INVESTIGATING THE EFFECT OF SLUDGE/CLAY RATIO AND FIRING TEMPERATURE ON THE COMPRESSION STRENGTH AND WATER ABSORPTION CAPACITY OF BRICK USING RESPONSE SURFACE METHODOLOGY

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ABSTRACT

Presently, the sludge generated from the Zero Liquid Discharge (ZLD) facility in Hawassa Industrial Park is collected inside a nearby big constructed shed without any treatment. This study investigated the utilization of this textile sludge as input for the production of brick construction material as the sludge management tool. The study focused on finding optimum parameters for the production of bricks using response surface methodology. The proportion of sludge mix and the firing temperature were selected as the study independent parameters with the compressive strength and water absorption as the experimental responses. Based on the analysis of variance (ANOVA), linear and quadratic model equations were selected for compressive strength and water absorption capacity, respectively. The regression analyses have shown that the model equations ware satisfactory enough to predict the selected responses within the experimental range with 94.98 % and 99.71 % variability in the same order. Based on numerical optimization, a sludge mix of 18.66 % and a temperature of 1000^{o}C were selected as optimum synthesis parameters predicting bricks with 3.5 MPa comprehensive strength and 16 % water absorption.

Keywords: Statistical Optimization, textile sludge, bricks, zero liquid discharge.

1. INTRODUCTION

The world's ever-increasing population and the progressive adoption of industrial-based lifestyle have inevitably led to an increased anthropogenic impact on the biosphere [1]. Generally speaking, environmental pollution is a serious global environmental issue due to an unavoidable consequence of economic development and people's desire to improve their standard of living [2]. Among the worldwide environmental problems, sludge generated from industrial wastewater treatment plants and dumped as worthless material is one of the major issues.

Industries have a decisive role in the economic development of a country. This can be seen from two main perspectives. Primarily, they employ a significant number of people and create a good opportunity for knowledge as well as technology transfer. Secondly, they generate a high amount of foreign exchange. More specifically, the textile industry is one of the prominent industries that plays a crucial role in the development of a country as far as it produces one of the basic needs of human beings - clothe materials.

The textile industry is both water and chemicals intensive in its manufacturing and treatment processes [3]. As a result, it discharges wastewater effluents containing various pollutants. For purifying such waste water to an allowable discharge limit, various treatment processes that use conventional or zero liquid discharge technologies are employed depending on the wastewater characteristics. In both ways, a large quantity of semisolid by-product known as sludge is generated from the treatment plant. The quality and quantity of the sludge produced depend upon the amount of wastewater and the type of treatment adopted for treating the wastewater [4].

Recently, several industrial parks which use common zero liquid discharge (ZLD) facility for the treatment of the wastewater are built in Ethiopia. However, the management or recycling of the sludge discharged from the treatment plants is not considered so far. Presently, Ethiopia has inaugurated the largest industrial park in Africa named Hawassa Industrial Park aiming to produce textile and garment products. The Industrial Park uses an advanced wastewater treatment plant, the so-called ZLD facility, aiming to recycle 90 % of disposed water fulfilling international standards. However, the main problem encountered in the industrial park is the sludge disposal. The sludge generated from the ZLD facility is collected inside a nearby big constructed shed without any treatment. Since the sludge contains the chemicals removed by the purification processes, it is considered to be a non-biodegradable waste material [5].

The disposal of sludge is an inescapable by-product of textile wastewater treatment processes due to the use of various chemicals like dyes, pigments, and other complex compounds used in the manufacturing and treatment processes [3]. For this reason, it includes a cluster of organic and inorganic compounds with high concentrations of heavy metals such as Fe, Cu, Cd, Zn, Pb, Cr, etc. It also contains obnoxious odor leading to potential health and environmental threats. Compositions of sludge vary considerably depending on the wastewater composition and the treatment processes used [6].As the magnitude of the sludge produced increases, the sludge disposal problem will be escalated exorbitantly [7].

There are different sludge management methods that are in use to date. These include use of the sludge as fertilizer for agriculture, incineration, land filling, mixing with cement, as a substrate for biogas generation, as an additive in construction materials such as concrete, bricks, tiles, etc. [8]. The conventional management methods like land filling and incineration may not be suitable. Landfill disposal of the sludge has drawbacks like high cost of transportation, difficulty in getting suitable sites for land filling, heavy metal contamination of the land, emission of foul gases, etc., [9]. The residues from the sludge incinerators can also induce secondary pollution [10]. The use of textile sludge in construction materials could serve as an alternative solution to disposal and reduce pollution provided that it satisfies the engineering properties of building materials and leaches toxic metals to a permissible limit [11]. Thus, this mechanism is a win-win strategy for the reason that it not only converts the wastes into useful construction materials but also improves the environmental quality [12]. This is owing to the destruction of the toxic elements and oxidation of organic matter in the sludge while at the same time eliminating pathogens during firing [13]. Some studies have revealed that during the firing process, heavy metals’ transformation may take place significantly reducing their concentration [14]. However, the amount of textile sludge used as a partial substitute in the production of construction materials such as bricks directly affects the quality of the final product. Furthermore, the production of textile sludge-based bricks mainly depends on the percentage of sludge mix and firing temperature. Therefore, in order to get a good quality of product in terms of mechanical strength and water absorption, the two parameters need to be optimized.

