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Development of Environmentally Friendly Bricks using Spent Diatomaceous Earth

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Abstract

Diatomaceous Earth (DE) is utilized as a filter material in breweries. Spent Diatomaceous Earth (SDE) is a type of industrial waste that is produced when the pores of DE get clogged by contaminants generated from the brewing industry. This SDE is thrown into dumping locations upon the final filtering process, results environmental pollution. As a viable solution for that problem, SDE can be mixed with clay to produce bricks. When clay is combined with SDE, the quality of the bricks improves. Incorporating SDE into the brick manufacturing can mitigate the negative environmental consequences. The purpose of this research was to evaluate the applicability of SDE as a raw material in brick production. Both X-Ray Fluorescence (XRF) and Thermo Gravimetric Analysis (TGA) were conducted to examine the physical properties of SDE. The TGA results revealed a significant difference between the residual amounts in wet (4.45%) and dry (65.29%) SDE. Sun-dried bricks containing 0, 5, 10, and 15 wt% of SDE were burnt at 950 °C for 6 hours with 10 °C min^{-1} temperature ramp. The obtained results have shown that raising the SDE weight percentage improves compressive and flexural strengths. The sample generated with 15 wt% of SDE has the maximum compressive strength of 4.78 MPa and flexural strength of 0.57 MPa. The inclusion of SDE is also worthwhile because no significant alterations in physical attributes are noticed. Further research and development, taking the technological, economic, and environmental elements of SDE into account, is recommended in order to produce SDE included bricks on a larger scale.

Keywords: Bricks, Clay, Spent diatomaceous earth

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1. Introduction

The development of waste materials and by-products from industry has become a serious environmental and societal issue around the world. Therefore, scientists are doing research to reuse or recycle the trash generated. Moreover, in order to safeguard the environment and society, the government and relevant authorities have adopted a number of regulations for governing the waste disposal. Hence, all industries seek to limit trash production or repurpose garbage for other productions [1]. Breweries create leftover diatomaceous earth as a waste and are looking for an environmentally appropriate approach to manage it.

Diatomaceous earth (DE) is made up of deposits developed by the sedimentation of fossilized skeletons of unicellular marine algae known as diatoms, which have siliceous skeletons and are connected with clay minerals and quartz [2,3]. The world's largest diatomite reserves are found in the United States, China, among other places. In 2013, the world produced 2.3 MT of DE, with the United States accounting for 33%, China 18%, Denmark 14%, and Peru 5.3%. Other 25 countries have small production of DE[3]. As a non-metallic, soft, friable, fine-grained, and silicious sedimentary rock, diatomaceous earth, also known as Kieselguhr (Fig. 1), can be used as a filter medium to separate very tiny particles. Water, juice, wine, spirits, syrups, and gelatine can all be filtered with diatomaceous earth [2].

The microstructure of diatoms is highly complicated, with many small pores and channels that assist the material maintain low specific weight, low heat conduction, high specific surface area, and high absorption capacity [2]. Because it includes silica-rich tiny particles with a very porous structure, DE is useful for insulation. By increasing the porosity inside the brick, organic waste combined with SDE can aid to minimize fuel consumption during the burning process and lower the weight of the brick. SDE also contains silica aluminate ($SiO_2.Al_2O_3$), which transforms into various crystal and glass phases during the bricks firing. As fluxes, $SiO_2.Al_2O_3$ contribute by lowering the fire temperature required to finish the firing process of bricks [1]. As a result, SDE can be utilized to create lightweight calcium silicate bricks with good thermal insulation [4].

Fig. 1. Diatomaceous Earth

Diatoms mixed with mud, sand, and water, were used in the construction of ancient Egyptian structures [2].

