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# CHARACTERIZATION OF DIATOMACEOUS EARTH TO EVALUATE ITS POTENTIAL AS A RESOURCE FOR GEOPOLYMER CONCRETE DEVELOPMENT

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## Abstract

Researchers have become interested in cutting-edge geopolymer technology and the creation of geopolymer composites as a means of achieving sustainability in the production of concrete. In this study, the feasibility of using diatomaceous earth from Nakuru, Kenya, as a source for geopolymer concrete was evaluated. The chemical and physical analysis of diatomaceous earth were carried out using standard techniques. Thermogravimetric (TGA) and Differential Scanning Calorimetry (DSC) analyses were performed on the diatomite for thermal characterization. The Sodium silicate/Sodium hydroxide alkaline activated diatomite-based brick specimens were moulded, and their mechanical and physical features were determined using standard test procedures. The diatomaceous earth's chemical composition showed that silica (SiO<sub>2</sub>) was the predominant component, with 88.12%. Calcium oxide (CaO) was 4.26% and alumina (Al<sub>2</sub>O<sub>3</sub>) was 4.25%. There were also trace levels of other oxides such as MgO, K<sub>2</sub>O, TiO<sub>2</sub>, MnO, Fe<sub>2</sub>O<sub>3</sub>, and P<sub>2</sub>O<sub>5</sub>. The thermogravimetric analysis showed a loss on ignition of 5.68 % and that its softening point is higher than 950 °C. The particle size analysis and the Atterberg limit test showed that the diatomaceous earth from Nakuru, Kenya, is a cohesive and medium plastic silt, with an average particle size of less than 50.4 µm. The diatomite-based specimens had an average compressive strength of 22.98 MPa, a density of 1.38 g/cm<sup>3</sup> and water absorption of 9.32 %. The chemical composition suggests that it is comparable to Class F pozzolan. The mechanical, physical and durability performance falls within the acceptable limits provided in literature. This research showed that Kenyan diatomite can be successfully employed as a silica source in geopolymer formulations, providing hopeful approaches to utilizing and recycling the resource.

**Keywords:** Diatomaceous earth, characterization, pozzolan, geopolymer, sustainability

## 1. Introduction

One of the fundamental issues of our time is meeting the basic requirements of growing populations while ensuring the integrity of vital ecosystems, tackling climate change, and fostering economic productivity and social inclusiveness (Klopp & Petretta, 2017). Building and maintenance are by far the largest emitters of harmful gases like CO<sub>2</sub> and this eco-footprint will only grow with the large population growth expected by 2050 (Rostami et al., 2015). Compared to the industrial and transportation sectors, building and construction consumes more than 40% of world energy and emit about the same amount of CO<sub>2</sub> (Pramanik et al., 2021). As a result, the construction industry is constantly challenged to reduce its environmental impact by incorporating the major dimensions of sustainable development. The simplest way for designers to begin implementing sustainable ideas into construction projects, according to Aghdam et al. (2018), is to carefully select ecological building materials.

To achieve sustainability in the manufacturing of concrete, numerous researchers have developed an interest in cutting-edge geopolymer technology and geopolymer composite production. This is due to the fact that geopolymer production utilizes a variety of wastes as either supplementary cementitious materials (SCM) or precursors in the synthesis of geopolymers, at low temperatures and with minimal energy, while reducing the CO<sub>2</sub> footprint of the cement industry by up to 80% (Duxson et al., 2007; El-Dieb, 2016).

To reduce consumption and dependence on cement, the use of pozzolanic materials has been a major research focus in the field of cement and materials in recent years (Danso H & Adu S, 2019). The most popular geopolymer precursors (aluminosilicate sources) that have been extensively studied thus far include fly ash, ground granulated blast furnace slag, metakaolin, silica fume, and rice husk ash (Elahi et al., 2020; Nodehi & Taghvaei, 2022). Natural or spent diatomaceous earth, also called diatomite, has received very little attention, despite being regarded as one of the geopolymer system components by Payá et al. (2018) among other experts. According to Zahajská et al. (2020), massive accumulations of fossil diatom frustules have reportedly been discovered in numerous lakes located in silica-rich environments, particularly in volcanic and hydrothermally active areas, including Yellowstone Lake in the United States, Lake Myvatn in Iceland, Lake Challa in Tanzania and Kenya, among others. In Kenya, Gevera et al. (2018) determined that diatomaceous earth sediments are found in the Nakuru-Elmenteita basin near Kariandusi.