In different preceding studies, these operational parameters have been investigated using the "one-factor-at-a-time approach". Although such kind of approach is a common and acceptable approach, it lacks in estimating the interaction effects between the factors and predictive capability [15].

This study has attempted to statistically optimize the sludge mixing proportion and firing temperature to the responses: suitability of mechanical strength and water absorption, using the central composite design (CCD) response surface methodology (RSM), Flowingly, brick samples were synthesized at the optimum statistically optimized condition and leaching tests were carried out to check the possibility of leaching of heavy metals to the environment.

2. MATERIALS AND METHODS

2.1 Raw materials

The textile sludge employed in the present study was collected from Hawassa industrial park Zero liquid discharge facility, Hawassa, Southern part of Ethiopia. The sludge was collected in the form of a semi-liquid by-product.

The clays (Red + White) and sand were collected from Ethio bricks factory, Addis Ababa, Ethiopia and nearby construction site, respectively. Red clay has relatively high amount of iron oxide, and a small amount of magnesia, but the white clay has small amount of iron oxide.

2.2 Design of experiment

The statistical optimization of synthesis parameters is used to determine the best condition of the selected operational factors that can be suitable to produce the best quality of final product bricks in terms of mechanical strength and water absorption. The experiments were designed based on the central composite design (CCD) of the response surface methodology (RSM). To examine the effects of synthesis parameters on the mechanical strength and water absorption, two important factors were selected and designated as A (percentage of sludge mix) and B (firing temperature). The selected factors and their levels are given in Table 1.

Based on RSM-CCD a total number of 13 experiments (8 factor and 5 center points) were determined using the following formula.

$$\mathrm{N} = 2^{\mathrm{k}} + 2\mathrm{k} + \mathrm{C} \qquad (1)$$

where N is the number of the experimental run, k is number of factors, 2^k points are in the corners, 2k axial points and C is a central replication point [16]. Table 2 shows all experimental points which are arranged according to RSM-CCD.

A second-order polynomial equation was used to find the interaction effects between the study factor and pinpoint the optimum synthesis parameter on the given response. For the selected two factors, the general equation can be expressed as:

$$Y = \beta o + \beta_1 A + \beta_2 B + \beta_{12} AB + \beta_{11} A^2 + \beta_{22} B^2 + \varepsilon \qquad (2)$$

where Y is the response factor which maybe mechanical strength or water absorption, βo is a constant term, A is sludge mix, B is the firing temperature, β_{is} are the coefficients for linear factor, β_{ij}s are the coefficients for cross-product interaction factors, β_{ii}s are the coefficients for quadratic factors and ε is the random error [17].

Table 1 Selected Experimental factors and levels

Designation	Factors	Unit	Levels		
			Low (-1)	Medium (0)	High (+)
A	Sludge Mix	%	10	15	20
B	Firing temperature	^{o}C	800	900	1000

The regression analysis and estimation of these coefficients on experimental data were performed using statistical software package Design-Expert® version 10.0.1.0 (Stat-Ease, Inc. 2016). Analysis of variance (ANOVA) was employed to evaluate the adequacy of the model equations. The goodness of fit of the model equations was expressed using a coefficient of determination (R^2), prediction coefficients of determination (Pred R^2), and adjusted coefficients of determination (adj-R^2). Furthermore, their statistical significance was evaluated based on the F-test, P-value, and coefficients of variation (CV)

2.3 Brick preparation

The textile sludge sample was kept in a refrigerator at 4℃ before use. It was then dried in an oven at 105℃ for 24h, was crushed to powder and sieved to less than 1 mm. Then, the sample was packed in airtight sealed polyethylene bags until further analysis and preparation of bricks. For the preparation of sludge-based bricks, the proportion of sludge mixing was varied from 10 to 20 % by dry weight and firing temperature from 800 to 1000^{o}C as indicated in Table 2. The textile sludge was used to replace both clays' proportions which are practiced in commercial brick factories i.e., the ratio of three white soil to one red soil.

