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CHARACTERISATION OF RAW AND TREATED DE-OILED BLEACHING EARTH FOR SUPPLEMENTARY CEMENTITIOUS MATERIAL

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ABSTRACT: This study assessed the potential of raw and treated De-Oiled Bleaching Earth (DOBE) as a supplementary cementitious material (SCM) by analysing its characteristics. DOBE, originating from Spent Bleaching Earth (SBE), is a waste of the palm oil refining industry, often discarded in landfills, hence presenting environmental hazards. Study on DOBE for its potential as an SCM is still limited, particularly on how its characterisation impacts its reactivity, which in this study was evaluated through the Strength Activity Index (SAI) test. Apart from examining its general properties, raw and treated DOBE were also analysed using X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy (SEM), and laser diffraction. The treatment at 700°C for one hour improved its pozzolanic reactivity by raising its amorphous silica content from 56.7% to 64.7% and removing residual oil and volatile contaminants. The Strength Activity Index (SAI) tests indicated that the treated DOBE achieved 98% (20.71 MPa) of the control mix strength at 7 days and 107% (29.05 MPa) at 28 days, exceeding that of ordinary Portland cement (OPC). The findings validate that treated DOBE complies with ASTM C618 criteria for Class N pozzolans, establishing it as a suitable SCM. Nevertheless, issues such as loss on ignition, moisture content, and oil content must be resolved to enhance its performance. This study emphasises DOBE's capacity to improve cement sustainability by reducing its dependency on cement and mitigating environmental concerns. Future studies need to include the assessment on its durability, shrinkage, and ideal replacement ratios in concrete mixes.

Keywords: De-oiled bleaching earth, Supplementary cementitious materials, Strength activity index, Heat treatment

1. INTRODUCTION

In the past, pozzolanic materials have been considered as inferior cement alternatives utilised mainly for economic reasons, resulting in limitations on their utilisation. However, their advantages as supplementary cementitious material (SCM) are now generally acknowledged, especially in improving concrete mechanical properties [1]. According to ASTM C618, pozzolans are siliceous and alumina-rich materials that, when finely ground and exposed to moisture, react with calcium hydroxide ($Ca(OH)_2$) to form cementitious compounds [2].

The use of SCM as partial cement replacement reduces the environmental impact of Portland cement production by lowering raw material consumption and carbon dioxide (CO_2) emissions [1]. The cement clinker production emits over 800 kg CO_2 per tonne, contributing about 7% of global emissions. Portland pozzolana cement lowers emissions by roughly 20% compared to ordinary Portland cement [3]. SCM, including fly ash, silica fume, and blast furnace slag, improve concrete performance through the filler effect and pozzolanic reaction, where their siliceous and aluminous content reacts with $Ca(OH)_2$ to form calcium silicate hydrate (CSH) gel, enhancing strength and durability [4]. The fineness, morphology, and mineral composition of pozzolans significantly influence their reactivity, with finer particles providing greater surface area for reactions and increasing compressive strength [5]. X-ray fluorescence (XRF) and X-ray diffraction (XRD) are commonly used to analyse their chemical composition and crystalline phases. The Strength Activity Index (SAI) is an indirect method used to evaluate the pozzolanic efficiency that measures the physical properties of a sample with cement replacement levels typically not more than 30% to ensure mechanical performance [5], [6].

Various industrial and agricultural byproducts, including rice husk ash, palm oil fuel ash, sewage sludge ash, and waste glass powder, have been investigated as SCMs, with their performance significantly influenced through activation method [4]. Among these methods, heat activation is frequently used for silica-rich materials as it eliminates residual organics and enhances the formation of reactive amorphous silica. Research on Spent Bleaching Earth Ash (SBEA) indicates that calcination at temperatures ranging from 600 to 800 °C improves pozzolanic activity, but elevated temperatures over 900 °C can cause recrystallisation of amorphous silica and reduce reactivity [5]. This finding offers a valuable reference and justification for the use of thermal treatments in this study. While previous studies have demonstrated the benefits of calcined SBEA, there has been no systematic evaluation of De-Oiled Bleaching Earth (DOBE), particularly linking its mineralogical and chemical characteristics to reactivity through SAI testing. This study addresses this gap.

