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Utilisation of De-Oiled Bleaching Earth as Fine Filler in Lightweight Foam Concrete: Material Characteristics and Curing-Dependent Strength

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ABSTRACT

Lightweight Foam Concrete (LFC) relies on thin cementitious walls surrounding entrained air voids, and their continuity strongly influences mechanical performance. This conference study evaluates De-Oiled Bleaching Earth (DOBE) as a fine filler in an LFC trial formulation containing 3.00 kg cement, 3.70 kg DOBE, 0.25 kg silica fume, 5.00 L water and 150 mL concentrated concrete foaming agent. Raw DOBE was characterised using X-Ray Fluorescence (XRF), X-Ray Diffraction (XRD), Field-Emission Scanning Electron Microscopy with Energy-Dispersive X-Ray Analysis (FESEM-EDX), and Atomic Force Microscopy (AFM). Brick specimens were subjected to steam curing at 70 °C for 48 h followed by 24 h ambient cooling, or to water and open-air curing for 28 days. XRF identified SiO_2 as the dominant oxide (50.79 wt.%), with CaO, Fe_2O_3 and Al_2O_3 also present. FESEM and AFM revealed irregular agglomerates and non-uniform nanoscale topography, with Ra = 0.971 nm and Rq = 1.24 nm. EDX showed predominantly siliceous-aluminous particles together with a local Ca-rich region. Steam-cured specimens achieved 5.75 MPa, compared with 5.42 MPa for water curing and 5.14 MPa for open-air curing. The results support DOBE as a physically and chemically heterogeneous fine filler and indicate that steam curing can accelerate strength development in DOBE-containing LFC.

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Introduction

Lightweight Foam Concrete (LFC) combines a low unit weight with good thermal and acoustic performance, making it suitable for blocks, partitions and other non-structural or semi-structural components [1-4]. Its mechanical capacity is governed by the solid paste skeleton, particularly the thin walls separating adjacent air voids. Discontinuity, insufficient hydration or local defects in these walls can reduce compressive strength even when the overall material remains dimensionally stable.

Industrial by-products are increasingly introduced into cement-based materials to reduce reliance on virgin resources and to modify particle packing or binder chemistry [5]. De-Oiled Bleaching Earth (DOBE) is generated after residual oil is removed from spent bleaching earth used in palm-oil refining. Previous work on spent bleaching earth ash, processed spent bleaching earth and thermally treated DOBE indicates that these materials can exhibit filler or supplementary cementitious behaviour, although performance depends strongly on mineralogy, residual organic content, fineness, treatment and dosage [6-9]. The use of untreated DOBE specifically as a fine filler in LFC remains comparatively underreported.

This study provides a conference-level assessment of a DOBE-containing LFC trial mix. The objectives were to:

(i) Establish the chemical and surface characteristics of raw DOBE using XRF, FESEM-EDX and AFM;
(ii) Compare compressive strength under accelerated steam curing and conventional water and open-air curing;
(iii) and Discuss, without overclaiming, how DOBE morphology and local composition may influence the cementitious walls surrounding air voids.

Experimental

Materials and Trial Mix

Ordinary Portland Cement (OPC) Type I, 52.5N was used as the principal binder. DOBE was incorporated as a fine filler rather than as a cement replacement. Silica fume, tap water and a commercial concentrated concrete foaming agent were also used. The as-received DOBE consisted of agglomerated solids; material selected for characterisation was crushed and sieved below 150 micrometres. The trial proportions are summarised in Table 1.

Table 1: Trial Formulation of DOBE-Containing LFC

Constituent	Quantity	Function
Ordinary Portland cement	3.00 kg	Primary binder
DOBE	3.70 kg	Fine filler
Silica fume	0.25 kg	Fine supplementary powder
Water	5.00 L	Mixing water
Concrete foaming agent	150 mL	Concentrated foaming agent

The water-to-total-powder ratio was approximately 0.72, based on cement, DOBE and silica fume.

Specimen Preparation and Curing

The cement, DOBE and silica fume were dry-mixed before water was added to form a uniform slurry. The measured concentrated foaming agent was then incorporated during mixing, and low-speed mixing was used to limit excessive foam collapse. The fresh mixture was placed in 100 × 200 × 60 mm brick moulds and levelled with minimal disturbance.

After demoulding, the specimens were divided into three curing groups. For accelerated curing, the steam chamber was preheated to 70 °C before the specimens were introduced. These specimens were steam-cured for 48 h, removed and cooled under ambient laboratory conditions for 24 h before testing. The other groups were subjected to water curing or open-air curing for 28 days.