Primarily, dry mixing of the aforementioned raw materials was done for proper homogenization. Thereafter, the mixture was mixed properly by adding a sufficient amount of water. Finally, it was transferred into an oil-lubricated mold to obtain the desired shape. Mold size of 10 x 10 x 5 cm^3 was used to shape the mixed materials into bricks. The molded brick specimens were then kept inside the laboratory (20^{o}C and % 51.1relative humidity) for a week and 2 days for uniform drying.

Before the firing process began, most of the water was evaporated in this process aiming to prevent cracking. After nine days of the air-drying period, the bricks were ready for firing. The dried bricks were fired in a muffle furnace (Nabertherm, LH 60/40, Germany) at temperatures of 800, 900 and 1000^{o}C, respectively. The sintering process was performed for 3 h with different heating rates corresponding with the temperature. Consequently, the bricks were allowed to cool naturally and transferred for physical and mechanical property analysis test.

2.4 Characterization of textile sludge

The textile sludge used in this study was characterized by various parameters. The organic content, proximate components, heavy metals and chemical constituents were determined as per by standard APHA method (APHA, 1998), ASTM and AOAC (2000), respectively. The pH and calorific value were determined using digital pH meter and adiabatic oxygen bomb calorimeter.

The proximate analysis includes the determination of moisture content, volatile organic matter, fixed carbon and ash content present in the sludge sample. The moisture content was determined by drying a weighed quantity of the sludge sample in an oven at 105℃ for 24 hours and taking the difference. It was determined according to the APHA method (APHA 2540 G). The amount of VOC was determined by igniting the oven-dried textile sludge sample in a muffle furnace according to ASTM (ASTM D 3175).

The ash content was determined by burning the sample in a muffle furnace according to AOAC (2000) using the official method 923.03. The percentage of fixed carbon was determined directly by deducting the sum of total volatile matter and ash percentage from 100. Furthermore, the extent of organic compounds was evaluated by chemical oxygen demand (COD), biological oxygen demand (BOD), and total organic carbon (TOC) test. More specifically, the BOD was determined by the BOD apparatus and using analytical reagents. The formula used to compute the BOD_5 was taken from the difference of initial and final dissolved oxygen according to standard methods for the examination of wastewater (APHA 5210 B). COD determination was used to measure the organic and inorganic matter content by digesting it in a COD reactor followed by spectrophotometer measurement. The total organic carbon (TOC) measurement was also performed using TOC analyzer (Shimadzu TOX-5000). In the same manner, the heavy metal analysis for Cr, Cd, Mg, Ca, Mn, Pb, Fe, Cu, Ni, and Zn were conducted using flame Atomic Absorption Spectrophotometer (PG 990, FAAS Hydride and cold Hg technique, Germany) by applying digestion of the textile sludge before investigation. Lastly, to determine the chemical constituents in the textile sludge X-ray fluorescence (Thermo Fisher ARL 9900, United States) and chemical analysis (ASTM C 114/00) methods were used.

The produced sample bricks were then tested for compressive strength and water absorption as per standards. Since the prime objective of this research was to use textile sludge waste as a resource material for making bricks, it was necessary to check the conformity of the product properties to standard values. In this regard, the compressive strength and water absorption are the most important properties that are used to determine the quality of building materials [10,18]. Accordingly, the compressive strength test was conducted using a compression testing machine (CONTROLS, USA) according to ASTM. The water absorption was measured from the ratio of the weight difference between the amount of water embedded by the brick after a 24h submersion of the test brick in water and dry weight of the unit over its dry weight.

2.5 Leachate test of the bricks

Leachate analysis test is highly important to know the concentration of the toxic heavy metals leached from the textile sludge-based bricks. The test was performed based on the procedure described by Xu et al. (2014) with minor modifications [19]. Accordingly, 10 g of brick powder (100 mesh, dry weight, precision 1 mg) and deionized water, with a powder-to-water ratio of 1:30, were mixed and stirred for 10 min, and, then, allowed to sit statically for 24 h in a conical flask with continuous stirring for 10 min every 8 h. Then, the leachate was separated from the residue by filtering through a filter paper and transferred into a 500-ml volumetric flask and diluted to the mark with deionized water. Finally, the heavy metals were measured by Inductively Coupled Plasma (ICP-OES) examination method.

3. RESULTS AND DISCUSSION

3.1 Characteristics of the textile sludge

Table 3 shows the overall physic-chemical characteristics of textile sludge. The textile sludge was alkaline with pH range of 8.1 – 8.4 with a mean value of 8.2. Previous studies have also reported pH values in the range of 7.8 to 9 similar to the present findings [12,20]. Such pH values may be due to the addition of coagulating chemicals during the pre-treatment stage of textile wastewater.

