

Effect of $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ on the Properties of Locally Developed Al_2O_3 Based Artificial Bone

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Abstract

The development of artificial bone using alumina and Nsu clay was carried out in order to determine the effect of $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ on the properties of locally developed Al_2O_3 -based artificial bone. The raw materials were used to formulate seven samples (A - G) and shaped using casting method. The physical properties investigated after sintering of the samples at 1400°C indicated that samples A-G had linear shrinkage ranging from 3.17% - 3.98% with percentage porosity ranging from 4.78% - 4.51% respectively. The result of bulk density of the samples ranges from $2.6\text{g}/\text{cm}^3$ - $4.20\text{g}/\text{cm}^3$ with corresponding compressive strength ranging from 424.5MPa - 489.1MPa. It was discovered that the lower the percentage of alumina content in the composition, the higher the bulk density, linear shrinkage and compressive strength while the lower the porosity of the samples, vice versa. Moreover, sample D of the artificial bone gave the most favourable result in view of the stated properties above. Therefore sample D can be used for production of artificial bone. However, the investigated physical properties gave results that are acceptable for a standard artificial bone, this product is an archetype, therefore, it requires a clinical/medical compatibility test before it can be put to use.

Keywords: Alumina; Artificial bone; Biomaterial; Bioceramics; Nsu clay; Compressive strength.

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1. INTRODUCTION

Biomaterial is a few substance, supplementary than a drug or blend of substances, artificial or innate in origin, which can be used for any period of time, as a whole or as a fraction of a system which take care of, enhance or reinstate or substitute any tissue, organ or function of the body [1, 2]. These includes ceramics, polymers, metals and composite materials. Biomaterial is not of a modern origin. The beginning of non-biological materials into the human body was renowned far rear in prehistory. In early 900s, bone plates were brought forward to aid in the fixation of long bone fractures [3]. Many of these early products broke due to primitive mechanical design, as they were too slight and had stress fixed corners. Also, materials such as vanadium steel was selected as biomaterial due to its good mechanical properties, but corroded quickly in the body and led to adverse effects on the therapeutic processes. When stainless steels and cobalt chromium alloys were introduced in the 1930s as biomaterials,

greater victory was recorded in fracture fixation and the first joint substitution surgeries were achieved [4].

Biomaterial is a nonviable (able to play its role effectively after implantation) material planned to work together with biological systems. Their usage within a physiological medium is achievable with the competent and dependable characteristics of the biomaterials [5]. These distinguishing features are made available with an opposite blend of chemical, biological, mechanical and physical properties, to devise entrenched biomaterials [6]. However, the most vital prerequisite of the biomaterial is corresponding of its physical properties with the desired organ/tissue in the living system where it is to be implanted. Biomaterials and devices should unavoidably have appropriate mechanical and performance necessities corresponding to that of the replacing organ/tissue.

Categorization of Bioceramics: The system of tissue attachment is directly connected to the type of tissue reaction at the implant interface, which largely depends on the type of material used. Bioceramics are categorized into four types: Nearly Inert Bioceramic (This is group of nearly inert crystalline bioceramics products like high-purity, high-density alumina, they are frequently used in load bearing prostheses and dental implants) [7]. Porous Bioceramics (Studies indicated that nearly inert porous ceramics can offer a functional implant when load bearing is not a primary obligation) [8]. Bioactive Ceramics (“A bioactive material is one that elicits a specific biological response at the interface of the material which results in the formation of a bond between the tissues and the material”) [9-10]. Resorbable Bioceramics (These are called provisional space fillers for new tissue to build up. Natural tissue rebuilding takes place concurrently with resorption) [11, 12].

Raw Material for Bioceramics: The uses of bioceramics have been revolutionizing the biomedical area of specialization in exploitation as implants for humans. As many as possible implant materials made of ceramics products have been put to use for the past many decades. The foremost types are alumina (Al_2O_3)-based ceramics and zirconia (ZrO_2)-based ones. However, some of other simple oxides such as $\text{CaO} \cdot \text{Al}_2\text{O}_3$, $\text{CaO} \cdot \text{TiO}_2$ and $\text{CaO} \cdot \text{ZrO}_2$ have been investigated for use in biomedical applications [13-17].