De-Oiled Bleaching Earth (DOBE) has recently been identified as a potential SCM due to its silica-rich composition. DOBE originates from Spent Bleaching Earth (SBE), a waste byproduct of palm oil refining, which presents environmental risks due to its widespread disposal in landfills. Malaysia produces more than 240,000 metric tonnes of SBE each year, posing concerns of fire and pollution [7]. Unlike SBE, studies on DOBE as an SCM are still limited. By linking its physical, chemical, and mineralogical properties to pozzolanic reactivity through SAI testing, it establishes a foundation for understanding DOBE’s performance, which provides novel validation of its potential as a pozzolan. Mixing pozzolans into concrete offers a sustainable substitute for conventional concrete, giving advantages in strength enhancement [8]. To enhance its pozzolanic properties, treatments such as thermal activation (300–900°C) [5], acid treatment, and prolonged grinding have been explored to increase the reactive amorphous silica content and eliminate unreactive components [9]. While thermal activation at 700 °C enhances the pozzolanic reactivity of DOBE by eliminating organics and increasing amorphous silica content, its energy demand must also be considered. However, this temperature is still significantly lower than cement clinkering (~1450 °C), implying lower CO_2 emissions compared to conventional cement production. According to ASTM C618, excessive pozzolanic replacement levels above 20% may increase the risk of autoclave expansion and compromise strength performance if not properly proportioned [2]. Moreover, it can lead to non-uniform reaction, microcracking, and higher shrinkage due to secondary hydration water desiccation [4]. Therefore, this study aims to characterise the properties of raw DOBE and DOBE treated at 700°C, focusing on its mineralogical transformation, pozzolanic activity, and suitability as a supplementary cementitious material. The findings of this research are expected to contribute to sustainable waste management by repurposing DOBE as an SCM, reducing cement consumption, and lowering carbon dioxide emissions. Additionally, optimising DOBE’s pozzolanic properties through thermal treatment may enhance concrete strength and durability, providing economic and environmental benefits to the construction industry. The results may serve as a foundation for future research on DOBE's application in cementitious technology.

2. RESEARCH SIGNIFICANCE

This research emphasises the importance of treating DOBE before its use as an SCM in construction. Heat treatment helps reduce residual oil and organic matter, improving DOBE’s quality and performance. The study compares raw and heat-treated DOBE to demonstrate how heat treatment enhances its suitability as an SCM. The findings support the use of treated industrial waste as a partial cement replacement, reducing landfill dependency and cement consumption while contributing to sustainability, resource recovery, and the development of environmentally friendly construction materials.

3. METHODOLOGY

The cement used in this study complies with CEM I standards and originated from Cement Industries (Sabah) Sdn. Bhd. River sand that was acquired locally from hardware stores in Kota Kinabalu was used as fine aggregate. It exhibited a specific gravity of 2.62 and a fineness modulus of 2.56, which complied with ASTM C33 standards.

DOBE was obtained from the Faculty of Engineering, UMS, provided by Gamalux Oils Sdn Bhd. The analysis examines the characteristics of both raw and treated DOBE to gain an understanding of its cementitious performance. In preparing the raw DOBE for testing, the sample was sieved using a 1.18 mm sieve. For the treated sample, the preprocessed raw material was further sieved using a 300-micron sieve before undergoing thermal activation, which involved heating at 700°C in a muffle furnace for one hour with a heating rate of 5 °C/min. The thermal treatment at 700 °C for one hour was selected based on previous studies on SBEA, which found that heating at this temperature improves the formation of amorphous silica and improves pozzolanic reactivity [5]. Both samples were then dried at 100°C for approximately 24 hours to evaluate their moisture content. The analysis of particle size distribution was conducted through laser diffraction, which gives the information on the particle size percentage and the specific surface area (SSA), while the specific gravity was measured according to ASTM C188 standards. The analysis of mineral composition utilised XRF, while crystalline phases were determined using XRD-Bruker D2 Phaser, and morphology was examined through scanning electron microscopy (SEM). The Loss on Ignition (LOI) was assessed in accordance with the ASTM C114 standard. Samples were weighed in a clay crucible and subjected to heating at 1000°C for 2 hours in a muffle furnace with a heating rate of 5 °C/min. After heating, the crucible was allowed to cool in the muffle furnace overnight and was reweighed until a steady mass was achieved. The LOI was determined as the percentage mass loss in relation to the initial sample weight, which was an average of 5 samples presented. The oil content was assessed through a modified Soxhlet method, where a cloth pouch was used in place of the traditional thimble to contain the sample due to equipment limitations. The extraction was performed on two particle size fractions of raw DOBE that passed through 1.18 mm and 0.3 mm sieves, respectively, and on the treated DOBE samples. This approach allows for a clearer understanding of the level of oil content with different size ranges for raw DOBE, alongside the treated DOBE sample.

