JST&M 9(2025), 30 – 43
DOI: 10.57131/jstm.2025.9.4

Original Scientific Paper

FROM WASTE TO RESOURCE: HYDROXYAPATITE-RICH BONE ASH DERIVED FROM CATTLE BONES FOR SUSTAINABLE CERAMICS

Temitope Ologunwa[1], Rasheed Kolade[2], Ayodeji Oke[3] and Oluyemi Ologunwa[4]

[1]Department of Industrial Design, Federal University of Technology, Akure, Ondo State, Nigeria
[2]Department of Industrial Design, University of Abuja, FCT, Nigeria
[3]Department of Quantity Surveying, Federal University of Technology, Akure, Ondo State, Nigeria
[4]Department of Project Management, Federal University of Technology, Akure, Ondo State, Nigeria

ABSTRACT

The increasing need for sustainable resource utilization has driven the exploration of waste-derived materials for industrial applications. This study investigates the valorization of cattle bone waste into high-purity hydroxyapatite-rich bone ash and its application in ceramics. The beneficiation process involved boiling, drying, controlled calcination at 850 °C, crushing, and milling, effectively removing organic contaminants while preserving the mineral structure. X-ray diffraction (XRD) analysis confirmed that the ash consists predominantly of hydroxyapatite (88 wt%) with high crystallinity, demonstrating its suitability as a ceramic raw material. The bone ash was incorporated into multiple ceramic ware formulations, which were fabricated into test samples and evaluated for physico-mechanical properties, including water absorption, apparent porosity, linear shrinkage, bulk density, and compressive strength. The formulation containing 55% bone ash, 30% feldspar, 12% kaolin, and 3% ball clay exhibited optimal performance, achieving low shrinkage (3.61%), minimal water absorption (1.69%), low porosity (2.72%), and high compressive strength (2.00 N/mm²), enabling the production of durable and high-quality ceramic wares.
These findings highlight the potential of bone ash to substitute conventional raw materials while promoting waste valorization and environmental sustainability.

Keywords: cattle bone, bone ash hydroxyapatite; xrd analysis; sustainable ceramics; waste management

Corresponding Author:
Temitope Peter Ologunwa,
Department of Industrial Design, Federal University of Technology, Akure, Ondo State, Nigeria
Tel.: +2348060912005
E-mail address: tpologunwa@futa.edu.ng

1. INTRODUCTION

The enhancement of ceramic materials involves mechanical and thermal processes—such as crushing, refining, and sizing—to optimize the quality of raw materials for industrial applications. These steps are critical for removing impurities (e.g., iron oxides) from clay, kaolin, and similar earthen materials, ensuring precise particle size distribution and purity to minimize defects in final products [1]. Recent advances highlight the potential of alternative raw materials, such as bone ash, to improve sustainability in ceramic production.

Bone ash, a calcined by-product of cattle bones, is rich in calcium phosphate phases, particularly hydroxyapatite $Ca_{10}(PO_4)_6(OH_2)$,

which constitutes 65–70% of its inorganic content [2]. Elemental analyses confirm that hydroxyapatite-derived bone ash contains 56.3% phosphate and 36.8% calcium, though compositional variations depend on bone source and processing parameters [3]. The controlled calcination of bones (800–1000°C) eliminates organic residues while preserving the mineral structure, making it suitable for high-value applications like bone china, biomedical ceramics, and sustainable construction materials. The beneficiation of cattle bones involves a series of physical, thermal, and chemical treatments designed to enhance purity, functionality, and economic value [2]. Cattle bones consist of organic components (e.g., collagen and fats) and inorganic minerals (primarily hydroxyapatite), which are processed under controlled conditions to remove impurities and modify their structural and chemical properties [3]. The resulting bone ash, rich in calcium phosphate compounds, has applications in fertilizers, ceramics, water treatment, and biomedical engineering [4]. The beneficiation process begins with preliminary treatments to remove residual meat, fatty oils, and other contaminants—a critical step to ensure purity and optimize downstream processing [5]. Subsequently, the bones undergo thermal treatment, where calcination at 800–1000°C in a controlled environment facilitates fragmentation and impurity removal while largely preserving the mineral structure [6]. This process yields a millable, high-purity bone ash suitable for industrial applications. The shift toward circular economy models has intensified interest in repurposing agricultural by-products like cattle bones, which are otherwise discarded as waste. With global ceramic industries seeking eco-friendly alternatives to finite natural resources, bone ash emerges as a viable substitute, reducing both environmental burdens and production costs [7].

Growing recognition of the untapped potential in animal by-products has highlighted their value as sustainable resources when properly utilized [8,9] Simply discarding these materials is no longer economically or environmentally viable, especially given their substantial volumes and potential for creating high-value products. Recent studies demonstrate how animal by-products from food industries can be successfully diverted to manufacturing sectors and transformed into value-added materials [10]. For instance, bone ash constitutes approximately 50% of bone china formulations and serves as a critical ceramic material that reacts with other components during firing to produce stable mineral phases like anorthite [11,12]

Poor management of bone waste not only escalates disposal costs but also creates significant environmental burdens [3]. Concurrently, excessive exploitation of natural resources has accelerated both waste generation and resource depletion [7]. These challenges necessitate a rapid shift toward circular economic models that prioritize efficient resource use. Consequently, the valorization of waste bones has emerged as a key sustainable waste management approach [13], with resource recovery from waste playing a pivotal role in closing industrial loops and advancing sustainability objectives.