Assessment of Biomaterial Performance: Prior to clinical application of any biomaterial, which is going to be used as an implant, in contact with living conditions of an organism, it should be strictly tested and proven to be harmless. The assessment of the biomaterial with respect to its usability in the biological system is extensively assessed through three ways, before it is put into usage: Assessment of Physical Properties (Physical strengths, thermal properties, photoreactivity, colour, calcification potency, surface structure, degradation resistance of materials needs to be modified for perfect adaptation to the biological environment). These properties are investigated in the laboratory before biologic behavioural tests takes place [2, 18]. The remaining two evaluation stages are In Vitro Assessment (In vitro term is used to refer to a test setup that produces cells extracted from a living organism outside the body in controlled laboratory conditions) [5] and In Vivo Assessment (The biologic behavioural of biomaterial is in vivo (animal) experiments) [19, 20].

Prospect Tips in Biomaterials: Biomaterials are the backbone of the medical device industry, an important element of health care globally. However, many challenges are there to be solved [21]. This research is basically on development of artificial bone using locally sourced clay with alumina and determination of the properties of the product compare

with international standard. If the local raw material is found suitable, it will help the medical personnel to meet up with demand.

2. MATERIALS AND METHODS

Materials

Nsu clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) is a large deposit of secondary (sedimentary) clay found in Eleme, Mbanjo Local Government Area of Imo State, Nigeria. Alumina (98.48% - Al_2O_3) used was sourced in processed form in a chemical shop at Aba market, Abia state, Nigeria.

Chemical Analysis

The chemical analysis of Nsu clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) was determined using minipal4 EDS – XRF machine.

Body Preparation and Production Processes

The Nsu clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) used was air-dried for 4 days after sourced and oven dried at 110°C for 24 hrs. It was crushed and milled using jaw crusher and ball mill machine, and sieved with mesh 60. The raw materials were used to compose seven samples A-G (of 11 pieces each) with variation of 10% in (90% - 30% Alumina and 10% - 70% Nsu clay) body compositions. Accurate weighing of samples as stated above were done using chemical weighing balance Metra TL 3000 model and they were used to prepare clay slip body. The samples were shaped using casting process method, allowed to air dried at room temperature for 7 days and dried in a drying cabinet for 5 days at a temperature of 110°C . They were sintered at temperature of 1400°C using front loading electric, J W Ratchliff, P U 131 type of kiln.

Determination of Properties of the Products

2.4.1 Linear Shrinkage Test

The linear shrinkage of the samples was determined (on eleven test pieces from each sample) by considering both the dried and firing (sintering) shrinkage. The test pieces produced were given mark at the top center (4cm) with sharp object. The percentage change in length of the samples after drying and sintering were determined to know the linear shrinkage using the following formulas;

$$\% \text{ drying shrinkage} = \frac{(WL-DL)CM}{WL(CM)} \times \frac{100}{1} \quad 1$$

$$\% \text{ Firing shrinkage} = \frac{(DL-FL)CM}{DL(CM)} \times \frac{100}{1} \quad 2$$

$$\% \text{ Linear shrinkage} = \frac{(WL-FL)CM}{WL(CM)} \times \frac{100}{1} \quad 3$$

Where DL = Dry Length, WL = Wet Length and FL = Fired Length

2.4.2 Apparent Porosity and Bulk Density

The tests were carried out on eleven test pieces from each sample. The pieces were weighted (w1) after

through drying. The pieces were evacuated in a vacuum vessel. Water was introduced and covered the specimens completely under pressure. Saturated specimens were weighed in air (w_2) and in water (w_3). The weight of the absorbed liquid (w_2-w_1) was used to deduce the volume since density of water is 1g/cm^3 . The value also represents volume of the open pores in cubic centimeters. The total volume of a specimen is (w_3-w_1).