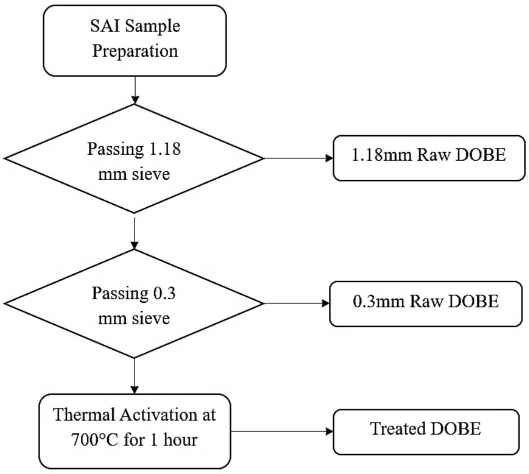

Fig. 1 SAI sample preparation

The evaluation of reactivity and pozzolanic attributes of DOBE was conducted through the SAI test in accordance with ASTM C311. Fig.1 illustrate the flow of sample preparation for SAI testing. Raw DOBE samples passing 1.18 mm and 0.3 mm sieves were tested to assess the effect of fineness on pozzolanic performance, while the treated DOBE sample was included to evaluate the impact of thermal activation. Finer and treated particles are generally expected to show different reactivity due to increased surface area and higher amorphous phase content. Three mortar cubes of 50mm x 50mm x 50mm were created with a 20% substitution of cement using raw and treated DOBE and compared against a control mix consisting of 100% OPC. Each mixture comprised 500g of cementitious material, 1350g of standard sand, and maintained a water-to-cementitious material ratio (w/c) of 0.484. The cementitious materials and sand were dry-mixed for 2 minutes, followed by gradual addition of water and mixing for a further 3 minutes using a laboratory mortar mixer. Three specimens were cast for each mix. ASTM C311 specifies 7 and 28 days curing periods with lime-saturated water at room temperature for assessing pozzolanic activity, and testing at these intervals provides a reliable foundation for verifying the reactivity of DOBE. Although longer curing durations could offer further insights into delayed pozzolanic reactions, they were beyond the scope of this study. This limitation is acknowledged, and extended curing evaluations are recommended for future research. The compressive strength of these samples was evaluated using a Matest compression testing machine, operated under the controlled loading rate of 2.4 kN/s. The result is then interpreted based on ASTM C618.

4. RESULTS AND DISCUSSIONS

4.1 Physical Properties of DOBE

4.1.1 Specific gravity and moisture content

Table 1 presents the physical properties of both raw and treated DOBE and OPC. The specific gravity of raw DOBE was recorded at 2.09, which increased to 2.43 after thermal treatment. The rise in specific gravity is due to the elimination of organic substances and volatile matter through the treatment process. This method increases the surface reactive area and enhances its reactivity [1]. The lower specific gravity of DOBE in comparison to OPC suggests a higher level of internal porosity. This similarity is observed in other supplementary cementitious materials, like fly ash, which exhibits a specific gravity of 2.2–2.6 [2] which increased the water demand [4].

Moisture content also influenced the performance of DOBE in concrete mixes. Raw DOBE sample exhibits a moisture content of 8.92%, compared to the 0.67% found in OPC. After the treatment, the moisture content of DOBE was totally eliminated. The raw DOBE surpasses the maximum moisture content limit of 3% set by ASTM C618 while treated DOBE comply with this requirement. Higher moisture content suggests that particles tend to support agglomeration [4]. By reducing its moisture levels, exposing reactive surface area subsequently enhances pozzolanic activity throughout the hydration process [6].

Table 1. Physical Properties of DOBE

Properties	OPC	Raw DOBE	Treated DOBE
Specific Gravity	3.01 ± 0.08	2.11 ± 0.02	2.43 ± 0.03
Moisture Content (%)	0.67 ± 0.47%	8.92 ± 0.05%	0%

4.1.2 Particle size analysis

The particle size distribution of both raw and treated DOBE are summarised in Table 2. A minor difference is noticed between raw and treated DOBE. Treated DOBE had a particle size D_{90} of 201.2 µm, while raw DOBE showed a finer particle size of 181 µm. This result aligns with the Specific Surface Area (SSA), showing that raw DOBE has a higher SSA than treated DOBE. The treatment suggested it has caused the tiny particles to cluster into conglomerates, causing particle consolidation and reducing micro voids [7]. During consolidation, smaller particles merge into bigger clusters, thereby diminishing surface irregularity and available interior porosity. This structural densification leads to a reduced SSA [8]. The reduction, however, is small and falls 2.93 m^3/kg from the standard deviation range of both samples, indicating only a marginal difference in fineness.

Table 2. Particle Size Distribution of Raw and Treated DOBE

Properties	Raw DOBE	Treated DOBE
Dv (10) (µm)	3.408 ± 0.02	3.446 ± 0.13
Dv (50) (µm)	27.4 ± 0.51	31.2 ± 1.45
Dv (90) (µm)	181 ± 13.3	201.2 ± 19.4
Specific Surface Area (m^2/kg)	629.06 ± 4.51	602.02 ± 19.6

Particle size distribution influenced the reactivity of pozzolans. ASTM C618 does not define specific limits for particle size distribution or surface area; however, it does state that at least 66% of the pozzolans must pass through a 45-micrometre sieve. While the results do not directly confirm compliance with this requirement, the treated DOBE exhibited a median particle size D50 of 31.2 µm, whereas the raw DOBE demonstrated a finer median particle size of 27.4 µm, indicating the raw and treated materials are slightly finer and it is likely that a substantial portion of each material would meet the 66% passing criterion as illustrated in Fig.2a and Fig.2b.

Finer particles, which have a greater surface area, enhance the interaction between SiO_2 and $Ca(OH)_2$. This promotes the formation of secondary CSH gel, which contributes to the strength and durability of the material [1], [2]. SCM with finer particle sizes have an enhanced pozzolanic activity due to the increased reactive surface area. Treated DOBE produces better packing within the concrete matrix, minimising voids and permeability [8]. This results in a more compact structure, improving the durability of the hardened concrete [4]. SCM with a D50 of under 20µm facilitates accelerated strength development relative to those with higher particle sizes [10].