Testing and Characterisation

Compressive strength was determined using a calibrated compression testing machine in accordance with BS EN 772-1. A minimum of three specimens was tested for each curing group, and the mean strength was reported. XRF was used to determine the oxide composition of DOBE. XRD was used to identify the principal crystalline phases and to obtain a semi-quantitative indication of crystalline and amorphous content. FESEM images were obtained at magnifications between ×5,000 and ×30,000, while EDX point analysis was conducted at five particle locations. AFM was performed over a 5 × 5 µm scan area, and the average roughness (Ra), root-mean-square roughness (Rq) and maximum height range (Rmax) were extracted.

Results and Discussion

Chemical Composition, Mineralogy and Local Elemental Variability

The major XRF oxides are listed in Table 2. DOBE was dominated by SiO2 (50.79 wt.%), followed by CaO (12.72 wt.%), Fe2O3 (9.56 wt.%) and Al2O3 (9.54 wt.%). The composition confirms a siliceous and aluminous material containing appreciable calcium- and iron-bearing constituents. For this trial, the result is more appropriately interpreted as evidence of a chemically heterogeneous fine filler than as proof of pozzolanic performance, because no direct reactivity test was conducted.

Table 2: Major Oxide Composition of DOBE Determined by XRF

Oxide	Content (wt.%)	Oxide	Content (wt.%)
SiO2	50.79	CaO	12.72
Fe2O3	9.56	Al2O3	9.54
MgO	6.05	P2O5	4.47
SO3	3.25	K2O	1.61

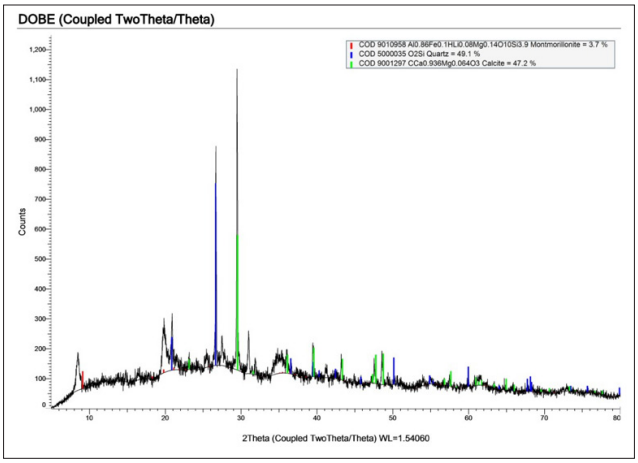

Figure 1: XRD Pattern of DOBE with the Principal Identified Crystalline Phases

XRD identified quartz and calcite as the dominant crystalline phases, together with a minor montmorillonite contribution. The instrument software also estimated a substantial amorphous fraction of approximately 74.6%. Because this estimate was obtained directly from the software output and no independent reactivity test was performed, it should be treated as semi-quantitative. The combination of crystalline silica, carbonate-bearing phases and an amorphous background supports a mixed filler composition, but it does not by itself confirm pozzolanic reaction.

EDX provided local rather than bulk composition. Four analysed points were Si-rich, with 17.3-21.8 wt.% Si and variable Al and Fe contents, whereas Spectrum 4 contained 33.8 wt.% Ca and only 4.7 wt.% Si (Table 3 and Figure 2). This contrast indicates the coexistence of siliceous-aluminous particles and local calcium-rich phases. Carbon was excluded from interpretation because the specimen mounting medium may contribute to the measured signal.

Table 3: Selected EDX Elemental Contents at Five DOBE Particle Locations

Spectrum	Si (wt.%)	Al (wt.%)	Fe (wt.%)	Ca (wt.%)	Interpretation
1	19.2	2.0	4.4	0.7	Siliceous
2	21.8	6.2	1.8	0.5	Si-Al-rich
3	17.3	4.3	3.4	0.6	Mixed siliceous
4	4.7	1.0	1.3	33.8	Ca-rich
5	20.7	6.7	2.2	0.6	Si-Al-rich