The brewing industry, which generates 364 million kilograms of SDE annually, is primarily responsible for producing SDE as waste [5,6]. Rice and malted barley, which are used as raw materials in the brewing process, are initially ground and then transferred for mashing. Then, using a lauter tun, the watery form of wort was separated from the grains and solids known as bagasse. After that, hops and other ingredients are added to the wort, and boiled. Clarified wort is cooled to a temperature where yeast can be introduced and aerated before fermentation. Solid particles are separated by centrifuge and filtered using Diatomaceous Earth, commonly known as Kieselguhr, for further filtrations after fermentation [4]. Due to the accumulation of organic material, cake mass can increase three times or more by the completion of the filtration, and this material cannot be reused in further filtration after saturation. Using SDE has been the subject of numerous studies. Some of them are concentrating on making bricks SDE.

Kieselguhr, which is produced as waste in breweries, is currently placed in landfills because of the large-scale production and lack of alternatives for reuse or value-adding. SDE should not be disposed of in landfills due to its toxicity. Land degradation, inland water pollution, loss of agricultural lands, deforestation, air pollution, and loss of biodiversity are all detrimental consequences of dumping SDE in landfills [2]. Although, the SDE can be used as animal feed, it can only be fed to ruminants due to its high cellulose and hemicellulose content. DE can be used to produce the bricks, which is one of the options for disposal [7]. Using waste DE to make bricks has various advantages, including lower waste disposal costs and environmental protection. In addition, the high water content of SDE reduces the amount of water used in the brick-making process. Rather than employing the traditional synthetic pore-forming material (expanded polystyrene), which has regulatory limits on effluent gaseous emission, this by-product provides a viable and cost-effective alternative for reducing the bulk density of ceramic bricks [2]. As a result, it is possible to reduce the use of clay in brick manufacture by replacing portion of clay with SDE. The environmental impact can be significantly lessen.

The SDE can be used as a soil conditioner in agricultural areas, as a raw material in the construction sector (concrete and brick manufacture), as a component in composting, or as a raw material for biofilter production [1,8]. According to early research, eliminating water from SDE improves storage and reduces the odour problem. However, the brewing sector may find this procedure to be prohibitively expensive. As a result, given the current environment, DE regeneration for the brewing industry is unlikely to be a realistic choice [2,8].

SDE recycling in the agricultural, construction, and brick-making industries appears to be possible, environmentally friendly, and economically viable options. In terms of cost, replacing the type of filter and filter aids utilized is unlikely to be worthwhile. The production of bricks utilizing waste DE; SDE (in both wet and dry form) was chosen as the most convenient and viable solution in the current study, taking several parameters such as time, energy, resources, and total cost into account. The objectives of this study are to identify the chemical composition of SDE and compare the changes of mechanical properties of bricks with the addition of SDE, and finally evaluate the suitability of recycling SDE as a silica precursor for brick manufacturing.

2 Experimental

2.1 Material

Fig. 2. a: Step 1: SDE Drying, b: Step 2: Mixing SDE with Clay, c: Step 3: Moulding, d: Step 4: Drying

SDE was provided by Lion breweries, Biyagama. It was kept for sedimentation in a container for a few hours. After that, it was sundried for three days to remove any residual moisture. Clay was gathered in from a clay pit located nearby Chilaw, Sri Lanka.

2.2 Sample preparation

The collected clay was combined with 0%, 5%, 10%, and 15% of dried Spent Diatomaceous Earth (SDE) until the mixture was homogenized. Fig. 2 (a,b,c,d) steps shows the procedure of brick production.

Initial sample weight was measured before drying to determine the moisture content of clay and SDE-wet form. The samples were then heated in a 70 °C oven until their weight remained stable. The dried samples were then weighed. The weights of the initial samples and dried samples were subtracted to determine moisture content. Based on the findings, an adequate amount of water was added to the SDE – Clay mixture to eliminate defects in the moulding process and provide proper plasticity.

Four bricks made entirely of clay were used as references. SDE-incorporated bricks' qualities were contrasted with those of the reference bricks. The mould’s dimensions were 220 mm x 105 mm x 65 mm, as specified by Sri Lankan standards (SLS 39 of 1981). Bricks that had previously been manufactured were dried in the sun for two weeks. The dried samples were then fired in a muffle furnace for 950 °C at a rate of 10°C/min, for 6 hours. The samples were then cooled to room temperature within the muffle kiln using natural convection.