The heating value of the textile sludge was found to be 7867.26 kJ/kg. The calorific value of the present study doesn't show a significant variation as compared to the result reported in different literatures. This might be due to the existence of a high level of organic chemical compounds in the sludge. Such calorific value can be related high carbon content which may reduce the strengths of concrete and increase the shrinkage upon drying.

Table 2 Central composite design experimental points with actual and predicted responses

Factors		Responses			
A: Firing Temperature(°C)	B: Sludge Mix (%)	Compressive strength (N/mm^2)		Water absorption (%)	
		Actual value	Predicted value	Actual value	Predicted value
900	15	3.71	3.63	14.9	14.77
900	15	3.57	3.63	14.56	14.77
1000	20	3.4	3.37	17.1	17.26
900	15	3.66	3.63	14.81	14.77
1000	10	4.2	4.32	12.4	12.36
900	10	4.17	4.10	13.22	13.38
900	20	3.03	3.15	18.98	18.74
900	15	3.67	3.63	14.72	14.77
800	20	2.93	2.94	21.33	21.41
1000	15	3.97	3.85	13.64	13.52
900	15	3.52	3.63	14.78	14.77
800	10	3.83	3.88	15.7	15.58
800	15	3.5	3.41	17.17	17.21

The results obtained for BOD, COD, and TOC were 535 mg/l, 9820 mg/l, and 51.3 %, respectively. These indicate that the textile sludge has a high amount of organic and inorganic pollutants which may be attributed to the addition of various chemicals in the production process as well as in the wastewater treatment stage. Patel & Pandey (2017) have reported the value of total organic carbon (TOC) in the range from 1.23 % to 17.82% [12]. However, this shows that the TOC found in our study is much higher than that reported above and this difference may be due to the difference in chemical doses and treatment processes practiced from industry to industry. The COD and BOD values were also found a bit higher as compared to the allowable discharge limits indicated in the Ethiopian provisional standards for industrial pollution control.

The proximate analysis results are given in Table 3. The results show that the moisture, ash, organic matter, and fixed carbon contents of the textile sludge were 82.5%, 44%, 48.4%, and 7.6%, respectively. This result shows that the sludge released from the ZLD facility has a high amount of moisture, ash and organic content (volatile matter) but with low amount of fixed carbon.

The investigation of the inorganic elements content of the textile sludge sample, particularly the heavy metals is also reported in the same table. The results show that iron is found as the leading constituent and the most harmful metals Cd and Ni are below discharging permissible limits by USEPA. The high concentration of iron could be owed to the addition of chemical coagulants such as iron chloride in the treatment process. The concentration of lead is also below the regulatory limits of the provisional standards for industrial pollution control in Ethiopia. On the contrary, among the toxic heavy metals that commonly exist in textile sludge, only Cu, Zn and Cr were found to be a little bit above the permissible limit values.

Table 3 Physico-chemical characteristics of textile sludge sample

physic-chemical analysis	Results
pH	8.2
Calorific value (cal/g)	1880.32
BOD (mg/l)	535
COD (mg/l)	9820
TOC (%)	51.3
Proximate analysis (%)	
Moisture content	82.5
Ash	44
Volatile organic matter	48.4
Fixed carbon	7.6
Heavy Metals (mg/kg)	
Cu	5.23
Zn	30.45
Fe	323.63
Pb	2.25
SiO_2	279.60
Ca	0.28
Mg	3.58
Cd	ND
Cr	8.08
Mn	6.75
Ni	ND

ND: not detected

The heavy metal concentrations reported in the literature differ from researcher to researcher. This is because the presence of heavy metals in the sludge is highly dependent on the dosage of the dyes, water and other chemicals utilized in the manufacturing process [5, 8, 10–12, 18, 21–23]. The literature results show the concentration of the heavy metals is quite high while the findings of this study are not as significant. Nevertheless, the disposal of this sludge to the environment can cause a considerable risk. So, it is very important to use it such as for the manufacturing of bricks or use as partial substitution of cement raw materials.

As can be seen in Table 4, the textile sludge is composed of high amounts of alumina (Al_2O_3) and silica (SiO_2) as compared to the other compounds. This chemical composition data indicates that the textile sludge has the potential to be used as a partial substitute of clay soil for making building materials such as bricks. However, it is hard to use it alone due to the less quantity of mineral oxides that do not fulfill the average amount of oxide requirements. The characteristics of good bricks is largely dependent on the main chemical constituents and respective proportions: silica (50-60%), alumina (20-30%), lime (2-5 %), ferric oxide (5-6%) and magnesia (<1%) [24].