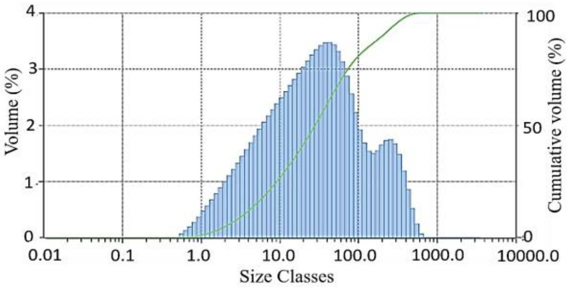

Fig. 2a Particle Size Distribution of Raw DOBE

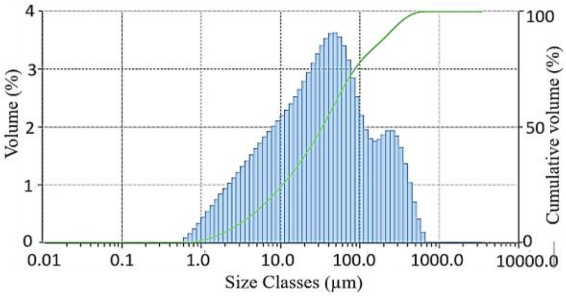

Fig. 2b Particle Size Distribution for treated DOBE

4.2 Chemical Properties of DOBE

4.2.1 Chemical composition

ASTM C618 states that Class N, F, or C pozzolans must contain at least 70% combined silicon dioxide (SiO_2), aluminium oxide (Al_2O_3), and iron oxide (Fe_2O_3). The raw and treated DOBE samples showed combined contents of 70.19% and 70.06%, respectively, achieving the requirement as shown in Table 3. The high SiO_2 content (51.27%) serves as a crucial component for the formation of CSH gel, which is the main factor in strength development in the pozzolanic system. Additionally, the increased Al_2O_3 (10.03%) of treated DOBE is expected to enhance pozzolanic activity, particularly in promoting the secondary hydration reactions [4], [11]. ASTM C618 states a maximum sulphur trioxide (SO_3) content of 5% to ensure durability and avoid excessive expansion. The SO_3 content of the treated DOBE sample is 3.31%. Limiting SO_3 in cement materials prevents sulphate attack, which can cause concrete to expand and crack [7].

Table 3. Oxides Composition of DOBE

Chemical Composition (%)			
Sample	Raw DOBE	Treated DOBE	ASTM C618 Requirement
SiO_2 %	51.20	51.27	-
Al_2O_3 %	9.61	10.03	-
Fe_2O_3	9.38	8.76	-
SO_3	3.18	3.31	<4
Na_2O	0.26	0.25	-
K_2O	1.59	1.56	-
CaO	12.42	12.14	-
MgO	6.30	6.83	-
P_2O_5	4.43	4.29	-
TiO_2	1.02	0.99	-
Mn_2O_3	0.21	2.00	-
Combine (S+A+F)	70.19	70.06	>70
Na_2O_{eq}	1.31	1.28	-
Class	N	N	N

Note: Values are based on single XRF determinations (n = 1). As such, standard deviations were not available.

4.2.2 Loss on Ignition (LOI)

Loss on Ignition (LOI) is a main parameter in evaluating the purity and suitability of pozzolanic materials. A high LOI indicates the presence of impurities such as unburned carbon and volatile substances [7], [9], which can negatively impact hydration and reduce the pozzolanic efficiency of DOBE. The LOI of raw and treated DOBE was analysed to assess its suitability as a pozzolanic material. ASTM C618 outlines a maximum loss on ignition LOI of 10% for natural pozzolans and 6% for fly ash [12].

The results in Fig.3 show that the raw DOBE has a high LOI of 27.5%. This indicates that the raw DOBE contain a significant amount of organic matter and residual impurities, which can negatively impact its performance as an SCM. The presence of unburned carbon in raw DOBE can interfere with the hydration process by adsorbing water and reducing the availability of free water needed for cementitious reactions. Additionally, high LOI materials tend to have lower pozzolanic reactivity, as the impurities hinder the formation of CSH, which is essential for strength development in concrete.

The thermal treatment at 700 °C plays a crucial role in reducing the LOI of DOBE by decomposing and volatilising organic matter, residual oil, and unburned carbon. Thermal treatment effectively reduces the LOI of DOBE from 27.5% to 1.25%, significantly lowering the presence of volatile impurities and unburned carbon. The reduction in LOI improves the pozzolanic activity of DOBE, allowing it to better contribute to the secondary hydration reactions when used as an SCM. On the other hand, OPC recorded an LOI of 3.67%, which is significantly lower than the 27.5% observed in raw DOBE but slightly higher than the 1.25% recorded in treated DOBE. This suggests that treated DOBE has fewer volatile impurities than OPC, potentially making it an even more stable material in terms of hydration. This improves the strength and durability of concrete [4],[13]. A lower LOI enhances the incorporation of SCMs within the cement matrix. This reduces porosity and enhances compressive strength [14].