While the composition of bone ash is generally known, the specific phase assembly resulting from a particular processing route determines its industrial suitability. Previous studies have often focused on extracting pure hydroxyapatite for biomedical applications; however, the valorization of bone waste for mass-scale industrial ceramics requires a different approach, prioritizing cost-effective, low-energy processing and a phase composition that benefits ceramic sintering.

This study thus addresses this gap by detailing a beneficiation protocol for cattle bone ash and characterizing its phase composition via XRD, underscoring its potential for ceramic applications such as bone china wares and porcelain tiles.

2. CATTLE BONES AS A SUSTAINABLE RESOURCE

The use of waste bone in different applications cannot be overemphasized.

Cattle bones, which are considered waste at one point, have become a sustainable resource and are considered a cost-effective, eco-friendly material. [14], explores the potential of using calcined cow bone powder (CBP) as a sustainable biofiller in hot-mix asphalt (HMA). Different CBP concentrations (5%, 10%, 15%) were tested in AP-5 bitumen, and various lab techniques (FT-IR, TLC-FID, viscosity tests, etc.) were used to analyze the filler-asphalt mix. The study discovered that CBP increased aromatics/asphaltenes but reduced resins without affecting saturates, and CBP improved stiffness, consistency, and high-temperature performance of asphalt. It concluded that waste cow bones enhance asphalt performance, reduce livestock industry pollution, and extend pavement lifespan.

Bone char is a promising solution for soil fertility enhancement, crop productivity, and heavy metal remediation, supporting sustainable farming while recycling slaughterhouse waste [15]. Its usage in soil and plant enhances phosphorus dynamics, redistributing soil phosphorus fractions for better fertility, and boosts effectiveness when combined with organic acids, sulfur/nitrogen fertilizers, or phosphate-solubilizing microbes. Moreso, heavy metals uptake by plants is reduced and lowers bioavailability in contaminated soils.

The quest for sustainable waste management practices catalysed the development of innovative approaches to transform waste into resourceful products. Cow bone waste (CBW) and calcium oxide (CaO) were tested for stabilizing Municipal Solid Waste Incineration (MSWI) fly ash from different incinerator types (grate-type and fluidized bed) in China. [12] optimized CBW and CaO blends effectively to reduce heavy metal leaching and enhance structural strength, offering a practical, eco-friendly solution for repurposing MSWI fly ash in construction.

Similarly, Cow bone ash has been used in ceramic applications as an alternative to traditional oxides, which are expensive and often imported, driving up ceramic production costs, aligning with industry needs for affordability, performance, and environmental responsibility. The Cow bone ash demonstrated comparable opacity, durability, and chemical resistance to conventional opacifiers. It provides clarity, stability, and resistance to chemical degradation, making it a viable substitute [16].

Hydroxyapatite HAp can be extracted from natural sources (e.g., cow, fish, and pig bones) via methods like thermal calcination, alkaline hydrolysis, or sub-critical water processing, which are cost-effective and environmentally friendly compared to complex synthetic methods [17,18,19]. However, the optimal extraction temperature remains unclear.

2.1 Composition of Bones - Structural and Functional Complexity

Bone is a complex, functionally graded structure that provides mechanical support, aids in hemostasis, and supports endocrine functions. It combines high stiffness and toughness through its hierarchical organization, spanning from millimeter to nanometer scales. Bone consists of an organic matrix (primarily Type I collagen) and a mineral component (calcium-deficient hydroxyapatite), intricately arranged to balance flexibility and strength [20]. Bone acts as a composite, protecting brittle hydroxyapatite and distributing stress. It is categorized into cortical (compact) and cancellous (porous) types, comprising of 65% mineral (hydroxyapatite) that contributes to stiffness; 20-30% Collagen (mostly type I) that provides flexibility; and 10% water that aids in load distribution and mineralization. [19], corroborate the fact that bone is composed of 30% organic (mainly collagen) and 70% inorganic (mineral) compounds. The mineral phase, primarily nonstoichiometric hydroxyapatite (HAp) (Ca/P ratio ≠ 1.67), provides stiffness and mechanical strength. HAp is chemically similar to natural bone minerals, making it an excellent biocompatible, osteoconductive, and bioactive material for bone substitutes, tissue engineering, drug delivery, and heavy metal removal.

2.2 Functional Advantages of Bone Ash

The use of bone ash or Hydroxyapatite (HAp) is enormous. Sustainable Valorization of Waste Animal Bones from Environmental Burden to High-Value Materials is overwhelming. Over 130 million metric tons of waste animal bones (WABs) are generated annually from slaughterhouses and food processing, which often end up in landfills, causing environmental pollution and health risks can now be recycled into high-value materials, aligning with circular economy goals. Study [3] highlights that the transformation of WABs into functional materials represents a key link between waste management and resource recovery, enabling eco-friendly industrial applications and mitigating environmental damage. Among its uses, to mention a few, are:

- Bio-Medical: Using Hydroxyapatite (HAp) Scaffolds has advanced Bone Tissue Engineering (BTE) [21]. Bone regeneration remains a major challenge in regenerative medicine due to fractures, diseases, and traumatic injuries. However, HAp ($Ca_{10}(PO_4)_6(OH)_2$) closely resembles natural bone mineral, enhances integration and regeneration. It supports bone cell growth and mineralization. This made it relevant in scaffolds, coatings, and bone cements to improve implant compatibility and bone repair.
- Similarly, Hydroxyapatite (HAp) nanoparticles are a promising biomaterial for bone implants and drug delivery due to their low cost, chemical similarity to human bone, and excellent bioactivity, biocompatibility, and osteoconductivity [22]. While HAp is widely used, its low mechanical strength and lack of antibacterial activity limit its effectiveness, but could be overcome by combining HAp with metals, polymers, or ceramics to improve mechanical strength and antibacterial properties without altering biocompatibility. HAp nanocomposites can also be used locally for drug delivery while maintaining biodegradability and avoiding systemic toxicity.
- Global Waste Management: Rapid urbanization and changing consumption patterns have intensified waste generation, with traditional methods like landfilling and incineration facing limitations due to space constraints, environmental harm, and health risks. Municipal Solid Waste Incineration (MSWI), while effective in volume reduction (85–90%) and energy recovery, produces hazardous fly ash (MSWI-FA) containing heavy metals, dioxins, and organic pollutants. China alone generates ~7 million tons/year of MSWI-FA, necessitating advanced treatment solutions [23]. The study explores a sustainable Solidification and Stabilization method by combining MSWI-FA with cow bone waste (rich in hydroxyapatite) and CaO, enhancing heavy metal immobilization. This invariably aligns with sustainable development goals by mitigating environmental risks of MSWI-FA, promoting waste-to-resource strategies in construction, and offering a cost-effective, scalable alternative to conventional methods.
- Ceramics: developed a standard ceramic coating formulation based on clay, kaolin, quartz, talc, and feldspar, with hydroxyapatite (HAp) added to enhance the mechanical properties of the final product. HAp serves as a filler, closing gaps in the microstructure, while its high calcium oxide content may also reduce the sintering temperature. X-ray diffraction analysis confirmed the presence of key mineralogical phases—mullite, quartz, and anorthite—in the sintered samples. Evaluation of physical-mechanical properties (water absorption, linear shrinkage, apparent porosity, and flexural strength) revealed that HAp incorporation (5 wt%, 10 wt%, and 20 wt%) significantly improved performance. The incorporation of hydroxyapatite (HAp) into ceramic formulations offers a sustainable solution for producing high-performance tiles with low water absorption, reduced porosity, and

enhanced flexural strength—key properties for porcelain tile applications. HAp significantly improves the mechanical strength and overall performance of sintered ceramic materials [24].

Similarly, hydroxyapatite (HAp), commonly used in bone china (up to 50% bone ash), can also act as a sintering aid in porcelain when added in small amounts (1–5 wt%). The study conducted by [25] tested CBB in a triaxial porcelain system, finding that 2% CBB reduced the sintering temperature by 50°C while maximizing tensile strength. Lower hydroxyapatite (HAp) additions (1–2%) promoted mullite formation without altering initial sintering temperatures, whereas 5% HAp lowered sintering onset due to enhanced liquid-phase formation. The study demonstrates HAp's dual role in porcelain as a strength enhancer and sintering promoter.

3. BENEFICIATION AND PROCESSING OF BONE ASH (HYDROXYAPATITE)

3.1 Pre-treatment Methods

The initial processing of cattle bones is a critical step aimed at thoroughly removing all organic residues, including fats, collagen, and residual soft tissues, to maximize the recovery of high-purity hydroxyapatite (HAp). This purification process is essential because organic contaminants can compromise the chemical and structural integrity of the final HAp product, affecting its suitability for ceramic applications. This is done by:

- Boiling & Degreasing: The initial boiling and degreasing stage represents a critical first step in the purification of raw cattle bones, designed to effectively remove the majority of organic contaminants. This comprehensive thermo-chemical treatment begins with carefully selected bovine bones that have been freshly sourced and preliminarily rinsed to remove gross contaminants. The bones are then systematically arranged in corrosion-resistant stainless-steel vessels and completely submerged in deionized water maintained at carefully controlled elevated temperatures (90-100°C) using precision temperature regulation systems. This process is corroborated in [26]

  The hydrothermal treatment is conducted for an extended period of 4-6 hours to ensure thorough extraction of organic components. After completion, the bones are removed using perforated baskets to allow proper drainage, then immediately transferred to the next processing stage while still warm to prevent reabsorption of extracted materials. This thorough degreasing treatment typically removes 85-95% of the initial organic content, preparing the bones for subsequent drying and mechanical cleaning stages while preserving the integrity of the mineral matrix.
- Drying: Following the boiling and degreasing stage, the purified cattle bones undergo a controlled drying process to eliminate residual moisture and further stabilize the material for subsequent calcination. The bones are carefully arranged in a single layer on stainless steel drying racks or mesh trays to ensure optimal air circulation and uniform heat exposure. The bones are placed in open-air drying yards with direct sunlight exposure. Ambient temperatures are maintained at approximately 35–45°C, while solar radiation elevates surface temperatures to ~105°C during peak daylight hours. This process spans three full days (72 hours), with bones periodically turned to ensure even dehydration.
- Manual Cleaning: The processed cattle bones undergo meticulous manual cleaning to eliminate any remaining organic contaminants that could compromise the purity and performance of the final hydroxyapatite (HAp) product. This labor-intensive but critical quality is ensured to remove pertinent soft tissue adherence, cartilaginous components, and other organic contaminants