The major end use for processed diatomite nowadays is as a filter aid; uses for filtration include the purification of beer, wine, and other alcoholic beverages, vegetable oil, syrup, pharmaceuticals, motor oil, and swimming pool water (Kogel & Society for Mining, 2006). According to U.S. Geological Survey (2022), around 55% of diatomite is used for filtration purposes. Consequently, spent diatomaceous earth (SDE) has become a significant source of industrial waste for industries like food processing and brewing (Galán-Arboledas et al., 2017; Mateo et al., 2017). For instance, approximately 378.1 million kilograms of SDE are produced annually by the brewing sector (Gong et al., 2019; Thiago et al., 2014). This used diatomite ends up in landfills or is applied to crops as organic fertilizer, both of which squander resources and harm the environment (Galán-Arboledas

et al., 2017). Additionally, the risk of leaching nitrogenous compounds present in the wasted diatomaceous earth could be increased by its use in agriculture. Moreover, due to the significant energy, labor, and cost requirements, the regeneration of SDE may not be a viable option. The use of SDE for more economically viable and environmentally sound applications, such as in geopolymer concrete technology is therefore of great importance.

The goal of this study is to evaluate the potential for using the diatomaceous earth resource to produce geopolymer concrete. A deeper comprehension of SDE's physical, chemical, mechanical, and pozzolanic characteristics is thus necessary for its adoption as a geopolymer resource.

## 2. Materials and methods

### 2.1 Materials

The raw diatomaceous Earth and alkaline activators were the main materials employed in this study. In this instance, sodium-based alkali activators were used, specifically a solution of sodium hydroxide and sodium silicate gel ( $\text{NaOH}/\text{Na}_2\text{SiO}_3$ ), as advised by Cong et al. (2021). The materials were collected from sources within Kenya; specifically, diatomaceous earth was acquired from Nakuru, while sodium hydroxide and sodium silicate were bought from one of the outlet suppliers in Eldoret. Figure 1 presents a geological map of Nakuru county in Kenya which hosts the diatomaceous earth reserves (Gevera & Mouri, 2018).