$$\text{The \% Apparent porosity} = \left[\frac{w_2-w_1}{w_2-w_3} \right] \times 100 \quad 4$$

$$\text{Bulk density} = \frac{w_1}{w_2 - w_3} \quad 5$$

2.4.3 Compressive Strength Test

Eleven test pieces each from each sample were crushed using compressive strength tester (Buehler hydraulic press). The load (Force) applied before the specimens fractured were recorded. Samples were mounted in turn on compressive strength tester and load was applied axially at a uniform rate by operating the pump handles in an up and down movement till it failed.

$$\text{Compressive strength (stress)} = \frac{F}{A} \quad 6$$

A = Area, F = Force

4. RESULT

4.1 Result of the Chemical Analysis

Table-1: Result of chemical analysis of the raw Nsu clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$)

Parameters	SiO ₂	Al ₂ O ₃	CaO	TiO ₂	Fe ₂ O ₃	MgO	K ₂ O	Na ₂ O	Loss on ignition	Other oxide
Nsu clay % Oxides composition	46.56	36.60	0.85	0.69	0.05	0.65	0.70	0.08	10.90	3.92

4.2 Graphical Presentation of Result of Physical Properties of the Artificial Bone Produced

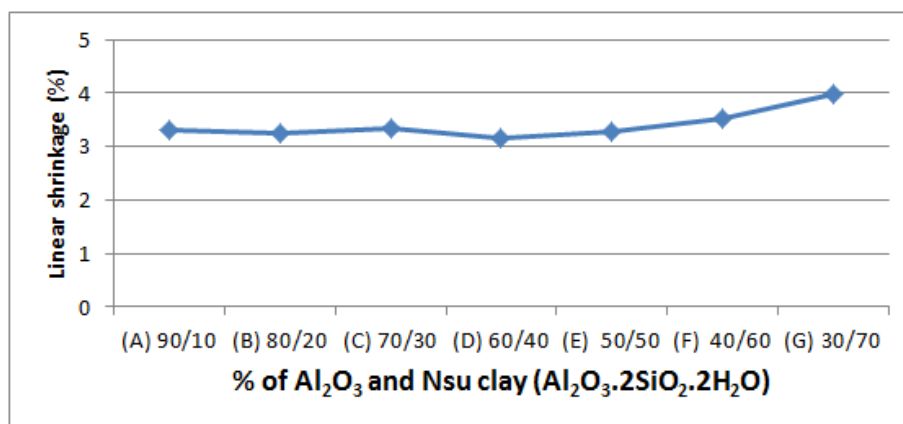


Fig-1: Effects of Percentage Composition of Al₂O₃ and Nsu Clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) on Linear Shrinkage of the Artificial Bones

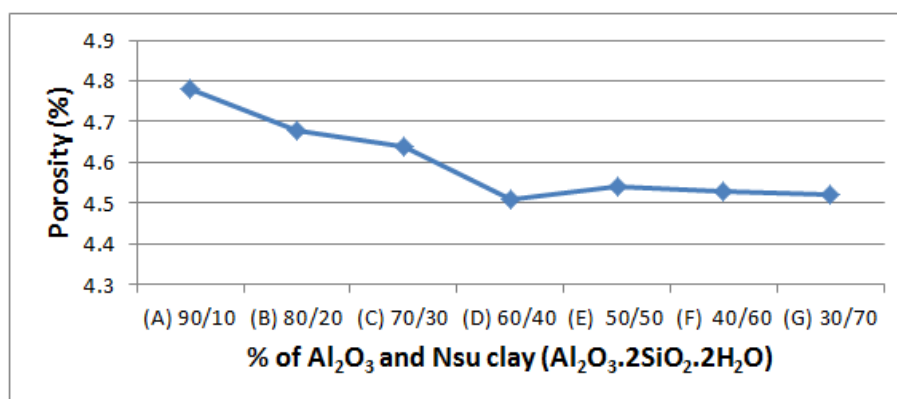


Fig-2: Effects of Percentage Composition of Al₂O₃ and Nsu Clay ($\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$) on Porosity of the Artificial Bones