3.2 Thermal Treatment (Calcination)

Controlled calcination serves as a critical thermal treatment step that transforms raw, defatted cattle bones into phase-pure hydroxyapatite (HAp) ash while ensuring complete elimination of residual organic components (e.g., collagen, lipids, and residual proteins). A calcination temperature of 850°C was selected to ensure the complete removal of organic matter while preserving hydroxyapatite (HAp) as the dominant phase, in alignment with the study's sustainable and cost-effective objectives. This high-temperature process not only purifies the inorganic bone matrix but also induces crystallographic changes that enhance HAp's thermal stability and chemical homogeneity. In this study, the calcination process was meticulously executed using a gas-fired kiln to ensure precise temperature control and uniform heat distribution. The prepared bones were carefully loaded into the kiln chamber in a single layer to prevent uneven calcination and then subjected to a ramp-up heating cycle (typically 5–10°C/min) until reaching the target temperature of 850°C as measured by a thermocouple located at the front of the kiln. Upon reaching 850°C, the kiln was held at this peak temperature (soaked) for 5 minutes to ensure thorough heat penetration and complete combustion of any residual carbonaceous material. This short dwell time was deliberately chosen to balance energy efficiency with the need for phase purity, as prolonged exposure could lead to excessive sintering or partial decomposition. After the holding period, the kiln was systematically shut down and allowed to cool gradually (~48 hours) under controlled ambient conditions.

The calcined bones were then carefully offloaded, inspected for uniformity (e.g., colour, texture), and subsequently milled into a fine ash powder for further characterization (XRD) and application testing. Figure 1 presents the initial stages of sample preparation, showing a) the raw cattle bones and b) the boiling process applied prior to further treatment. Figure 2 illustrates the key steps of the thermal treatment procedure, including a) sample loading, b) the calcining process, and c) the thermocouple used for temperature monitoring.



a) b)

Figure 1. a) Raw cattle bones, b) Boiling



a) b) c)

Figure 2. a) Loading, b) Calcining process, c) Thermocouple

3.3 Post-Treatment (Crushing, Milling, and Refining)

Final processing ensures uniform particle size for ceramic applications. This was done by crushing the bones into smaller forms using a mortar and pestle, and milled using a milling machine to obtain well-refined bone ash. These processes include:

- Crushing & Milling: The calcined bone samples were initially subjected to manual crushing using a mortar and pestle to achieve a preliminary reduction in particle size, facilitating subsequent mechanical processing. The fragmented hydroxyapatite particles were then further comminuted using a grinding machine to obtain granules with a diameter below 5 mm, ensuring uniformity and optimal handling for the next stage of refinement. Finally, the processed granules were subjected to ball milling to achieve an ultra-fine particle size distribution, with the majority of particles falling within the 100-micrometer (µm) range using an Endecotts sieve shaker and a set of Tyler sieves, thereby enhancing their suitability for advanced material applications.
- Ball Milling: To optimize the physicochemical properties of hydroxyapatite (HAp), the influence of particle size reduction was considered using a high-energy ball milling process. This mechanical treatment facilitated the efficient comminution of coarse hydroxyapatite granules, resulting in a significant reduction in particle size. The ball milling process was meticulously controlled to achieve a uniform particle distribution within the range of 50–100 µm, transforming the initially coarse granules into a finely powdered form. This refinement in particle size is critical for enhancing the material's functional characteristics, including its sinterability and surface reactivity.

Figure 3 provides an overview of the particle-size reduction procedure, showing a) crushing with a mortar and pestle, b) milling of bone using a grinding machine, c) sieving with an Endecott shaker, and d) final refinement in a table ball mill.

3.4 XRD analysis

The powdered samples were homogenized and sieved to achieve a uniform particle size of ≤74 µm (200 mesh) to ensure optimal sample packing and diffraction reproducibility. The sieved powder was then uniaxially pressed into dense pellets to minimize preferred orientation effects. For XRD analysis, each pellet was carefully mounted on a high-purity aluminum alloy sample holder (35 mm × 50 mm) placed on a flat glass substrate to ensure consistent surface alignment. To prevent contamination, the samples were handled exclusively with powder-free nitrile gloves during preparation. A thin, non-reflective paper cover was applied to avoid sample

displacement while maintaining X-ray penetration.

X-ray diffraction analysis was conducted using a Rigaku D/Max-IIIC diffractometer (Rigaku International Corp., Tokyo, Japan) equipped with a Cu-Kα radiation source (λ = 1.5406 Å) operating at 40 kV and 20 mA. The instrument was calibrated using a silicon standard before measurements. Diffraction patterns were acquired in Bragg-Brentano (θ/2θ) geometry over a 2θ range of 0°–70° at a scanning rate of 2°/min with a step size of 0.02°. All measurements were performed under ambient conditions (25 ± 1°C, 50 ± 5% relative humidity).

The acquired diffraction profiles were processed using Rigaku PDXL 2.0 software, and the interplanar spacings (d-values) and relative peak intensities were extracted. Phase identification was performed by matching the experimental diffraction patterns with reference data from the International Centre for Diffraction Data (ICDD) PDF-4+ database, which contains standard crystallographic parameters for over 300,000 inorganic and mineral phases. A minimum match index of 85% was applied to confirm phase purity and crystallinity.



a) b) c) d)

Figure 3. a) Crushing with a mortar and pestle, b) Milling of bone with a grinding machine, c) Endecott Sieve shaker, d) Table Ball Mill