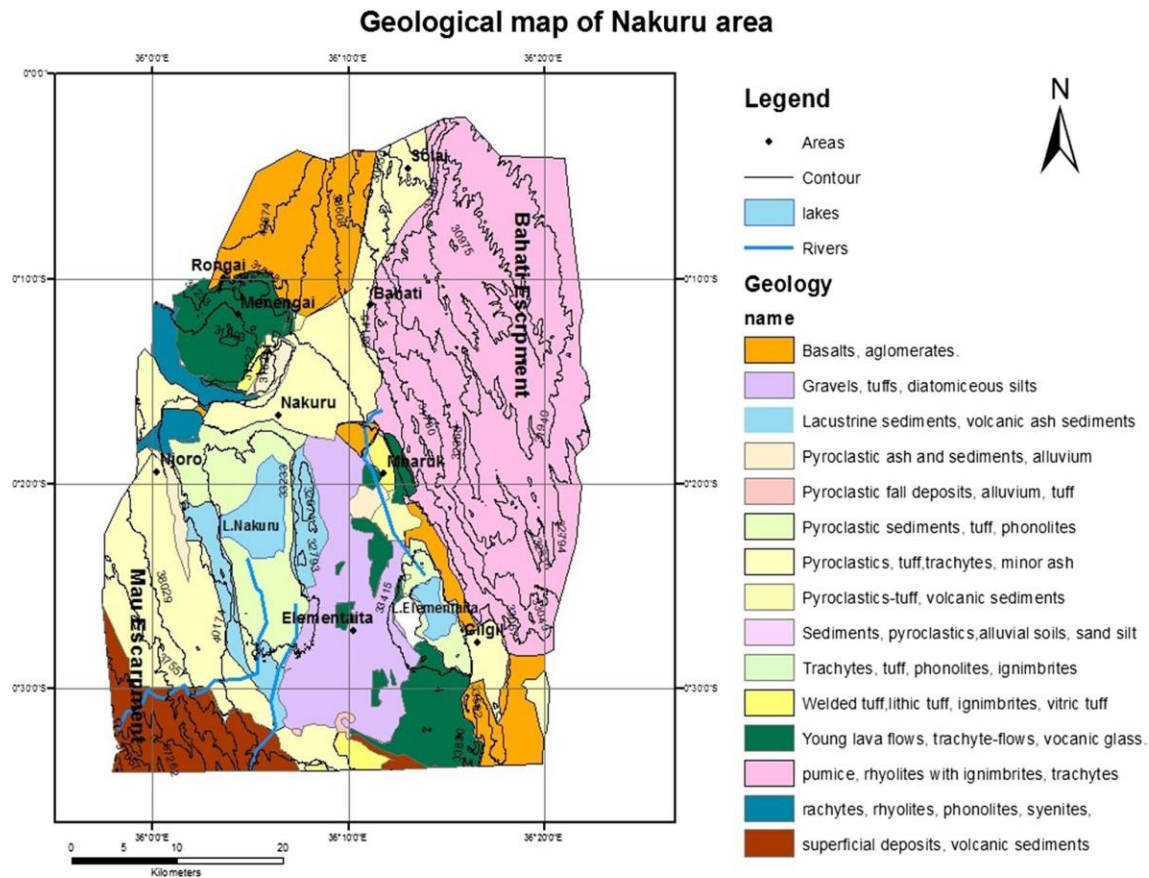


Figure 1. Geological map of Nakuru county-Kenya

## 2.2 Diatomaceous earth characterization

The diatomaceous earth had already been finely powdered when received. To get rid of all the moisture, it was oven dried at 110°C for 24 hours. The X-Ray fluorescence (XRF) apparatus was used to determine the chemical composition as per ASTM C114-10 (2010). To determine the crystallographic structure of the diatomite for further mineralogical analysis, the X-Ray diffraction (XRD) was employed. The Atterberg limits test, conducted in accordance with ASTM D4318-17 (2017), was used to determine the clayey nature of diatomaceous earth. Utilizing an LS 13 320 Laser Diffraction Particle Size Analyzer and following ASTM B822–17 (2017) guidelines, a particle size distribution analysis was performed. In line with ASTM-D854, (2010) and ASTM D 7348-13, (2013), respectively, the specific gravity and loss on ignition were carried out.

The thermal characterization of diatomaceous earth was performed through the use of Thermogravimetric (TGA) and Differential Scanning Calorimetry (DSC) analysis techniques according to ASTM E1131, (2015). A TGA-550 thermal analyser operating in a nitrogen atmosphere, flowing at a rate of 50 mL/min was employed. The weight change with respect to temperature was computed within a programmed temperature range of 25 °C to 950 °C at a rate of 10 °C/min. The DSC-Q200 machine was employed, operating in a static air environment between -50 °C and 450 °C at a rate of 10 °C/min.

## 2.3 Geopolymer sample preparation and performance evaluation

According to the insights provided by Mohammed et al. (2021) in their literature review, an alkaline activator consisting of a mixture of Sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) gel and Sodium hydroxide (12M NaOH) solution was used to create the geopolymer specimens. Based on the findings of the literature review conducted by Zhang et al. (2020), the ratio of sodium silicate to sodium hydroxide was maintained at 2.5. 480 grams of NaOH was dissolved in 1000 ml of distilled water to create a 12M NaOH solution. The diatomite and the alkaline activator were mixed at a constant liquid-binder ratio of 0.7 following the design of experiment in Table 1. The geopolymer bricks were shaped in a mould of dimensions 160mm by 40mm by 40mm at a constant compaction pressure of 8MPa as suggested by Danso (2016). Upon demolding, the brick specimens were heat treated at 70°C in an oven for 24 hours and thereafter stored at room temperature for 28 days before performance evaluation.

Table 1: Design of experiment for chemical activation of diatomite

A:	B:	
Diatomite	Alkaline activator	
% wt	$\text{Na}_2\text{SiO}_3$ % wt	NaOH % wt
100	50	20

The Mitutoyo ABSOLUTE Digimatic Vernier Caliper (500 series), which has a 0.01 mm precision, was used to measure the dimensions of the brick samples. The compressive strength of the bricks was determined using a 50kN-WP 310 universal Materials testing machine, following the ASTM C109/C109M (2007) standard. According to ASTM-C642, (2013), the bulk density was determined. The water absorption experiment was conducted following ASTM C373-14, (1999).

Compressive strength, density and water absorption experimental tests were carried out since according to Teixeira et al. (2020), compressive strength and durability tests are regarded as key indicators of the viability of masonry.

### 3. Results and discussion

#### 3.1 Diatomaceous earth characterization

##### i. Chemical and physical analysis

The diatomaceous earth (DE) chemical analysis results are shown in Table 2.