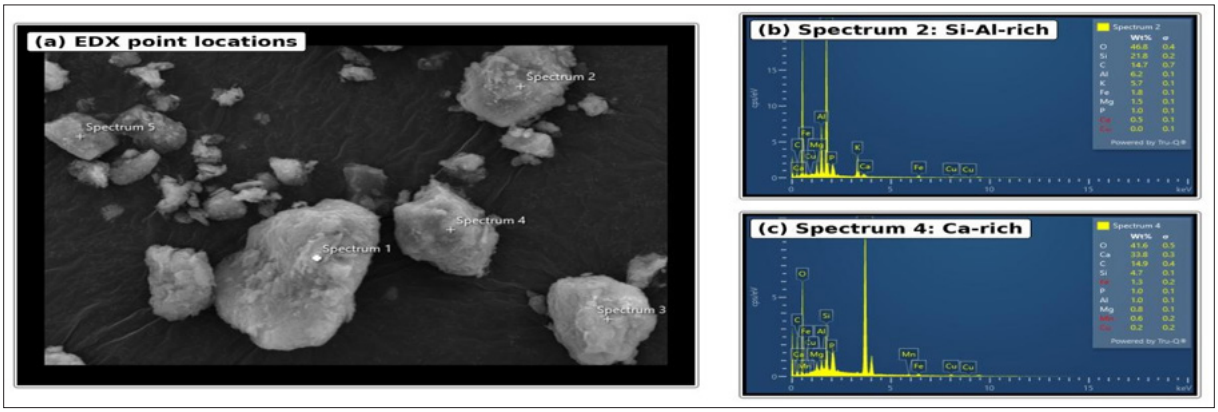

Figure 2: EDX Analysis of DOBE: (a) Point Locations, (b) Representative Si-Al-Rich Spectrum 2 and (c) Representative Ca-Rich Spectrum 4

Surface Morphology and Nanoscale Topography

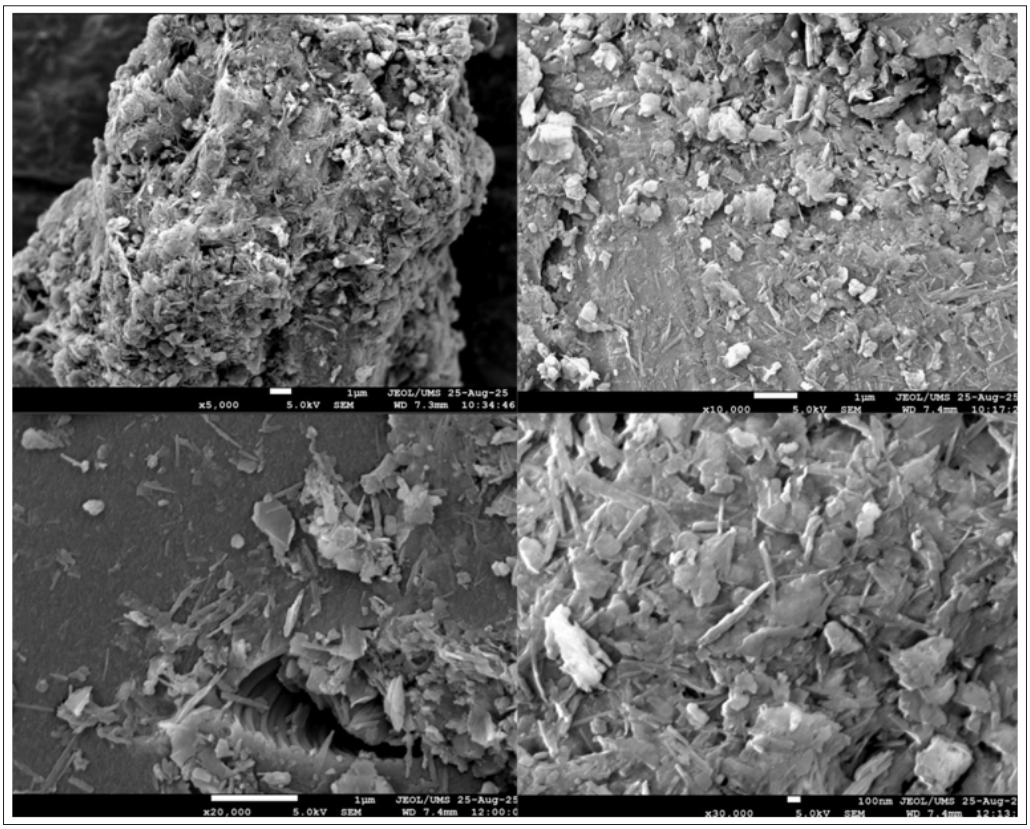

Figure 3: FESEM Micrographs of DOBE at ×5,000, ×10,000, ×20,000 and ×30,000

FESEM revealed strongly agglomerated DOBE particles comprising irregular flakes, plate-like fragments and smaller elongated features. Inter-particle gaps and attached fines were visible across the magnification range. The morphology suggests two competing effects in LFC: sufficiently dispersed fine particles may occupy spaces within the paste skeleton, whereas agglomerates may increase water demand or create local discontinuities if they are not adequately wetted and dispersed.

