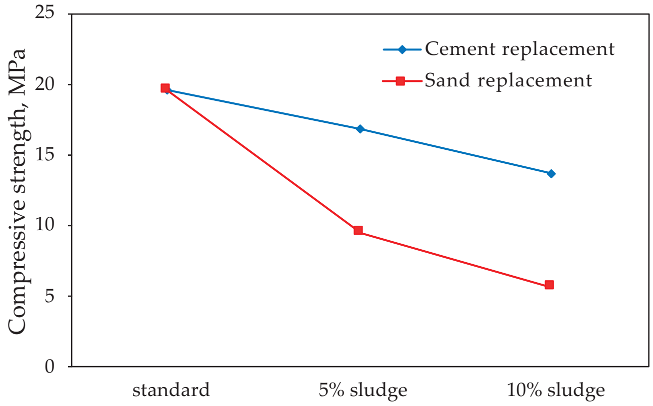

Fig. 12. Concrete compressive strenght with replacement of cement and sand

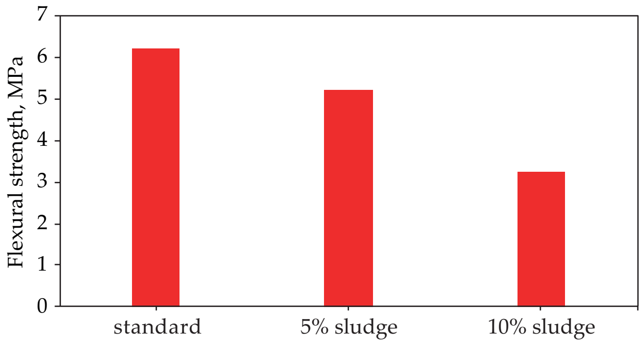

Fig. 13. Flexural strength of concrete

T a b l e 8. Cost analysis of dry sludge processing

Parameter	Cost (PKR)
Cement	610,000/ton 610/kg
Transposttaion	38,125/m^3
Purcahsing sludge	15,250/m^3
Sludge drying (200°C; 4 hrs)	380,000/m^3
Semi-solid sludge	53.37/kg
Dry sludge	486.745/kg

of the approximate cost comparison between cement and semi-solid sludge is provided in Table 8. Accordingly, the comparison implies that using dry sludge with 5% of water content will reduce the overall concrete cost by approximately 21%, which would bring great benefit.

CONCLUSIONS

The addition of sludge in mortar and concrete delays the cement setting process and reduces compressive and flexural strength of the materials. With the increment in percentage of ETP sludge (5% and 10%) the compressive strength of the concrete decreases. Moreover, it was found that replacing the fine aggregate with sludge is not a viable option as it resulted in significant decrement in compressive strength of concrete. It would be more suitable to replace cement with sludge without major reduction in strength.

It was found that the inclusion of the sludge in the composition also increases water absorption of the mortar and concrete.

The mechanical properties of the sludge-based mortar and concrete were found to meet the ASTM standards requirements for non-structural materials, indicating that the denim-based textile ETP sludge can be used for making non-structural building components where lower strength is permissible.

Pre-treating dried sludge with lime may remove the elements resulting in reduced strength of structures. On the other hand, this sludge can be utilized in the areas where strength is not higly required, such as, hollow block concrete wall, brick masonry wall, pavement block, screed, leveling, cement concrete flooring tiles etc.

Utilization of textile ETP sludge is expected to minimize the environmental impact. The carbon dioxide emitted by ordinary world production of Portland cement corresponds to approximately 7% of the total emissions of greenhouse gases into the atmosphere, so if textile sludge could be used in cement production, it would reduce the CO_2 emission which would be cost effective and eco-friendly.

It is also important that there will be a considerable reduction in the cost of concrete and mortar if cement is replaced by textile denim sludge.

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