4.2.3 Oil Content

DOBE is a waste material derived from the palm oil refinery process, and as such, it may contain traces of impurities, including residual oil. As shown in the LOI analysis (Section 4.2.2), untreated DOBE exhibits a high LOI content, indicating a significant amount of volatile impurities. However, upon thermal treatment, the LOI is significantly reduced, suggesting the removal of these impurities. Soxhlet extraction was conducted on both untreated and thermally treated samples to further verify the presence of oil in DOBE.

The results presented in Fig.4 show that 1.18 mm raw DOBE has 0.194% oil content, while 0.3 mm raw DOBE has 0.187% oil. The slightly lower oil content in finer particles may be attributed to a larger surface area-to-volume ratio, which promotes the volatilisation of oil during the refining process. Residual oil in DOBE can create a layer on DOBE particles, hindering their proper reaction during hydration, which is crucial for pozzolan reactivity. Oil reduces the bond strength between DOBE particles and the cement matrix, resulting in higher porosity, lower compressive strength, and long-term durability problems like cracking [9].

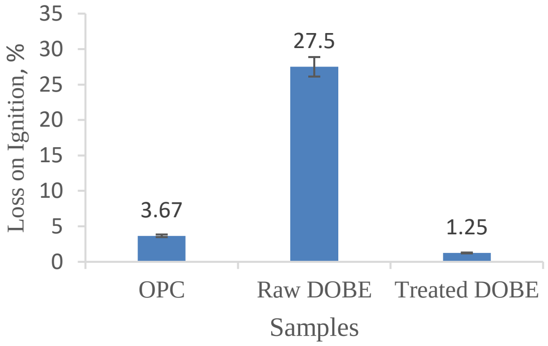

Fig.3 LOI Result

After thermal treatment at 700°C, all residual oil is eliminated, regardless of particle size. However, while oil is completely removed, the LOI of treated DOBE remains at 1.25%. This indicates that, besides oil, untreated DOBE contains other volatile impurities, such as moisture, organic matter, and possibly unburned carbon, which contribute to its high LOI. Such a reduction indicates that calcination effectively removes combustible impurities, thereby enhancing the purity of DOBE. By eliminating residual oil, treated DOBE enhances concrete performance by allowing full hydration and proper integration into the cement matrix through the elimination of oil content.

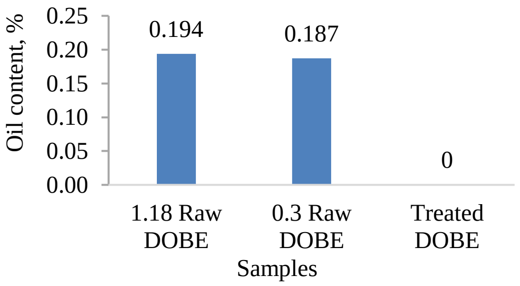

Fig.4 Residual Oil Content in Raw and Treated DOBE Samples

4.3 Mineralogy Properties of DOBE

The mineralogical assessment of raw and treated DOBE, performed using X-ray diffraction (XRD) with a wavelength of 1.54060 across a 2θ range of 5° to 80°, shows significant changes in composition after thermal treatment, as in Table 4 below.

Raw DOBE primarily comprises coesite at 33.8% and calcite at 34.9%, both of which are crystalline phases. A minor quantity of periclase, present at 0.9%, may contribute to long-term strength through gradual hydration, forming $Mg(OH)_2$, which reacts with SiO_2 to form magnesium silicate hydrate (MSH) [14]. However, excessive MgO can lead to expansive reactions and the formation of non-cementitious magnesium silicate hydrates, which may compromise strength and durability if not properly controlled [14]. Reactive phases, like lime and mullite, are not detected in raw DOBE.

Table 4. Mineralogical Composition of Raw and Treated DOBE Determined by XRD Analysis

Mineral Phase	Raw DOBE (%)	Treated DOBE (%)
Coesite (SiO_2)	33.8	18.5
Calcite ($CaCO_3$)	35	12.5
Quartz (SiO_2)	-	12.8
Lime (CaO)	-	0.5
Mullite ($Al_2O_3 \cdot SiO_2$)	-	1.4
Periclase (MgO)	0.9	1
Potassium Carbonate (K_2CO_3)	-	2

After treatment, the mineral composition undergoes significant changes. Calcite drops to 12.5% and coesite decreases to 18.5%; however, quartz appears at 12.8%. Lime, detected at 0.5%, contributes to the development of CSH. When CaO reacts with water, it forms CH, which is a key component in the pozzolanic reaction that produces CSH gel [15]. The presence of 1.4% mullite suggests the formation of potentially reactive aluminosilicates [9]. MgO is maintained at 1.0%, facilitating incremental strength development [16]. Potassium carbonate (2.0%) is produced, enhancing alkalinity and facilitating pozzolanic activation [17].