The existence of these components plays a significant role in the property of bricks. The presence of silica prevents cracking, shrinking and warping of raw bricks. The occurrence of alumina imparts plasticity to earth so that it can be molded easily. Simultaneously, iron oxide acts as a flux to cause the grains of sand to melt and this helps to bind the particles together and it imparts red color to brick on burning [5].

The chemical composition of the sludge is very much related to the minerals found in clay but with a lower amount. As given in Table 4, the main components of textile sludge are CaO (2.56%), Al_2O_3 (21.4%), SiO_2 (16.1%), and Fe_2O_3 (2.32%). This shows that the textile sludge has similar components to clay, which means a likeness in terms of composition to clay, although not in percentage quantities. Conversely, as can be seen in the same table, the quantity of the silica content in the sludge is exceptionally low which is 16.10 to 18.5 % according to the chemical and XRF analysis. The test result implies that it has a much lower amount of silica than that in the standard range, i.e., 50 – 70 % in building materials.

Several researchers have described the great role of silica content in the strength of bricks. For instance, Hegazy et al. [25], revealed that the strength of brick is highly dependent on the amount of silica in the raw materials which implies that the higher the silica, content the stronger are the bricks.

Moreover, the less amount of silica in the sludge indicates it lacks the binding property if it is used as an additive in building material. On the other hand, the quantities of the rest of the principal chemical compounds satisfy more or less the minimum percentage requirements. Also, the high value of the loss on ignition (L.O.I), with weight loss of approximately 51% indicates the availability of high volatile and organic content in the sludge [19].

The present study shows the same chemical compositions as reported by Rahman et al.[11]. However, it is not quite consistent with other results reported in different studies in the literature [10,12,26]. This might be due to the variation of the chemicals and other compounds utilized in the wastewater treatment plant and the manufacturing processes.

Therefore, the above results suggest that textile sludge is suitable to make bricks by partially replacing some percentage of the other materials such as clay material to get the required amount of chemical composition in the mixture.

3.2 Statistical analysis and optimization of bricks synthesis parameters

For statistical analysis and optimization of bricks synthesis parameters, a total of 13 experimental runs were carried out according to RSM-CCD. The results from bricks characterization, i.e., compressive strength, and water absorption, were used as study responses. Table 2 shows the experimental runs with the actual and predicted responses. To obtain the best model equation that would describe the relationship between the two study factors and the responses, the sequential model sum of squares model fitting was employed. Accordingly, the linear and quadratic model equations were selected for compressive strength and water absorption capacity, respectively. The two model equations can be written in terms of coded study factors: firing temperature (A) and sludge mix (B) as follows:

$$\text{Compressive strength} = 3.08 + 2.18 \text{ x } 10^{-3}A - 0.09B \qquad (3)$$

$$\text{Water adsorption } = 76.738 - 0.118\text{A} - 0.592\text{B} - 4.650 \text{ x } 10^{-4}\text{AB} + 5.937A^2 + 0.052B^2 \qquad (4)$$

Table 4 Chemical composition of textile sludge

Composition	Chemical analysis Content by weight (%)	XRF analysis Content by weight (%)
CaO	2.56	9.8
SiO_2	16.10	18.15
Al_2O_3	21.40	25.3
Fe_2O_3	2.32	3.12
MgO	1.51	3.11
SO_3	0.94	5.88
LOI	51.3	ND
Na_2O	ND	2.75
K_2O	ND	0.27

ND: not detected

The adequacies of these models were also investigated using the regression coefficient (R^2), which were found to be 0.9498 and 0.9971, respectively. These indicate that the model equations are adequate to predict the selected responses in the experimental range with 94.98 % and 99.71 % variability respectively. In other words, only 5.02 and 0.29 % of residual variability can be seen in the estimation of compressive strength and water absorption capacity, respectively. This is further confirmed from the actual and the predicted values that are in agreement as depicted in Table 2. The Pred R^2 show that the model equations for compressive strength and water adsorption give good predictions with 90.88 % and 97.88 % variability, respectively. In addition, the adj-R^2 of 93.98 % for compressive strength and 99.50 % for water absorption were in a reasonable agreement with Pred R^2 values i.e., the difference is less than 0.2. The degree of precision and reliability can be explained by the low values of coefficients of variable (CV) which were 2.58 % for compressive strength and 1.11 % for water absorption. Furthermore, adequacy precision for each response was found to be 31.025 and 76.906. Adequacy precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The results indicated in this study show an adequate signal. Therefore, the two models can be used to navigate the design space.