4. RESULTS AND DISCUSSION

4.1 Phase Identification and Quantitative Composition:

X-ray diffraction (XRD) analysis of the prepared sample, shown in Figure 4, indicates that it is predominantly composed of hydroxyapatite (HAp), providing key insights into the material's phase purity, crystallographic properties, and potential synthesis artifacts. Table 1 summarizes the qualitative phase analysis, presenting the identified crystalline phases together with their formulas, figures of merit, registration details, space groups, and corresponding database card numbers. Quantitative analysis, based on the Rietveld method, suggests the sample consists of 88±3 wt% hydroxyapatite ($Ca_{4\cdot866}(PO_4)_3(OH)_{0\cdot708}$), crystallizing in the hexagonal space group $P6_3/m$ (ICDD #01-074-9775), consistent with biologically relevant apatite structures, as reported in synchrotron-based XRD studies of rat bone [27]. Figure 4 presents the phase composition of the synthesized bone ash as determined by XRD Rietveld refinement. The high HAp content suggests good biocompatibility, indicating that the material could be suitable for biomedical uses such as bone grafts, implant coatings, and other ceramic applications. The reported presence of synthetic hatrurite (Ca_3SiO_5) and quartz (SiO_2) is questionable. Hatrurite, a high-temperature phase, is unlikely to form under the relatively low synthesis temperature of 850 °C. Similarly, the apparent quartz fraction is higher than expected and may result from minor impurities in the raw materials or incomplete homogenization during synthesis. These observations may also reflect limitations in Rietveld analysis, particularly in detecting trace or low-intensity phases. Therefore, the inclusion of hatrurite and the reported quartz fraction cannot be reliably confirmed. Complementary characterization techniques, such as SEM/EDS and FTIR, are recommended to verify the actual phase composition.

Overall, the sample demonstrates a highly crystalline hydroxyapatite matrix, with minor secondary phases that are either negligible or uncertain, emphasizing the effectiveness of the synthesis protocol in producing phase-pure HAp.

4.2 Structural and Microstructural Analysis

Crystallite size analysis using the Scherrer equation indicates that hydroxyapatite particles range from 431 to 696 Å, with the largest crystallites corresponding to the peak at 49.66° (1.834 Å). Peaks associated with hatrurite and quartz are either weak or likely artifacts; however, if considered, their apparent crystallite sizes are 160–484 Å for hatrurite and ~53 Å for quartz. Peak broadening observed for these minor reflections may result from nanostructuring, internal strain, or instrumental effects, rather than the presence of significant quantities of these phases.

Notably, some hydroxyapatite peaks (e.g., at 49.66°) display asymmetry, suggesting anisotropic strain or preferred orientation, likely introduced during uniaxial pressing in sample preparation. The highest-intensity diffraction peak for HAp occurs at 31.97° (2.797 Å) with a normalized intensity of 100%, confirming its dominance. The low uncertainties in phase quantification (±3 wt% for HAp) and high figures of merit (FOM > 0.7) support the reliability of the Rietveld refinement for the primary phase, while caution should be exercised in interpreting minor phases.

4.3 Findings and Discussion

The results confirm that the synthesized material consists predominantly of highly crystalline hydroxyapatite, while the minor phases registered during Rietveld refinement—identified as hatrurite and quartz—are unlikely to represent actual crystalline components of the sample. Their apparent presence can be attributed to peak overlap, trace impurities in the raw materials, incomplete homogenization, or limitations of the refinement algorithm in handling weak or broadened reflections. This interpretation is consistent with the relatively low synthesis temperature of 850 °C, at which the formation of hatrurite is thermodynamically improbable, and with the expectation that quartz, if present at all, should only appear as negligible residual impurities. These observations emphasize

the need for optimized synthesis and purification protocols to ensure maximum phase purity. Improved raw material treatment—such as acid washing, sieving, or controlled calcination—could effectively minimize the contribution of impurity-related peaks that may be misinterpreted during XRD analysis. Furthermore, the integration of complementary analytical techniques (SEM/EDS, FTIR, or TEM) would provide a more reliable basis for confirming whether any minor non-HAp phases are genuinely present. From an application standpoint, the dominance of a stable and well-crystallized hydroxyapatite matrix supports the suitability of the material for ceramic uses, particularly where phase purity and compatibility are essential. The findings align with previous studies [28–30], demonstrating that controlled processing conditions can yield high-quality hydroxyapatite with minimal extraneous components.

Table 1. Qualitative Analysis Report

Phase name	Formula	Figure of merit	Phase reg. detail	Space Group	DB Card Number
Hydroxylapatite	$Ca_{4.866}(PO_4)_3(OH)_{0.708}$	0.733	S/M (PDF-4 Minerals 2025)	176:63/m	01-074-9775
Hatrurite, syn	Ca_3SiO_5	1.908	Import (PDF-4 Minerals 2025)	160:R3m:H	00-016-0406
Quartz	SiO_2	3.146	Import (PDF-4 Minerals 2025)	154:P3221	00-001-0649