Table 5. Amorphous and Crystalline Phases of DOBE

Phase	DOBE Raw	DOBE Treated
Amorphous	56.7%	64.7%
Crystalline	43.3%	35.3%

The transformation in the mineralogical phases of raw and treated DOBE is shown in Table 5. The amorphous and crystalline contents of raw and treated DOBE were determined using the automatic quantitative phase analysis function of the XRD system (XRD-Bruker D2 Phaser). Raw DOBE is composed of 56.7% amorphous phase and 43.3% crystalline phase. After thermal treatment, the amorphous content of treated DOBE increased to 64.7%, while the crystalline phase dropped to 35.3%. The change in phase composition plays a crucial role in enhancing the pozzolanic properties of DOBE, as the amorphous phase exhibits greater reactivity than its crystalline counterpart. Amorphous materials possess a disordered atomic structure that facilitates dissolution and reaction with $Ca(OH)_2$ during cement hydration [7], [18]. CSH forms when amorphous silica reacts with $Ca(OH)_2$ during hydration [11].

The XRD pattern of raw DOBE in Fig.4a has several sharp peaks, especially within the 2θ range of 20° to 30°, indicating the presence of clear crystalline phases. The high and thin intensity peaks justify the crystalline characteristics of the material, together with a reduced background hump, indicating the existence of an amorphous component.

On the other hand, the XRD pattern of treated DOBE, seen in Fig.4b, exhibits a notable decrease in peak intensity and sharpness. The peaks seem wider and less defined, accompanied by an elevated background hump, indicative of amorphous formations. The change in the diffraction profile indicates a conversion of some crystalline phases into amorphous states during thermal treatment. Thermal treatment converts crystalline silica to an amorphous state, enhancing its ability to mix with cement and engage in hydration, making treated DOBE a more effective supplementary cementitious material [9]. The reduction in crystalline content from 43.3% to 35.3% indicates a reduction in nonreactive phases, which enhances the pozzolanic potential of DOBE. Crystalline phases like quartz are largely inactive and do not significantly contribute to pozzolanic reactions [7]. These findings align with existing literature showing that materials with lower crystallinity and higher amorphous content perform better in cementitious applications [18].

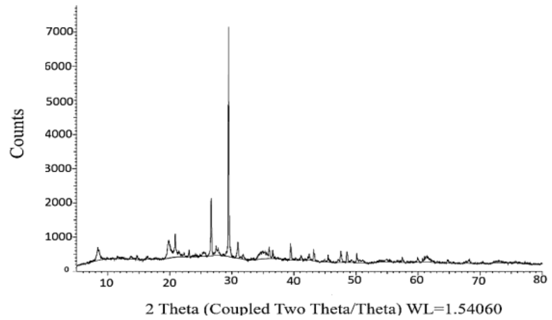

Fig.5a XRD result for Raw DOBE

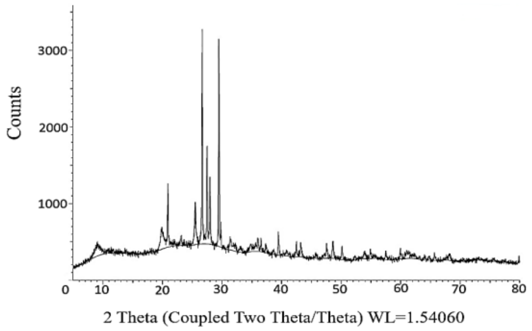

Fig.5b XRD Result for Treated DOBE

4.4 Morphology Properties of DOBE

Thermal treatment significantly alters the surface texture, particle arrangement, and microstructural characteristics of DOBE, which directly impacts its pozzolanic activity. The differences between raw and treated DOBE, as observed in Scanning Electron Microscopy (SEM) images, highlight the influence of heat treatment on the material's structure and its potential for enhanced cementitious performance. Fig. 6a and Fig. 6b illustrate the morphological characteristics of raw DOBE at 100 µm and 20 µm magnification, which display irregular, angular particles characterised by rough surfaces. The particle arrangement has a loose packing arrangement with significant voids and varying sizes, resulting in reduced packing efficiency. These characteristics, along with organic impurities and residual carbon, constrain the reactive surface area for pozzolanic activity and hinder effective hydration in cementitious systems [11].

After thermal treatment, the SEM images of treated DOBE, as shown in Fig.7a and Fig.7b, exhibit a more refined and consolidated microstructure. Thermal treatment enhances particle surface definition and encourages particle aggregation into a denser structure [9]. This aligns with the observed slight increase in median particle size (Section 4.1.2), which suggests that smaller particles might have fused or agglomerated, forming larger, denser structures. Furthermore, the removal of organic contaminants and the conversion of crystalline structures into reactive amorphous silica improves pozzolanic reactivity [19].

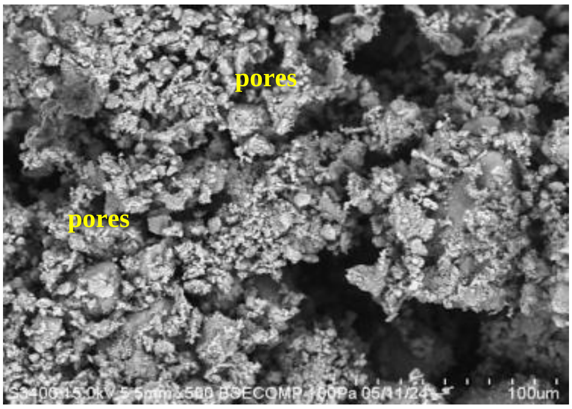

Fig. 6a Microstructural image of Raw DOBE at 100 µm magnification

4.5 Strength Activity Index (SAI) of DOBE

The pozzolanic reactivity of raw and treated DOBE is determined using the Strength Activity Index (SAI). It is determined by comparing the compressive strength of mortar containing DOBE as a partial cement replacement with that of a control mix made solely of cement. A higher SAI indicates greater pozzolanic activity and improved cementitious properties.