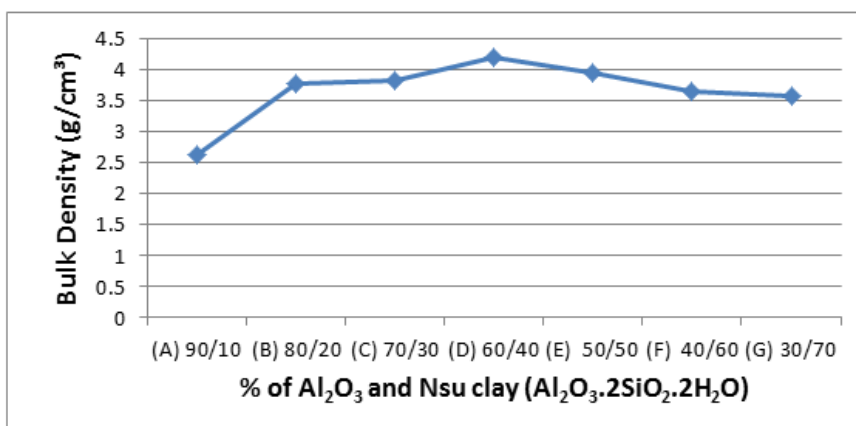


Fig-3: Effects of Percentage Composition of Al₂O₃ and Nsu Clay (Al₂O₃·2SiO₂·2H₂O) Bulk Density of the Artificial Bones

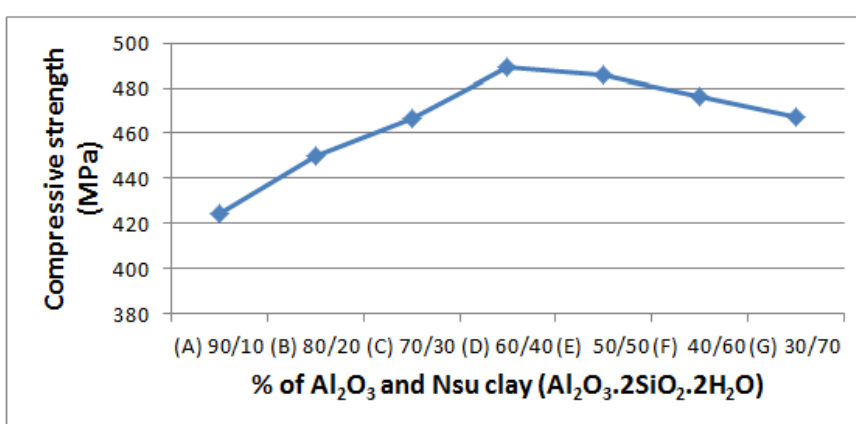


Fig-4: Effects of Percentage Composition of Al₂O₃ and Nsu Clay (Al₂O₃·2SiO₂·2H₂O) on compressive strength of the Artificial Bones

4. DISCUSSION

The result of chemical analysis of the raw material as presented in table.1 revealed that the contents of SiO₂ and Al₂O₃ in Nsu clay (Al₂O₃·2SiO₂·2H₂O) were 46.56 % -SiO₂ and 36.60 % -Al₂O₃. This indicated that the percentages of SiO₂ and Al₂O₃ contents of the clay minerals are within the acceptable range of (40% and above for SiO₂) and (23-45% for Al₂O₃) for particular clay useful for porcelain production [22-24]. Also, the iron content (0.05 % - Fe₂O₃) is very low and is of required acceptable standard for a bioceramics product [25]. It was revealed that Nsu clay is a kaolinitic clay deposit. The bonding system of the ceramics plays a vital role in dictating its properties. When ceramics products are exposed to corrosive liquids at particular temperatures, the degree of corrosion/erosion depends on the products grains and the chemical bonding system of the products.

In this research, Alumina (98.48% - Al₂O₃) and Nsu clay were used to compose seven samples A-G (of 11pieces each) with variation of 10% (90% - 30% Alumina and 10% - 70% Nsu clay) in body composition to produce artificial bone. The Sample were shaped using casting method, dried and sintered at 110°C and

1400°C respectively before subjecting them to physical properties test.

The linear shrinkage result of the samples revealed that samples A-G had range of 3.17% -3.98% (Figure 1). This result indicated that the lower the % composition of alumina the higher the rate of shrinkage. This may be as a result of high loss on ignition (10.90%) of the Nsu clay as shown on table.1. The more a ceramic product shrinks, the less porous it is, hence the denser it becomes [26]. The result therefore shows that, the value obtained are within the recommended range (2-10%) for porcelain [27] and these values fall within the acceptable shrinkage values of dental porcelain and other bioceramics products [25].