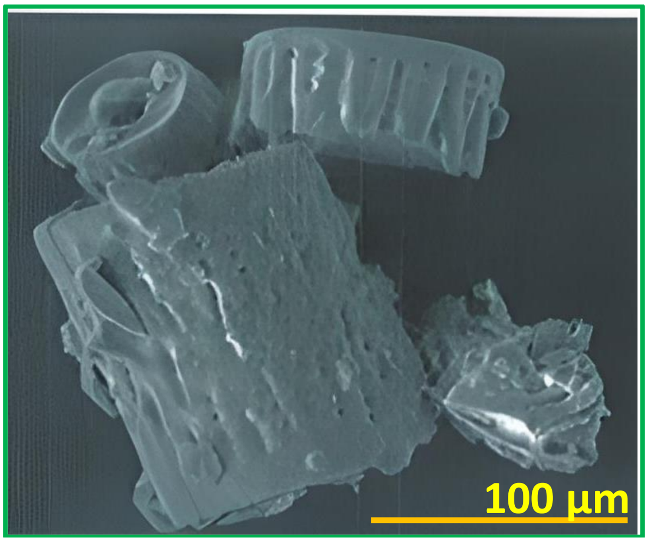


Fig. 3. SEM image of SDE before adsorption

2.3 Characterization of the raw materials and brick

The Hitachi SU6600 Field Emission Scanning Microscope was used to examine the microstructure of SDE. For Scanning Electron Microscope (SEM) imaging, the sample was mounted to the sample stub and images were taken after 15 seconds of gold sputter coating. The moisture content of SDE was calculated using the SDTQ600 Thermo Gravimetric Analyzer. The temperature was changed from ambient to 1000 °C at a rate of 10 °C/min. High purity Nitrogen was used as a purge gas.

The chemical components of SDE were determined by X-Ray Fluorescence (XRF) analysis using HORIBA Scientific XGT – 5200 X-ray Analytical Microscope. Chemical compositions were determined by analysing six distinct spots per sample. The diameter of the XGT (X-ray Guide tube) was set at 100 µm, and the X-ray tube voltage was kept at 50 kV. In addition, the processing time was set to P4 and the live time to 300 seconds. The Compressive and flexural strengths were measured in the Material Laboratory Department of Civil Engineering, University of Peradeniya. compressive strength of a brick was determined by gradually increasing the load on the load-bearing surface until it fractured. The area of force applied was calculated by taking the average of both load-bearing surfaces. The applied force at the fractural point of the brick was divided by the average area of load-bearing surfaces, expressed in MPa, to compute compressive strength. A three-point bending strength test using a 5 kN load cell, 100 mm as the distance between the points of support, and a displacement rate of 10 mm/min was used to measure bending strength.

2.4 Characterization of SDE

Wet and dry forms of SDE need to be characterized in order to determine the proper composition of SDE that should be mixed with soil samples. Thus, characterization of samples with TGA, XRF, SEM coupled with EDX (Energy Dispersive X-ray) have been carried out. SEM images are obtained to analyse the morphology and porous structure of SDE in dry forms and the porous nature was observed as shown in Fig. 3.