The analysis of variance (ANOVA) for the suggested linear and second-order quadratic models shows F-values of 94.62 and 480.89 for compressive strength and water absorption, respectively. The P–values for the two responses also obtained are <0. 0001.These two results confirm that the adequacy of the model fits and the significance of each of the coefficients in the model equation [27].

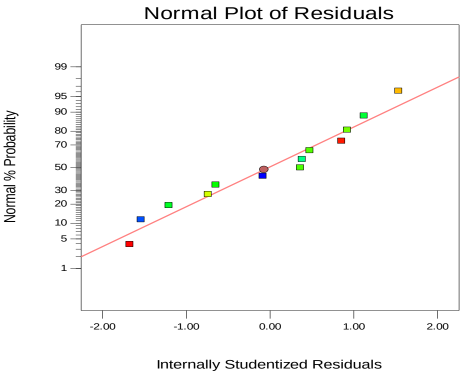

Figure 1 Normal plot of residual for compressive

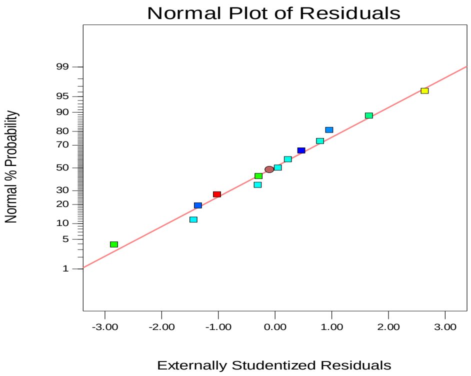

Figure 2 Normal plot of residual for water absorption

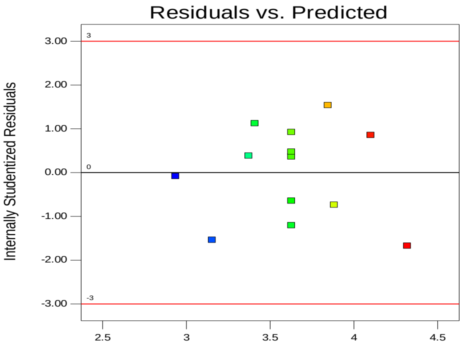

Figure 3 Residual vs. predicted response plot for compressive strength

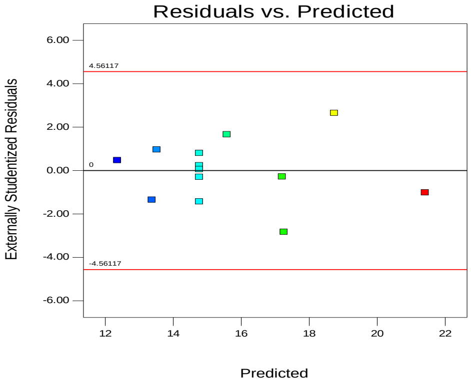

Figure 4 Residual vs. predicted response plot for water absorption

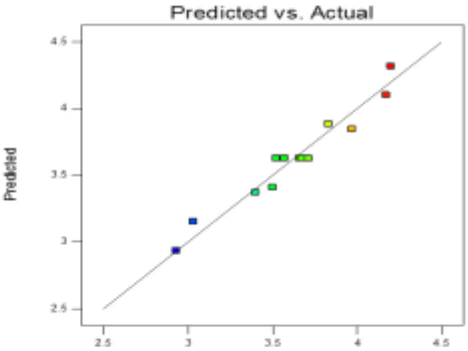

Figure 5 Actual and predicted values plot for compressive strength

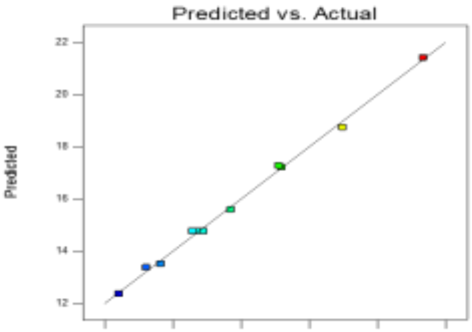

Figure 6 Actual and predicted values plot for water absorption

Furthermore, the adequacy of the model was checked by constructing different diagnostic plots for the two responses as shown in Figures 1, 2, 3, 4, 5 and 6. The normal % probability plots of residuals for the responses were normally distributed, as they lie reasonably close to the straight line and show no deviation of the variance (Figures 1 and 2). Internally studentized residuals plots that were constructed to facilitate the satisfactory fit of the developed model and the plots (Figures 3 and 8) show that all the data points lie within the limits (±3). The predicted values obtained from the suggested models were quite close to the experimental values and lie reasonably close to the straight line and indicate the adequate agreement with real data (Figure 5 and 6).