The result of percentage porosity indicated that the Samples A-G had range of 4.78% - 4.51% (Figure 2). It was discovered that the decrease in the % composition of alumina which lead to increase in % composition of Nsu clay brought about reduction in porosity of the samples (Figure 2). It was noticed that the higher the percentage of Al₂O₃ the higher the pores and the higher the porosity of the artificial bone. Also, the higher the percentage of Nsu clay the stronger the bonding system, the lesser the pores and the lower the

porosity of the product (Figure 2). The reduction in values of porosity may be due to the growth of the glass phase which introduced the hindrance of the large pores in the products after sintering. Moreover, when the sintering temperature of a material reached, the more obvious becomes the density, which leads to reduction in porosity [26].

The physical properties of the produced artificial bone investigated shows that samples A-G had range of bulk density of 2.63g/cm³ - 4.20g/cm³ (Figure 3). It was revealed that, the lower the percentage of Alumina the high bulk density of the samples. This may be due to high percentage of loss on ignition of the Nsu Clay and the absolute value of bulk density increases with increasing Nsu clay content. This implies that at higher temperature, densification occurs which resulting in the reduction of porosity and increase in bulk density as indicated in figures 2&3. Bioceramic must have high density with load bearing capacity and excellent corrosion resistance [7]. Samples D and E of the products falls within the acceptable range of values of bulk density for bioceramics (>3.90 g/cm³) products [28, 29].

The result of compressive of the samples shows that samples A-G had range of 424.5MPa - 489.1MPa (Figure 4.). It was revealed that the lower the percentage of alumina the higher the compressive strength of the samples, while the higher the percentage of Nsu clay the stronger the bonding and the higher the compressive strength of the artificial bones. The high compressive strength value may be the outcome of the development of glassy phase which led to the bonding strength of the very low porous artificial bones. Furthermore, the availability of CaO, Na₂O and K₂O in the chemical composition (Table. 1) of the Nsu clay is enough to enhance the process of glassy phase development. Also, the closer to the sintering spot a material is heated, the more obvious becomes the sintering at the temperature [26], thereby reducing percentage porosity, increasing density and linear shrinkage which led to increase in compressive strength (Figure 1-4) value of the artificial bones. Only sample D of the products fell within the recommended ranges of 487 - 669MPa for bioceramics made of inert alumina while others fell with the values of 350 - 550MPa for bioceramics made of porcelain but higher than compressive strength values of the natural bones i.e 130 - 180MPa [28,30].

The result of the physical properties investigated revealed that the lower the alumina contents in the artificial bone compositions which brought about increase in the percentage composition of Nsu clay in the samples, led to the higher bulk density, linear shrinkage and compressive strength with lower percentage porosity of the products (Figures 1-4). However, sample D of the artificial bones gave a better result when considering the moderate high bulk density

of 4.20g/cm³, Low shrinkage of 3.17% and low porosity of 4.51% with high compressive strength of 489.1MPa (Figure 1- 4). Furthermore, the investigated physical properties gave results that are acceptable for a standard artificial bone, this is an archetype product, hence requires a clinical/medical compatibility test (In Vitro and In Vivo Assessment) before it can be put to use.

5. CONCLUSION

The development of artificial bone using alumina and Nsu clay was carried out. The physical properties of the artificial bone produced investigated after Sintering at 1400 °C shows that variation in percentage composition of alumina and Nsu clay had effect on the properties. It was revealed that the lower the alumina the higher the bulk density, linear shrinkage with compressive strength and the lower the porosity. Sample D had the most favourable result when considering the properties stated above. However, the investigated physical properties gave results that are acceptable for a standard artificial bone, this is a prototype product, hence requires a clinical/medical compatibility test.

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Ethics

This article is original and contains unpublished material. The corresponding author confirms that all of the other authors have read and approved the manuscript and no ethical issues involved with declaration of no conflict of interest.

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