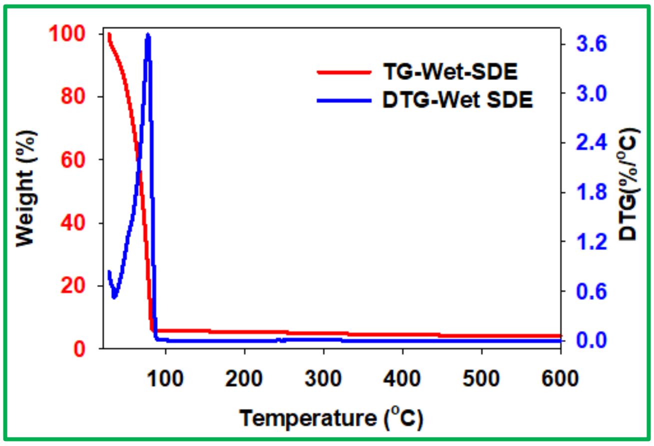
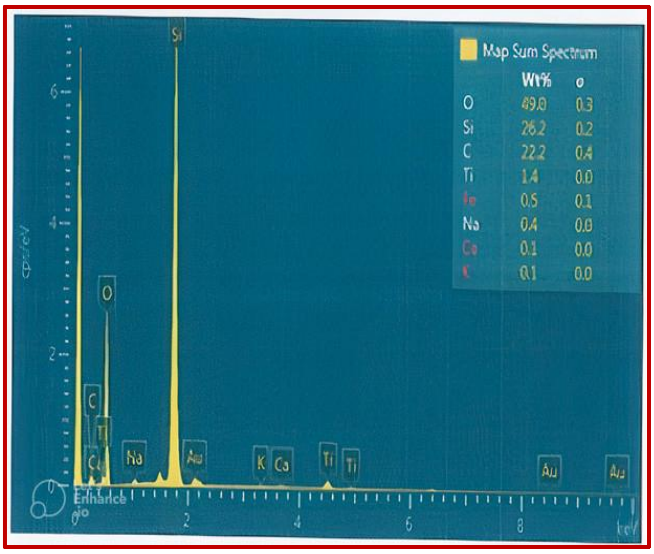


Fig. 4. TG and TDG analysis of wet SDE



Map Sum Spectrum

	Wt%	σ
O	49.0	0.3
Si	26.2	0.2
C	22.2	0.4
Ti	1.4	0.0
Fe	0.5	0.1
Na	0.4	0.0
Ca	0.1	0.0
K	0.1	0.0

Fig. 2. EDX spectrum of SDE dry form

Dry and wet foam of SDE were subjected to thermogravimetric (TG) and differential TG analyses (DTG). This analysis was carried out to determine the percentage of water in both samples, which will be used to calculate the amount of water that should be added to mix the selected soil with various SDE percentages. It was also discovered that SDE-wet form has 89.32 % of water (See Fig. 4), and SDE-dry form contains 28.87 % of water. The residual in SDE wet and dry form is 4.45 % and 65.29 %, respectively.

The elemental composition of the dry form SDE was determined using EDX. Fig. 5 shows EDX analysis of SDE dry form. Oxide composition of SDE obtained from XRF are shown in Table 1, and elemental composition of SDE obtained from EDX are displayed in Table 2.

As shown in Table 1, SiO_2 (95.43 %) is the most abundant oxide in SDE, followed by Al_2O_3 (Aluminium oxide) (2.53 %). SDE contains fluxing oxides (K_2O) and auxiliary fluxing oxides ($CaO + Fe_2O_3$) [1]. Because of the oxides, the firing temperature can be reduced. Therefore, SDE was found to be a viable secondary raw material for brick production based on its chemical composition.

Table 1: XRF analysis of SDE (wet form) and oxide composition

Oxide	SDE wt %	Clay wt% [1]
Al_2O_3	2.53	12.12
SiO_2	95.43	55.28
SO_3	0.28	-
K_2O	0.25	2.78
CaO	0.47	9.21
TiO_2	0.13	0.83
MnO_2	0.01	-
Fe_2O_3	1.17	4.83
ZnO	0.01	-
MgO	-	1.49
Na_2O	-	0.49
P_2O_5	-	0.12

Table 2 shows the SDE's elemental composition in terms of C, O, Si, and Fe. Carbon content is 22.2 %, indicating the amount of organic stuff. As a result, SDE might be used as a fuel throughout the firing process to meet the heating requirements. The SDE does not produce SO_2 during the firing process because it does not contain Sulphur.

Table 2: ELEMENTAL COMPOSITION OF DRY FORM OF THE SDE

Element	Weight percentage (%)
C	22.2
O	49.0
Si	26.2
Ti	1.4
Fe	0.5
Na	0.4
Ca	0.1
K	0.1

3 Results and discussion

3.1 Possible chemical reactions occurred inside the bricks during firing

During the firing e bricks, several processes are occurred, including water loss (exterior and chemically associated), mineral disintegration and conversion, carbon burnout, quartz inversion, and vitrification. When the firing begins, loosely bound water molecules evaporate at a temperature of roughly 100 ℃ [9]. The second weight loss can be attributed to the burning of organic materials in the clay between 200 ℃ and 500 ℃ [1]. At high temperature, a process known as dihydroxylation can also be occurred.