Table 2: Chemical analysis results for diatomaceous earth

Specimen type	Chemical content (%)								
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	TiO <sub>2</sub>	MnO	Fe <sub>2</sub> O <sub>3</sub>	P <sub>2</sub> O <sub>5</sub>
<b>Diatomite (Raw)</b>	88.120	4.254	4.257	0.861	0.673	0.130	0.02	1.528	0.073

The findings of the chemical analysis of DE show that the tested material is an acidic rock with a dominant proportion of SiO<sub>2</sub> (88.12%) and relatively low contents of Al<sub>2</sub>O<sub>3</sub> (4.25%), CaO (4.26%), and Fe<sub>2</sub>O<sub>3</sub> (1.53%), with the content of each of the remaining oxides being below 1%. According to ASTM C618 (2014), the diatomite could be considered as a Class F normal type of pozzolan or a silicate glass material since its total content of SiO<sub>2</sub>, Fe<sub>2</sub>O<sub>3</sub>, and Al<sub>2</sub>O<sub>3</sub> was beyond 70% by weight with less than 10% CaO content. Nyale et al. (2014) clarified that a geopolymer binder is considered siliceous when the three key constituents, SiO<sub>2</sub>, Al<sub>2</sub>O<sub>3</sub>, and Fe<sub>2</sub>O<sub>3</sub>, total up to 70% or when their total and the reactive calcium oxide is less than 10%. The diatomite's low CaO level is proof that it can be used as a geopolymer precursor. According to Okeyinka et al. (2019), low-calcium binders are best for creating geopolymers because excessive calcium concentrations can slow down the polymerization-setting rate.

This indicates that the diatomite under study is an acidic rock belonging to the opal A + CT category, as described by Stefanou et al. (2022). The diatomite's alumina (Al<sub>2</sub>O<sub>3</sub>) content revealed that it wasn't clayey because it was lower than the 14–16% range suggested by the literature (Chen et al., 2020; Fragoulis et al., 2004; Stefanou et al., 2022; Yilmaz & Ediz, 2008). There were also trace levels of other oxides such as MgO, K<sub>2</sub>O, TiO<sub>2</sub>, MnO, Fe<sub>2</sub>O<sub>3</sub>, and P<sub>2</sub>O<sub>5</sub>.

The XRD analysis (Figure 2) showed that cristobalite was the predominant mineral in the Kenyan sampled diatomaceous earth. With reference to the classification done by Ejigu et al. (2022), the observed diffraction peaks are typical peaks for paracrystalline silica polymorph opal-CT derived from the volcanic environment.

The strongest reflection peak is at about 21.5°, with weaker peaks at around 29°, 32°, 36°, 45°, 57°, and 65°. The resulting diffraction peaks show the presence of  $\alpha$ -cristobalite together with variable degrees of stacking disorder, which causes maxima that are linked to tridymite.

The X-ray diffractometry (XRD) mineralogical finding strongly supported Kogel & Society for Mining (2006) and hypothesis that the Kenyan Rift Valley hosts diatomaceous earth deposits which appear to be of lacustrine origin (from lacustrine diatomite diagenesis) pre-dating one or more episodes of faulting and vulcanicity.

The XRD results were also in agreement with the XRF chemical analysis output which showed that the silica (SiO<sub>2</sub>) was the predominant chemical compound with a percentage of 88%.

Diatomites brutes (Coupled TwoTheta/Theta)