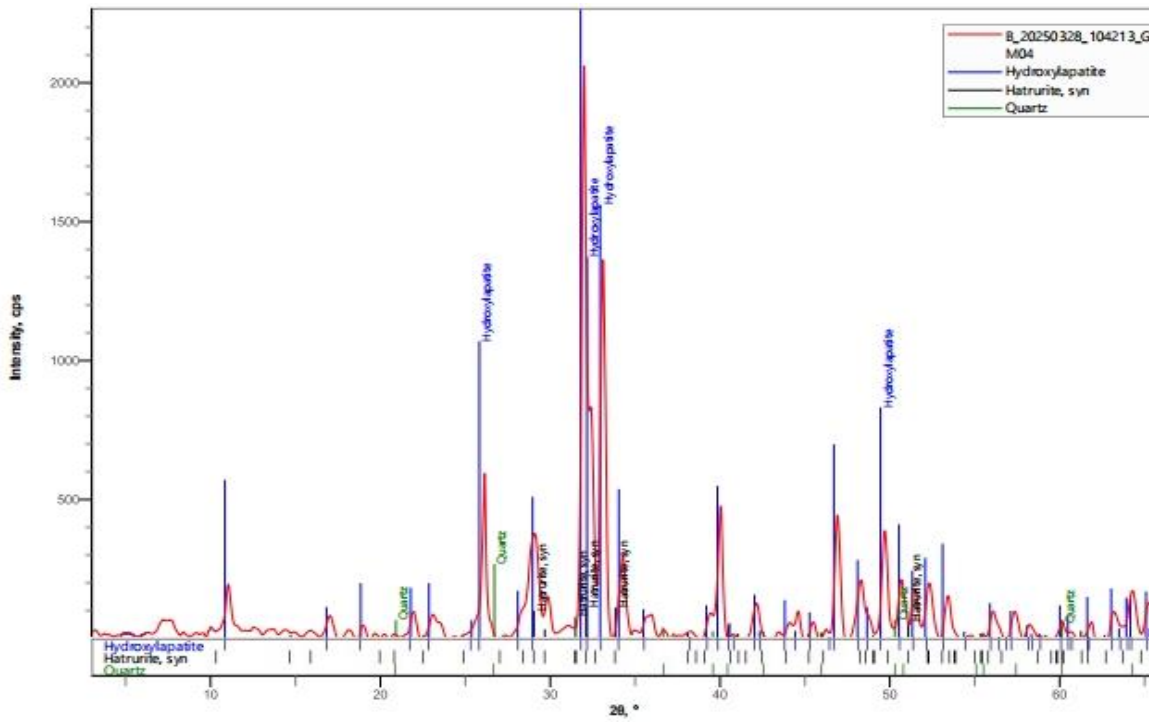


Figure 4. XRD Diffractogram of Processed Bone Ash

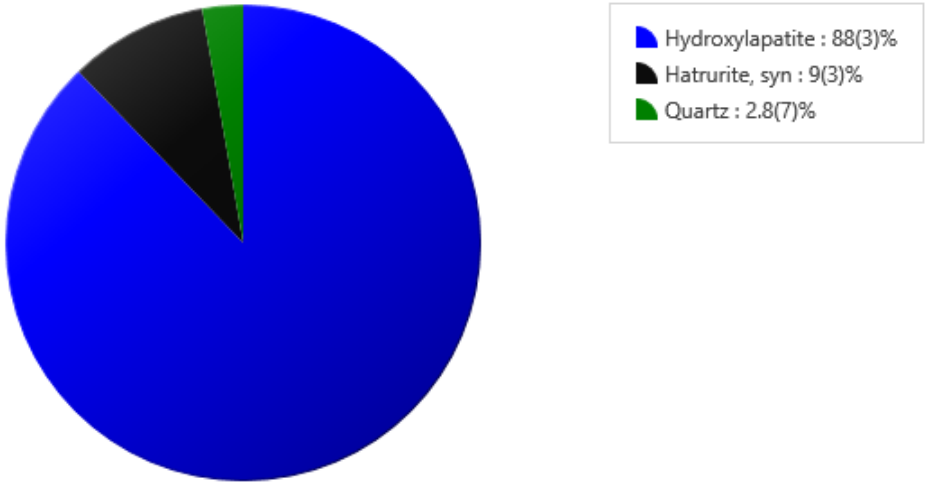


Figure 4. Phase composition of the synthesized bone ash based on XRD Rietveld refinement

4.4 Formulation of Ceramic Ware Body

Five distinct body compositions were formulated for this study, as summarized in Table 2. Sample A corresponds to the authors' original formulation, whereas the other compositions (B-F) were reproduced in our laboratory according to the exact compositional proportions reported in previous studies [27–31]. The primary objective of this study was to identify the composition exhibiting the most favorable physico-mechanical properties for optimal performance and durability. To ensure reliability and reproducibility, six replicates were prepared for each formulation, resulting in a total of thirty samples, Figure 5, subjected to detailed analysis and comparative evaluation. Physico-mechanical test results for the synthesized samples are provided in Table 3. The results indicated that Sample C, composed of 55% bone ash, 30% feldspar, 12% kaolin, and 3% ball clay, exhibited the most favorable physio-mechanical and optical properties. This formulation achieved low shrinkage (3.61%), minimal water absorption (1.69%), low porosity (2.72%), appropriate bulk density (1.61 g/cm³), and high translucency. Thus, a prototype of ceramic home accessories was produced from the best-performing sample, Figure 6.



Figure 5. Ceramic Samples



Figure 6. Ceramic Wares Produced from Hydroxyapatite-Rich Bone Ash

5. CONCLUSION

This study successfully demonstrates the valorization of cattle bone waste into high-purity hydroxyapatite-rich bone ash through a systematic beneficiation process involving

boiling, calcination, and milling. XRD analysis conducted confirmed the dominance of hydroxyapatite (88 wt%), validating its suitability for sustainable ceramic applications such as bone china materials. The incorporation of produced bone ash into ceramic ware formulations produced samples with promising physico-mechanical properties. Among the tested compositions, the formulation containing 55% bone ash, 30% feldspar, 12% kaolin, and 3% ball clay exhibited optimal performance, with low shrinkage (3.61%), minimal water absorption (1.69%), low porosity (2.72%), and high compressive strength (2.00 N/mm²). The findings highlight the dual environmental and industrial advantages of repurposing agricultural by-products, aligning with circular economy principles by reducing waste and substituting finite natural resources. While the material's phase purity and crystallinity meet ceramic standards, further optimization of calcination parameters could minimize secondary phases for specialized applications. This work not only advances eco-friendly material science but also provides a scalable model for transforming waste into high-value functional ceramics, contributing to sustainable industrial practices and resource conservation. Future research should explore large-scale integration of bone ash in ceramic formulations to assess performance under industrial firing conditions.