The kaolin dihydroxylation ($Al_2O_3.2SiO_2.2H_2O$ into $Al_2O_3.2SiO_2$ and $2H_2O$) between 400 ℃ and 700 ℃. There are two processes to dehydroxylation in montmorillonite. The first phase is larger than the second and occurs between 600 - 800 ℃. The second stage reaction takes place between 850 and 925 ℃ can be shown as.

$Al_2O_3.4SiO_2.H_2O \longrightarrow Al_2O_3.4SiO_2 + H_2O$.

Muscovite mica, a mineral found in clay, dehydroxylates at around 700 ℃. Additional minerals such as pyrite (FeS_2) and limestone can be found in some clay types ($CaCO_3$). The red colour of the bricks was caused by the oxidized iron which is given by the two-stage decomposition of pyrite.First reaction, $FeS_2 + O_2 \longrightarrow FeS + SO_2$ occurs at 380 ℃ and the second reaction, $FeS + 7\ O_2 \longrightarrow 2Fe_2O_3 + SO_2$ occurs around 412 ℃ [9].

Also, at 800 ℃, $CaCO_3$ decomposes into CaO (lime) and CO_2 [1], increasing the porosity of the brick [10]. CaO is transformed to $Ca(OH)_2$ in the presence of water. In the presence of ambient carbon dioxide, it eventually changed to calcium carbonate. As a result of these reactions, volume increases, leading to the formation of fissures, a process known as lime blowing [11], which increases porosity even further. Lime and periclase (MgO), which are decomposition products of dolomite, contribute to the porosity of brick by transforming portlandite and brucite ($Mg(OH)_2$) in the presence of water. In the presence of carbon dioxide, the latter may transform into hydromagnesite ($Mg_5(CO_3)_4(OH)_2 \cdot 4H_2O$) [12].

3.2 Technological properties of waste – clay mixtures

When SDE was in its slurry form, it was difficult to create a homogeneous combination during the manufacture of the SDE-Clay mixture. As a result, SDE was sun-dried before being mixed with clay, and since SDE was dried using natural heat, there was no extra cost for this process.

Table 3: Bricks' compressive strength according to SLS 39:1978 [13]

Characteristic	Type 1	Type 2	
		Grade I	Grade II
Average compressive strength average not less than (MPa)	10	4.8	2.8

Table 3 specifies the minimum compressive strength values to be hold for commercial bricks used in Sri Lankan

Table 4: Variation of compressive and bending strengths of brick with SDE incorporation percentage

SDE % (Wet form)	Length (mm)	Width (mm)	Height (mm)	Breaking Load (KN)	Compressive Strength (MPa)	Bending Strength (MPa)
0	207	103	57	46	2.20	0.41
5	208	103	57	56	2.75	0.32
10	206	104	55	56	2.77	0.42
15	207	104	58	97	4.73	0.55

standardization. The minimum compressive strength for type 2, grade II, shown in the Table 3, is 2.8 MPa. As a result, the bricks with 15% SDE inclusion met the minimal criteria required for type 2, grade II bricks. Higher SDE percentages can therefore be combined to create bricks while still falling within the standards' acceptable range.

When the moisture level of the brick is high, the expansion of entrapped water causes swelling and bloating during the firing process. Excess moisture was gradually removed throughout the drying process to avoid this problem [12,14]. However, the loss of mechanically and physically bonded water causes to shrink the bricks significantly during both the drying and firing processes. Table 4 demonstrates how the finished bricks' three dimensions were shrunk in relation to the initial mould size. To ensure that the finished bricks have uniform dimensions, those reductions should be considered when making bricks.