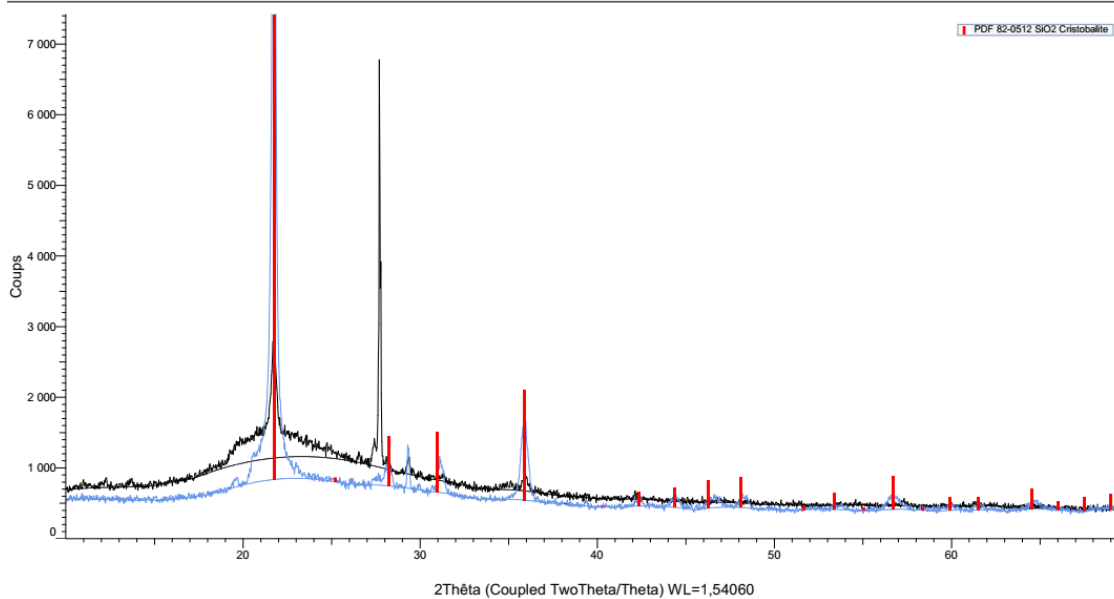


Figure 2. XRD result for diatomaceous earth.

The physical characteristics of the diatomaceous earth used in this study are shown in Table 3.

Table 3: Physical Analysis of diatomaceous earth

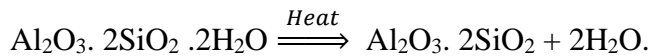
Specimen type	Physical property			
	Permeability (mD)	Porosity (%)	Specific gravity	Bulk density (g/cm <sup>3</sup> )
Diatomite (Raw)	2.3	40	2.1	1.4

The permeability of the diatomite was determined to be 2.3 mD and the porosity was 40%. The values obtained were within the ranges of 0.1-10 mD for permeability and 35-65% for porosity as provided by Reka et al. (2021). The specific gravity value falls within the acceptable range of 1-2.6 for organic soils as stated in the literature by Roy & Kumar Bhalla (2017).

ii. TGA-DSC thermal characterization of diatomaceous earth

Regarding the diatomite TGA analysis depicted in Figure 3, there was a significant mass loss which may have been caused by the release of both free and bound diatomite as well as any organic stuff that may have been present.

A small weight loss after 200 °C can be attributed to the dehydroxylation of OH-groups and the release of structural water from its impurities and amorphous silica structure to form the amorphous metakaolin, as explained by Ibrahim & Selim (2012) and illustrated by the below chemical reaction equation.



The weight loss starts to stabilize as soon as the temperature hits 800 °C, signifying the complete dehydration of the diatomite structure and the emergence of a new silicate substance. The total weight loss (loss on ignition) for the diatomite at 950 °C was 5.682 %. This resulting loss on ignition (LOI) value was still below the maximum value of 6% allowed by ASTM.C618 (2014).