Table 2. Samples Compositions Mixtures A-F (%)

Authors	Bone ash (%)	Feldspar (%)	Kaolin (%)	Quartz (%)	Ball Clay (%)	Total (%)
Sample A	50	25	20	-	5	100
Sample B	50	30	17	-	3	100
Sample C	55	30	12	-	3	100
Sample D	33	30	27	7	3	100
Sample E	50	20	27	-	3	100
Sample F	50	25	22	-	3	100

Table 3. Physico-Mechanical Test Results for Produced Samples

Sample Code	Linear Shrinkage (%)	Apparent Porosity (%)	Water Absorption (%)	Bulk Density (g/cm^3)	Comprehensive Strength (N/mm^2)
A	3.81	8.37	5.08	1.65	1.97
B	5.53	2.03	1.20	1.69	1.97
C	3.61	2.72	1.69	1.61	2.00
D	5.34	10.04	6.62	1.52	1.96
E	7.76	6.22	3.88	1.60	1.97
F	9.41	2.73	1.73	1.58	2.00

Acknowledgment: The authors acknowledged the funding received from the Tertiary Education Trust Fund (TETFund) Nigeria through the 2022-2024 Institution-Based Research Fund (IBRF) grant for this study (TETF/DR&D/CE/UNI/AKURE/IBR/2024). The Federal University of Technology, Akure, Nigeria, is also appreciated for providing a conducive environment & facilities support.

Conflicts of Interest.
The authors declare no conflict of interest

6. REFERENCES

[1] S. D. Salahaddin, J. H. Haido, and G. Wardeh, The behavior of UHPC containing recycled glass waste in place of cementitious materials: A comprehensive review, *Case Stud. Constr. Mater.*, 17(2022), p. e01494, doi: 10.1016/j.cscm.2022.e01494.

[2] N. A. S. Mohd Pu'ad, R. H. Abdul Haq, H. Mohd Noh, H. Z. Abdullah, M. I. Idris, and T. C. Lee, Synthesis method of hydroxyapatite: A review, *Mater. Today Proc.*, 29 (2020), pp. 233–239, doi: 10.1016/j.matpr.2020.05.536.

[3] A. Hart, E. Komonibo, E. Peretomode, H. Onyeaka, O. F. Nwabor, and K. C. Obileke, Value-added materials recovered from waste

bone biomass: technologies and applications, *RSC Adv.*, 12 (2022), pp. 22302–22330, doi: 10.1039/D2RA03557J.

[4] D. K. Datta, T. Biswas, E. Castonguay, and P. Ni, Sustainable Stabilizer Derived from Calcium- and Phosphorus-Rich Biowaste for Remediation of Heavy Metal-Contaminated Soil: A Critical Review, *Sustainability*, 16 (2024), 20, doi: 10.3390/su16208841.

[5] N. Salahudeen and A. A. Mukhtar, Effect of Beneficiation on the Characterization of Getso Kaolin, *Min. Rev.*, 27 (2022) 4, pp. 72–77, doi: 10.2478/minrv-2021-0036.

[6] P. C. Okpe, O. Folorunso, V. S. Aigbodion, and C. Obayi, Hydroxyapatite synthesis and characterization from waste animal bones and natural sources for biomedical applications, *J. Biomed. Mater. Res. B Appl. Biomater.*, 112 (2024) 7, p. e35440, doi: 10.1002/jbm.b.35440.

[7] A. J. Lag-Brotons, A. P. M. Velenturf, R. Crane, I. M. Head, P. Purnell, and K. T. Semple, Editorial: Resource Recovery From Waste, *Front. Environ. Sci.*, 8 (2020), doi: 10.3389/fenvs.2020.00035.

[8] K. Jayathilakan, K. Sultana, K. Radhakrishna, and A. S. Bawa, Utilization of byproducts and waste materials from meat, poultry and fish processing industries: a review, *J. Food Sci. Technol.*, 49 (2012) 3, pp. 278–293, doi: 10.1007/s13197-011-0290-7.

[9] R. Thakur *et al.*, Characteristics and application of animal byproduct-based films and coatings in the packaging of food products, *Trends Food Sci. Technol.*, 140 (2023), p. 104143, doi: 10.1016/j.tifs.2023.104143.

[10] E. R. Palomino-Guzmán, A. González-López, J. Olmedo-Montoya, L. A. Sanchez-Echeverri, and N. J. Tovar-Perilla, A Sustainable Approach Using Beef and Pig Bone Waste as a Cement Replacement to Produce Concrete, *Sustainability*, 16 (2024) 2, doi: 10.3390/su16020701.

[11] N. L. Bih *et al.*, The Effect of Bone Ash on the Physio-Chemical and Mechanical Properties of Clay Ceramic Bricks, *Buildings*, 12, (2022) . 3, doi: 10.3390/buildings12030336.

[12] Z. Khalid *et al.*, Utilization of cow bone waste and calcium oxide for the solidification and stabilization of MSWI fly ash: Towards sustainable practices, *Process Saf. Environ. Prot.*, 190 (2024), pp. 829–841, doi: 10.1016/j.psep.2024.08.095.

[13] A. Kara and R. Stevens, Characterisation of biscuit fired bone china body microstructure. Part I: XRD and SEM of crystalline phases, *J. Eur. Ceram. Soc.*, 2 (2002) 5, pp. 731–736, doi: 10.1016/S0955-2219(01)00371-5.