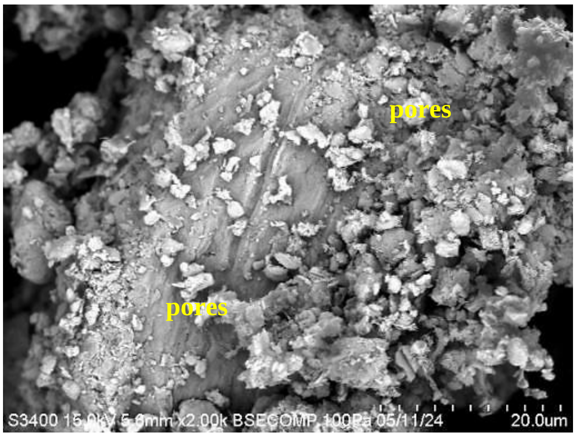

Fig. 6b Microstructural image of Raw DOBE at 20 µm magnification

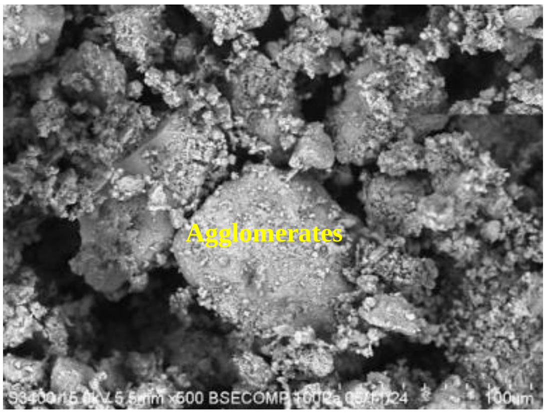

Fig.7a Microstructural image of Treated DOBE at 100 µm magnification

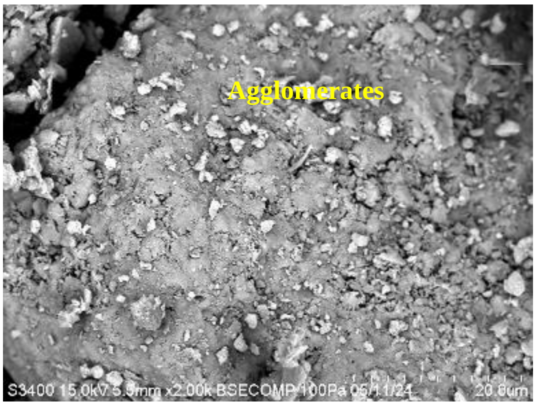

Fig.7b Microstructural image of Treated DOBE at 20 µm magnification

Although no direct hydration study was performed, the increase in SAI suggests that it contributes to secondary hydration reactions. In this way, pozzolanic activity can be indirectly correlated to improved hydration [20]. This section evaluates the SAI of raw and thermally treated DOBE to understand the impact of particle size, impurities, and thermal treatment on its reactivity.

Table 6. Strength Activity Index (SAI) Result

Curing Age	Compressive strength, MPa, (SAI)	
	7 Days	28 Days
Control	21.11 ± 0.45 (100%)	27.21 ± 0.70 (100%)
1.18mm Raw DOBE	11.06 ± 0.15 (52%)	14.07 ± 0.38 (52%)
0.3mm Raw OBE	11.99 ± 0.37 (57%)	15.76 ± 0.52 (58%)
Treated DOBE	20.64 ± 1.39 (98%)	29.21 ± 2.54 (107%)

The control mix reaches 100% strength at both 7 and 28 days, serving as a benchmark for comparison, as indicated in Fig.8a and Fig.8b, respectively. In contrast, raw DOBE passing a 1.18mm sieve exhibits a notably lower SAI, reaching just 52% of the control mix strength at both curing ages. Similarly, raw DOBE passing through a 0.3 mm sieve shows only a minor improvement, reaching 57% and 58% of the control mix strength at 7 and 28 days, respectively. According to ASTM C618, a pozzolan must achieve at least 75% of SAI compared to OPC to be considered an effective SCM, meaning both DOBE samples fall below the required threshold. The slight strength increase in the finer DOBE sample (0.3 mm) is attributed to its higher specific surface area, which enhances reactivity [9][21].

The poor performance of raw DOBE is associated with oil content, organic impurities, and residual carbon, as shown by high LOI and SEM images. These traits hinder the pozzolanic reaction and diminish the material's ability to improve hydration [9], [11]. Table 3.5 indicates that the oil content in the 0.3 mm DOBE sample is 0.187%, slightly lower than the 0.194% oil content in the 1.18 mm sample, which explains the minor improvement in pozzolanic performance. Despite this, pozzolanic reactivity remains constrained by the presence of crystalline phases and impurities in the raw material, further limiting its effectiveness as an SCM [10], [7].