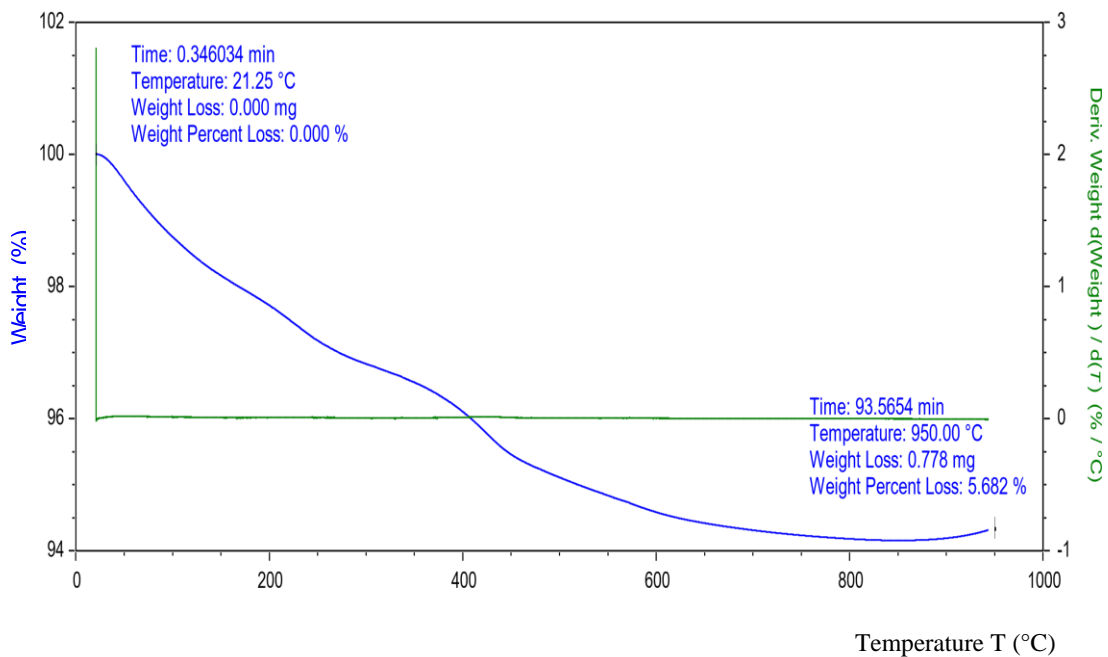


Figure 3. Weight loss analysis of raw diatomite



The DSC thermograms for the diatomite shown in Figure 4 revealed endothermic peaks between 120 °C and 280 °C, which are caused by the dehydration process (water evaporation), as a result of diatomite's high water absorption capacity. These peaks are the consequence of the separation of the opal component and the water that was bound to the diatomite particles.

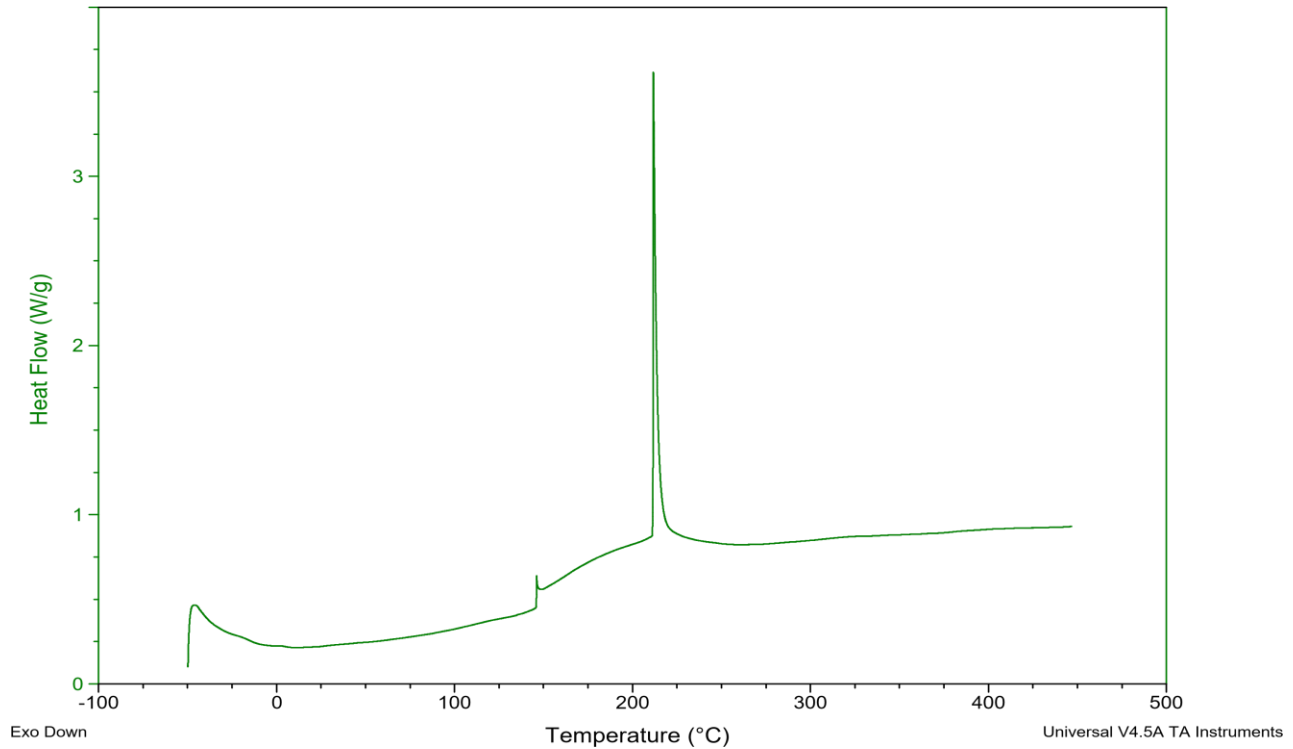


Figure 4. DSC analysis for raw diatomite

It is evident from the TGA experiment that diatomite is a thermally stable raw material and that its melting temperature is greater than 950 °C.