3.3 Effect of sludge content on bricks compressive strength

Figure 7 shows effect of textile sludge mix proportion on the compressive strength of bricks. The mixing ratio of sludge has a great influence on the compressive strength of the bricks. As shown in the figure, compressive strength is found to be inversely proportional to the sludge content. This may be because the sludge has much less amount of silica and therefore an increase in sludge content creates low binding power with the clay [28].The study shows that the sludge content (10 %) gives quite high compressive strength (4.20 MPa) at 1000 ^{o}C firing temperature. It is worth to note that the minimum compressive requirement of bricks which is 3.5 MPa according to ASTM is surpassed by all bricks prepared up to the sludge composition of 15% and fired at all temperatures investigated. In this study, the values of compressive strength obtained for 20 % of sludge mix at all firing temperature were below the minimum compressive strength requirement.

3.4 Effect of sludge content on water absorption

As can be observed from Figure 8, water absorption increases with increase in sludge content. This is because of increased number of pores in the brick resulting from burning of organic matter present in the sludge. More than 21 % of water absorption was recorded for the sample prepared at 20 % sludge mix and firing temperature of 800 ^{o}C. In general, the higher the percentage of sludge, the higher the water absorption becomes, resulting in lower quality bricks since the two important quality parameters, i.e., durability and resistance to the natural environment are inversely proportional to water absorption of the bricks [10].

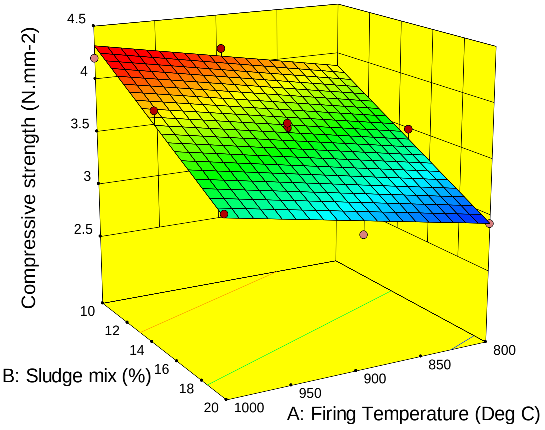

Figure 7 Three-dimensional response surface plots for compressive strength as a function of sludge mix and firing temperature

3.5 Effect of firing temperature on bricks compressive strength

The burning process of bricks has a direct impact on the final product quality of the brick material because the firing process essentially involves the oxidation of organic matter, the transformation of inorganic components into less harmful compounds and the elimination of the pathogens that exist in the sludge to reduce public health impact. The mineral compositions are transformed to increase the durability and strength. During firing the minerals are fused and undergo chemical reactions forming complex compounds at high temperature. For this reason, firing temperature is an important factor that greatly affects the property of the final brick such as compressive strength.

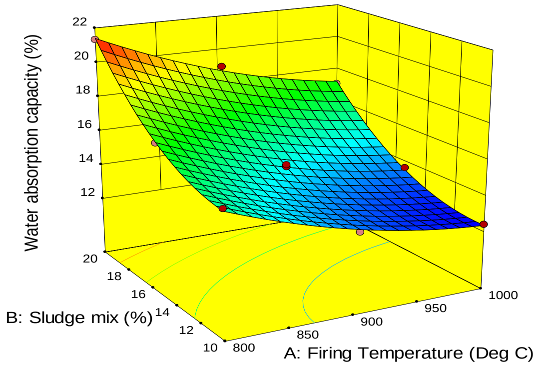

Figure 8 Three-dimensional response surface plots for water absorption as a function of sludge mix and firing temperature

Figure 7 shows the effect of firing temperature on compressive strength in the temperature range of 800°C-1000°C.As can be observed from the same figure, the compressive strength is seen to increase with temperature for all the mixing proportions of the sludge. The compressive strength increased from 2.93 to 4.20 MPa as the temperature increased and sludge content decreased from 20% to 10%. More specifically, the compressive strength of 10% sludge content fired at higher temperature nearly reaches the maximum compressive strength of 4.2 MPa.

Furthermore, the 20% sludge content fired at 1000 was cracked and deformed its shape exceptionally. In general, during the firing process the atomic bonding of particles increase by the mechanism of diffusion creating a denser material and conversely, this leads the brick to shrink progressively [21].

3.6 Effect of firing temperature on bricks water absorption

Water absorption does not only depend on the proportion of sludge mix but also on firing temperature (Figure8). In this particular study, water absorption of the bricks was found to decrease with increase in firing temperatures for all sludge mix proportions. In general, water absorption is expected to increase at maximum sludge mix and firing temperature due to formation of void space during the baking process [28]. However, after a certain firing temperature these voids collapse and shrink and result in lower water absorption. As shown in the figure, for brick samples made at different sludge mix show a significant increment of water absorption from 12.4 to 21.33% when they were subjected to different firing temperatures. Generally, lower water absorption bricks are more durable than those with higher water absorption.