Treated DOBE demonstrates a notable increase in reactivity, achieving 98% of the control mix strength at 7 days and, importantly, surpassing the control at 28 days with 107%. This result clearly confirms that treated DOBE meets and exceeds the ASTM C618 requirement of 75% and validates its viability as a supplementary cementitious material (SCM). Thermal treatment significantly enhances the material by removing organic matter, eliminating oil content, reducing LOI to 1.25%, and increasing amorphous silica content. These changes improve packing density, boost reactive site availability, and enable better interactions with $Ca(OH)_2$, resulting in C-S-H formation. Studies have shown that calcination effectively enhances the pozzolanic reactivity of waste-based materials, including Spent Bleaching Earth Ash (SBEA) and eco-processed pozzolans (EPP), by increasing their amorphous silica content and reducing impurities [9], [16]. The strength increases beyond 100% at 28 days shows the continued pozzolanic reaction of treated DOBE, improving its role as a supplementary cementitious material.

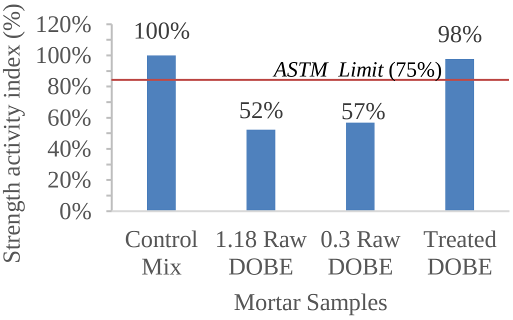

Fig.8a Strength Activity Index (SAI) of Raw and Treated DOBE (7 days)

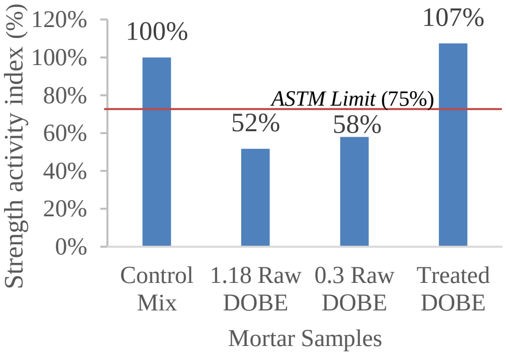

Fig.8b Strength Activity Index (SAI) of Raw and Treated DOBE (28 days)

5. CONCLUSION

Based on the results and analysis, several conclusions can be drawn. Treated DOBE showed a higher specific gravity of 2.43 compared to raw DOBE at 2.09, indicating the effective removal of organic matter and an increase in particle density. The moisture content of treated DOBE was reduced to 0%, while the loss on ignition (LOI) significantly decreased from 27.5% to 1.25%, suggesting the elimination of carbon and other volatile impurities.

Particle size analysis revealed a slight increase in the particle size of treated DOBE, with Dv(90) measured at 201.2 µm compared to 181 µm for raw DOBE. This is likely due to the agglomeration of finer particles during the treatment process. Despite this, the treated DOBE retained a particle size distribution suitable for use as a supplementary cementitious material.

Chemical analysis through X-Ray Fluorescence (XRF) confirmed that both raw and treated DOBE fulfilled the requirements for Class N pozzolans under ASTM C618, as the combined content of SiO_2, Al_2O_3, and Fe_2O_3 exceeded 70%. Furthermore, X-Ray Diffraction (XRD) analysis showed an increase in the amorphous phase content from 56.7% in raw DOBE to 64.7% in treated DOBE, indicating enhanced pozzolanic potential due to thermal treatment.

Microstructural analysis using Scanning Electron Microscopy (SEM) demonstrated that the treated DOBE developed a denser and more refined structure compared to the raw sample. This structural improvement can contribute to better interaction with cementitious matrices. Additionally, Soxhlet extraction testing confirmed complete removal of residual oil, which is essential to prevent interference with cement hydration reactions.

Finally, the reactivity of treated DOBE was significantly improved. It achieved 98% of the control mix strength at 7 days and exceeded the control at 28 days with a strength of 107%. Exceeding 100% SAI at 28 days not only confirms its pozzolanic efficiency but also provides strong evidence that treated DOBE is a viable and competitive SCM for construction applications. In contrast, raw DOBE showed poor reactivity, reaching only 52–57% of the control strength at 7 days and 52–58% at 28 days. These findings affirm the potential of treated DOBE as a viable SCM in construction applications, as demonstrated by its SAI performance exceeding 100% at 28 days. These SAI results provide indirect evidence for higher pozzolanic reactivity, which is associated with increases in secondary hydration. Although it is important to note that this study was limited to a curing period of 28 days and did not include direct hydration analyses, while comparisons with other SCMs and cost assessment were beyond its scope and are recommended for future study.

6. ACKNOWLEDGMENTS

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