### iii. Analysis of diatomaceous earth particle size

The diatomite particle size distribution as obtained from the laser particle analyzer was Dv (10): 7.58 μm, Dv (50): 23 μm, and Dv (90) 50.4 μm. Relating to Osborne's (2013) analysis, the diatomite was found to be more similar to cement, in terms of particle size, since about 90% of its particles were smaller than 50.4 μm.

The particle size distribution of diatomaceous earth depicted in Figure 5 shows that the diatomite is a fine-grained earth material hence suitable for use as geopolymer precursor following the argument by Makusa (2012) that fine-grained granular materials are the easiest to stabilize due to their large surface area to their particle diameter.

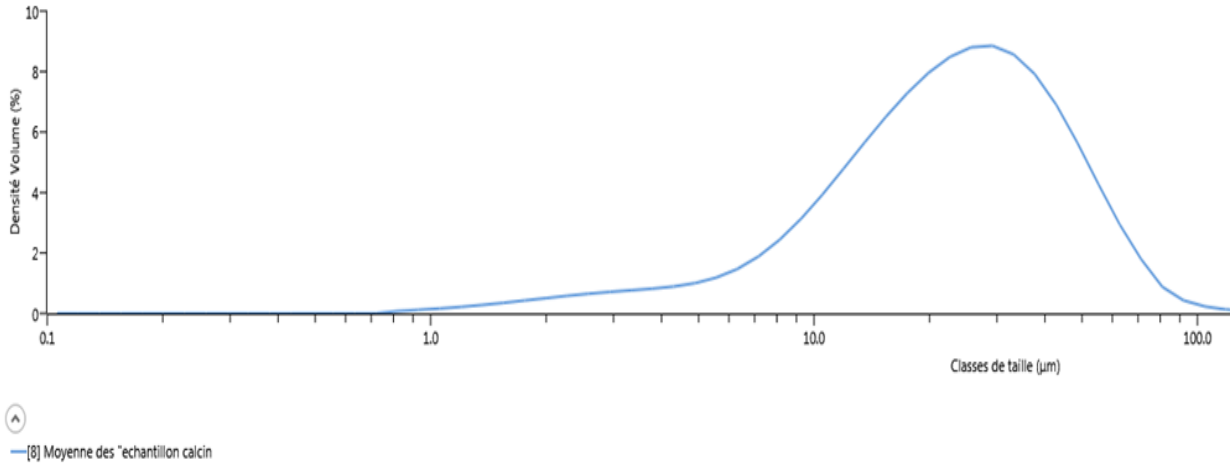


Figure 5. Raw diatomite particle size distribution

iv. The Atterberg limits test

The plastic limit (PL), liquid limit (LL), and plasticity index (PI) were obtained using the Atterberg limit test as described by ASTM D4318-17 (2017). Average values resulting from three (3) trials are presented in Table 4.

Table 4: Atterberg Limits Data

Item details	Raw Diatomite (grams)
Mass of empty can	20
Mass of can and wet soil	35.14
Mass of can and dry soil	30.79
Mass of dry soil	10.79
Mass of pore water	4.35
Water content, w%	40.34
No. of drops (N)	233
Plastic Limit (PL)	40.34
Liquid Limit (LL)	53
Plasticity Index (PI)	12.66

According to the data from the Atterberg limits experiment, the plasticity index for the diatomite was around 13. As suggested by Hall et al. (2012), diatomaceous earth employed in this study generally appears to be favourably good for usage as an earth construction material as its PI, is less than 16%.

Comparing the Atterbergs categorization described by Roy and Kumar Bhalla (2017) with the findings of this study reveals that raw diatomite is a cohesive and medium plastic silt clay.

### 3.2 Geopolymer performance evaluation

The results for the performance evaluation for the NaOH/Na<sub>2</sub>SiO<sub>3</sub>-activated diatomite geopolymer brick are presented in Table 5.

Table 5: Performance properties of NaOH/Na<sub>2</sub>SiO<sub>3</sub> activated diatomite

	28 <sup>th</sup> -day properties		
	Compressive Strength (MPa)	Density (g/cm <sup>3</sup> )	Water Absorption (%)
<b>Average</b>	<b>22.98</b>	<b>1.38</b>	<b>9.32</b>
min.	21.74	1.33	8.54
max.	24.33	1.43	10.00
std (+)	1.22	0.05	0.78
std (-)	1.36	0.05	0.68

A bar graph of the performance characteristics obtained for the created geopolymer concrete specimens is shown in Figure 6.