3.7 Optimization and verification of bricks synthesis parameters

Based on Derringer's desirability function approach for multiple response processes, the desired set of bricks synthesis parameters were determined through numerical optimization which searched the design space, using the two model equations created during analysis to find factor settings that meet the defined goals. To get the best optimum parameters, the compressive strength was targeted to be 3.5 MPa (according to ASTM minimum requirement), water absorption was set to be minimum, sludge mix set to be maximum in order to utilize more textile sludge and the firing temperature was selected to be in the range. Accordingly, the optimum synthesis parameters were found to be the proportion of sludge mix 18.66% and firing temperature of 1000 °C with 0.802 % desirability to predict the compressive strength of 3.5 MPa and 16 % water absorption. Some researchers have suggested to use up to 30 % textile sludge to replace Portland cement in the preparation of building materials and reported that as the sludge mix is getting higher, the compressive strength of the brick drops gradually[8,21]. Based on the results obtained by Baskar et al. (2006), the addition of textile sludge up to 9% is effective enough to make brick material at a temperature of 800 and obtain a compressive strength of 3.54 MPa. Besides, according to Jahagirdar et al. [28], textile sludge content of up to 15% was recommended to obtain values of the compressive strength exceeding 3.5 MPa. The water absorption also should be less than 20% in order to get more durable bricks. To validate the predicted two response values, three samples were prepared under the selected optimum parameters and mean compressive strength and water absorption value of 3.87 ± 0.27 % and 15.12 ± 0.56 were obtained, respectively. The values are very close with the data obtained from numerical optimization analysis and the model equations can be potentially used to predict synthesis parameters in the same preparation conditions.

3.8 Leachate test

The textile sludge-based sample brick prepared at the optimum synthesis parameter was subjected for leaching test. Table 5shows the leachability result for the textile sludge-based bricks. The results indicate that the elements Tn, Cr, Ni, and Hg have shown relatively maximum concentrations in descending order whereas other metals such as Cd, As, Pb, Zn, and Cu have shown minimum leachability from the brick sample. However, as the test results show, the concentrations of all heavy metals were found within the allowable limits as given in the USEPA standard. This may be due to the phenomenon that some of the heavy metals in the sludge were transformed at the high firing temperature during sintering process, thus reducing the heavy metals inside the brick. The results indicated that the addition of textile sludge up to 18 -19 % composition into clay to produce bricks is possible and the heavy metals concentration was reduced significantly when incorporated into fired clay bricks. Therefore, this research shows the suitability of textile sludge as a partial substitute of clay for making bricks but recommended with quite moderate quantity which is up to 19 % and fired at high temperature (1000 ^{o}C).

Table 5 Leachate analysis test results for sample bricks prepared at optimum synthesis parameter

Parameters		Content (mg/l)	USEPA Concentration limit (mg/l)
Copper	Cu	0.003	100
Iron	Fe	0.006	ND
Zinc	Zn	0.003	500
Nickel	Ni	0.048	1.3
Cobalt	Co	0.004	ND
Manganese	Mn	0.006	260
Chromium	Cr	0.053	5.0
Cadmium	Cd	0.001	1.0
Mercury	Hg	0.045	0.2
Tin	Sn	0.066	ND
Lead	Pb	0.003	5.0
Arsenic	As	<0.001	5.0
Boron	B	0.0295	ND

ND not detected

4. CONCLUSIONS

In the present study, the textile sludge-based synthesis parameters were investigated and statistically optimized using the RSM-CCD. The study has revealed that the statistical experimental method can be used as an excellent tool to identify the interaction effects of the individual synthesis parameters for bricks production. It was found that the compressive strength drops gradually as sludge proportion increases and increases progressively with firing temperature. Consequently, a good compressive strength result can be achieved at a higher temperature and smaller sludge proportion. Besides, the study has shown that the addition of sludge affects the water absorption to rise gradually with increase in sludge content. Under the optimized synthesis and firing condition, i.e., sludge mix of 18.66 % and firing temperature of 1000 ^{o}C, 3.5 MPa comprehensive strength and water absorption of 16 % were predicted. Leachate analysis test shave shown minimum leachability of heavy metals. Based on the present findings, it can be concluded that utilization of textile sludge as partial substitute of clay in bricks production can potentially reduce textile sludge disposal and associated environmental problems.

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CONFLICT OF INTEREST

The authors declare that they have no conflict of interest.

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