[14] N. Nciri, (PDF) Uncovering the Hidden Value of Waste Cow Bones Towards their Use as a Sustainable Biofiller for Hot-Mix Asphalt Paving Applications, *ResearchGate*, Apr. 2025, doi: 10.4028/p-xxox30.

[15] A. E.-E. A. Z. Amin, Using Bone Char as a Renewable Resource of Phosphate Fertilizers in Sustainable Agriculture and its Effects on Phosphorus Transformations and Remediation of Contaminated Soils as well as the Growth of Plants, *J. Soil Sci. Plant Nutr.*, 24 (2024) 4, pp. 6980–6998, doi: 10.1007/s42729-024-02018-y.

[16] L. O. Ajala, I. O. Odewale, and N. O. Osonwa, Revolutionizing Ceramic Artistry: Harnessing the Potential of Cow Bone Ash as a Sustainable Opacifier in Enamel Production, *J. Mater. Sci. Res. Rev.*, 7 (2024) 1, pp. 74–79

[17] F. Arjamend, Extraction of pure natural hydroxyapatite from the bovine bones bio waste by three different methods, *J. Mater. Process. Technol.*, Sept. 2013, doi: 10.1016/J.JMATPROTEC.2008.07.040.

[18] O. S. Mahdi, Preparation of Hydroxyapatite from Natural Resources Literature Review, Nov. 2022, Accessed: Aug. 06, 2025. [Online]. Available: https://www.academia.edu/91711764/Preparation_of_Hydroxyapatite_from_Natural_Resources_Literature_Review

[19] M. Wildan, Preparation and Characterization of Natural Hydroxyapatite: A Comparative Study of Bovine Bone Hydroxyapatite and Hydroxyapatite from Calcite, *Mater. Sci. Forum*, Mar. 2015, doi: 10.4028/0-87849-462-6.1441.

[20] P. Dey, Bone Mineralisation, in *Contemporary Topics about Phosphorus in Biology and Materials*, IntechOpen, 2020. doi: 10.5772/intechopen.92065.

[21] W. Liu, N. Cheong, Z. He, and T. Zhang, Application of Hydroxyapatite Composites in Bone Tissue Engineering: A Review, *J. Funct. Biomater.*, 16 (2025) 4, doi: 10.3390/jfb16040127.

[22] N. A. Abdul Halim, M. Z. Hussein, and M. K. Kandar, Nanomaterials-Upconverted Hydroxyapatite for Bone Tissue Engineering and a Platform for Drug Delivery, *Int. J. Nanomedicine*, 16 (2021), pp. 6477–6496, doi: 10.2147/IJN.S298936.

[23] Z. Khalid *et al.*, Utilization of cow bone waste and calcium oxide for the solidification and stabilization of MSWI fly ash: Towards sustainable practices, *Process Saf. Environ.*

Prot., 190 (2024), pp. 829–841, doi: 10.1016/j.psep.2024.08.095.

[24] F. P. Avelino, W. M. P. de Araujo, R. A. Lima Soares, R. Peña-Garcia, and A. O. Lobo, Porcelain Ceramic Tile Manufactured with the Addition of Hydroxyapatite in Ceramic Formulations, *Minerals*, 13 (2023) 9, doi: 10.3390/min13091120.

[25] D. Gouvêa, T. Tisse Kaneko, H. Kahn, E. de Souza Conceição, and J. L. Antoniassi, Using bone ash as an additive in porcelain sintering, *Ceram. Int.*, 41 (2015) 1, Part A, pp. 487–496, doi: 10.1016/j.ceramint.2014.08.096.

[26] P. O. Etinosa *et al.*, In-depth review of synthesis of hydroxyapatite biomaterials from natural resources and chemical regents for biomedical applications, *Arab. J. Chem.*, 17(2024) 12, p. 106010, doi: 10.1016/j.arabjc.2024.106010.

[27] T. S. Toludare, S. S. Owoeye, A. Kenneth-Emehige, and O. E. Isinkaye, Microstructure evolution and physico-mechanical properties of bone china porcelain compositions using two selected kaolinite clays from Nigeria, *Sci. Afr.*, 3 (2019), doi: 10.1016/j.sciaf.2019.e00066.

[28] Y. Zhang *et al.*, Fabrication and characterization of bone china using synthetic bone powder as raw materials, *Ceram. Int.*, 42 (2016), doi: 10.1016/j.ceramint.2016.06.131.

[29] Z. Zakaria and H. Haron, Characterisation of Local Bone Ash for Bone China Production, *J. Teknol. Sci. Eng.*, 66 (2014) 1, 2014, doi: 10.11113/jt.v66.2157.

[30] S. Bragança and C. Bergmann, A comparative study between bone China and hard porcelain, *Ind. Ceram.*, 28 (2008), pp. 189–194,

[31] F. Güngör, Investigation of Sintering Behaviour of Bone China Bodies Produced by Bone China Wastes, *Uluslar. Muhendislik Arastirma Ve Gelistirme Derg.*, pp. 481–488, June 2019, doi: 10.29137/umagd.480632.

[32] ASTM International, ASTM C373-88, Standard Test Method for Water Absorption, Bulk Density, Apparent Porosity, and Apparent Specific Gravity of Fired Whiteware Products. Accessed: Dec. 02, 2025, [Online], Available: https://store.astm.org/c0373-88r06.html

[33] ASTM International, ASTM C326-09, Standard Test Method for Drying and Firing Shrinkages of Ceramic Whiteware Clays'. Accessed: Dec. 02, 2025. [Online]. Available: https://store.astm.org/c0326-09r18.html