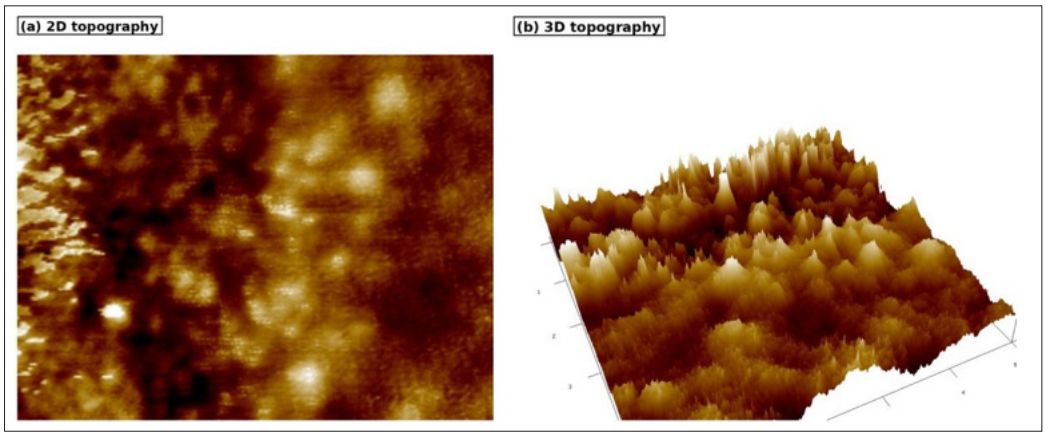

Figure 4: AFM Topography of DOBE over a 5 × 5 µm scan area: (a) Two-Dimensional Height Image and (b) Three-Dimensional Surface Representation

AFM confirmed non-uniform nanoscale topography within the scanned region. The whole-image values were Ra = 0.971 nm, Rq = 1.24 nm and Rmax = 9.95 nm. These measurements support the FESEM observation of a textured surface, although they do not independently establish chemical reactivity. The surface condition may influence wetting and local contact with the cementitious phase, but this effect remains dependent on dispersion and available mixing water.

Compressive Strength Under Different Curing Regimes

Table 4: Mean Compressive Strength Under Accelerated and Conventional Curing

Curing regime	Duration and conditioning	Mean strength (MPa)
Steam curing	70 °C for 48 h + 24 h ambient cooling	5.75
Water curing	28 days	5.42
Open-air curing	28 days	5.14

The steam-cured specimens recorded the highest mean compressive strength of 5.75 MPa, approximately 6.1% above water curing and 11.9% above open-air curing. The result is notable because the steam group was tested after 48 h of heating and 24 h of cooling, whereas the other groups were tested after 28 days. The comparison therefore demonstrates accelerated strength development rather than equal-age superiority.

At 70 °C, elevated temperature and retained moisture can accelerate early cement hydration and promote faster formation of a continuous solid skeleton. Water curing produced the intermediate result, consistent with sustained moisture availability, while open-air curing gave the lowest strength, likely because uncontrolled moisture loss restricted continued hydration [10,11]. The strength differences were modest and should not be interpreted as statistical significance because only mean values are reported.

Implications for the Foamed Paste Matrix

In LFC, external load is transmitted through the thin paste walls surrounding the air voids. The FESEM-AFM results indicate that DOBE may contribute physically by occupying local spaces and increasing solid contact where particles are well dispersed. Its EDX and XRF compositions also indicate chemical compatibility with a Ca-Si-Al-bearing cementitious environment. Conversely, particle agglomeration and water demand may interrupt paste continuity. Accordingly, the present evidence supports a plausible filler-assisted mechanism, but direct bonding or secondary reaction at the hardened bubble walls was not proven because FESEM-EDX and AFM were performed on raw DOBE rather than on hydrated LFC.

Scope and Limitations

This work was designed as a preliminary conference investigation and used one DOBE-containing trial formulation. Because a DOBE-free control mixture and matched density measurements were not included, the strength values cannot quantify the isolated contribution of DOBE or separate filler effects from possible differences in air content. In addition, the steam-cured specimens were tested after 48 h of heating and 24 h of cooling, whereas the conventional groups were tested after 28 days. The comparison is therefore limited to accelerated strength development rather than equal-age performance.

The microstructural interpretation is also based on raw DOBE. FESEM-EDX and AFM did not examine hydrated paste or the actual bubble walls, and the XRD amorphous estimate was software-derived. Future work should include a DOBE-free control, fresh and dry density measurements, replicate variability, equal-age curing comparisons, and FESEM-EDX of hardened bubble-wall regions. These additions would allow the proposed filler-assisted mechanism to be evaluated directly.

Conclusion

This conference study assessed DOBE as a fine filler in a lightweight foam concrete trial formulation. XRF, XRD and EDX showed a chemically heterogeneous material dominated by siliceous-aluminous particles with local calcium-rich regions. FESEM and AFM identified agglomerated, irregular particles and non-uniform nanoscale topography, with Ra = 0.971 nm and Rq = 1.24 nm. Steam curing at 70 °C for 48 h followed by 24 h cooling produced a mean compressive strength of 5.75 MPa, compared with 5.42 MPa for 28-day water curing and 5.14 MPa for 28-day open-air curing. The result indicates accelerated strength development under steam curing. Overall, DOBE shows preliminary suitability as a fine filler in LFC; however, future work should examine hydrated bubble-wall regions directly before claiming pozzolanic reaction or interfacial strengthening.

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