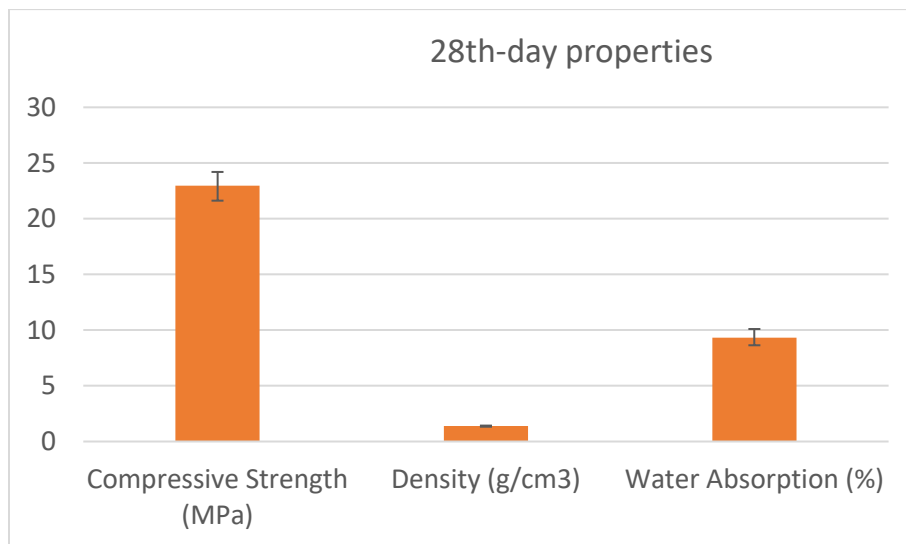


Figure 6: Performance properties of NaOH/Na<sub>2</sub>SiO<sub>3</sub> activated diatomite

The developed diatomite-based geopolymer's average 28-day compressive strength of 22.98 MPa falls within the permitted range of 8-115 MPa for light, normal, and heavyweight concrete as specified by European Standard EN 206-1 (EN 206-1, 2000).

The NaOH/Na<sub>2</sub>SiO<sub>3</sub> activated geopolymers had an average density of 1.38 g/cm<sup>3</sup>. The density values of the NaOH/Na<sub>2</sub>SiO<sub>3</sub> activated geopolymer were found to be within the permitted range of 1.2-2 g/cm<sup>3</sup> for lightweight concrete, according to Day et al. (2013). The low relative density and high porosity of diatomaceous earth could have contributed to the development of lightweight specimens. Based on the classification by TS EN 206 (2016), which defines lightweight concrete as having density of between 800 kg/m<sup>3</sup> and 2000 kg/m<sup>3</sup>, the produced geopolymer concrete can be categorically referred to as lightweight.

The water absorption for the diatomite-based geopolymer specimens was satisfactory based on both the Australian and Malaysian standards as provided by Ahmad et al. (2017). This is because the average water absorption value attained was less than the preset maximum limit of 20%.

#### **4. Conclusion**

This research sought to evaluate the viability of using diatomaceous earth from Nakuru, Kenya, as a resource for geopolymer concrete. It was determined that the diatomite under examination is an acidic rock falling within the opal CT category, according to the chemical composition, which revealed silica (SiO<sub>2</sub>) to be the major component, accounting for 88.12 percent of the sample. Therefore, the siliceous nature of Kenyan diatomaceous earth is of a rather high grade, comparable to a silicate glass material or a typical Class F kind of pozzolan. The Kenyan diatomaceous earth samples tested may have originated as biogenic silica opal-A before dissolving or re-forming as opal-CT as a result of thermal alteration of the rock caused by the high heat flow rates created in the rift zones by the penetration of dacite sills as a mechanism of the volcano-sedimentary succession.

The Atterberg limit examination and particle size analysis revealed that diatomaceous earth is a fine, cohesive, and medium silt material. Additionally, the diatomite is a material that is thermally stable and has a melting point greater than 950 °C, according to the TGA experiment. Moreover, significant diatomite loss on ignition (LOI), as determined by TGA, revealed that the material is porous, with low thermal conductivity and high thermal insulation capacity.

Importantly, the Kenyan diatomaceous earth can be successfully employed as a silica source in geopolymer concrete formulations, fostering the production of sustainable concrete.

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