Fig. 6. SDE incorporated burnt bricks

The compressive strength of bricks was raised when the incorporated SDE percentage was increased. Table 4 shows that introducing 5 wt% SDE into the bricks to reach the compressive strength of 2.75 MPa, which further increased to 4.73 MPa when 15 wt% SDE is incorporated. I proved that the dry form of spent diatomaceous earth has a significant impact on the compressive strength of SDE-prepared bricks. As stated in [1], the development of open porosity causes the compressive strength to decrease as a result of concentrated stresses acting on microscopic defects and irregularly shaped pores. The mechanical properties of the bricks were improved by introducing a higher weight percentage of SDE into the clay. This type of behaviour may occur when the SDE's melting capacity (as a silica precursor) is greater than the SDE's effect on the development of porosity in bricks, or it may result from the chemical binding properties of the SDE. The most important mechanical index for building materials is compressive strength.

When walls must withstand lateral stresses such as wind and earth pressure, the bending strength of bricks is critical. The bending strength of a brick is particularly significant for flexurally stressed components that are loaded or minimally loaded, such as cellar walls beneath patios, veneer, non-loading, and freestanding walls [15]. Though the addition of SDE has no significant influence on bending strength, the highest bending strength of 0.55 MPa has been achieved with 15 wt% SDE, whereas the bending strength of reference bricks is 0.41 MPa. As a result, including SDE has a positive impact on a brick's bending strength as well.

During the stages of mixing, drying, and firing, SDE-incorporated bricks showed no observable differences in appearance from the reference bricks. As seen in Fig. 6, after the burning stage, all of the bricks were in the same colour. The burnt bricks had no visible bloating, cracking, wrinkles, or pyroplastic flaws. At the fractured cross-sections of specimens, there is no dark core. The absence of a black core indicates that all main gaseous chemicals have been liberated and all organic matter in the SDE has been totally burned [16,17]. Due to the degradation of the content of organic matter of the spent diatomaceous earth, a strong smell has emitted during storage. However, this odour has released during the mixing and drying processes. Further, this odour can be decreased by using odour inhibitors [18].

4. Future expectations

Since the increasing SDE weight percentages has improved both compressive and bending strength, this investigation can be expanded to include high weight percentages of SDE incorporation such as 20, 25, 30, and 35 percent. In addition, the chemical composition of clay has a considerable impact on the ultimate qualities of bricks. Therefore, samples can be prepared using various clay types gathered from different areas of the country. The chemical composition of each clay type is expected to be examined prior to mixing. By comparing the compositions of the bricks before and after burning, the effect of clay chemical composition can be studied. According to literature, the mechanical properties of bricks fluctuate dramatically with the firing temperature. Therefore, experiment can be done further for different firing temperatures. Compressive strength, bending strength, tensile strength, and water absorption capacity are among top factors that the building industry looking from bricks. Identifying the relationship between those parameters and maintain optimum firing temperature, clay composition, and SDE incorporation percentage will aid in determining the best conditions to maintain the firing and drying in order to produce bricks with the desired properties. Changes in microstructure and bond forms can be detected by using XRF and SEM imaging of bricks before and after burning

4. Conclusion

The utilization of SDE as a silica carrier and pore-forming agent for brick manufacture was studied in this study. SDE was a viable alternative raw material for mixing with clay to make bricks since it has a high silica concentration of 95.43 %. SDE has been mixed with clay in various quantities (0, 5, 10, 15 wt%) and burned at 950 °C. The proportion of SDE in the mix has no effect on the brick's appearance. However, when the percentage of SDE increases, it has a considerable impact on the mechanical properties of bricks. A higher SDE % has a favourable impact on a brick's compressive strength. In addition, increasing the amount of SDE incorporation enhanced the bending strength. The 15 wt% of SDE incorporation has recorded the maximum compressive strength of 4.73 MPa and the bending strength of 0.55 MPa, higher than the reference bricks. Reusing SDE in bricks is a sustainable waste disposal technique when compared to landfill deposition. Despite extensive study, practical production of bricks made from spent diatomaceous earth is currently limited. The processes for creating SDE-based bricks, the possibility for SDE contamination, the lack of appropriate standards, and the gradual acceptance of SDE-based bricks by industry and the general public are all probable issues to be considered.

Conflicts of Interest

The authors declare no conflicts of interest.

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