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Crystallization of Combeite in 45S5 Bioglass using Bamboo leaf, Turkey eggshell and Sodium oxide

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Abstract: *The development in bioglass ceramic materials has led to its wide applications in medical, biomedical, and tissue engineering. Bioglass ceramics has been used in wound and disease treatment, bone fixing, tissue recovery, scaffolding and in the formation of implantable devices. Bioglass ceramics for these types of applications are expected to be bioactive with good mechanical properties that can suit the desired application. The synthesis of bioglass ceramics containing pure combeite phase has made some of the medical and tissue engineering application possible. Combeite is known to possess mechanical properties that are desirable in bioglass ceramics. It is the phase with strong mechanically features that is formed during the crystallization of 45S5 bioglass at high temperature. The crystallization of 45S5 bioglass ceramics with only combeite ($Na_2Ca_2Si_3O_9$) phase has been of challenge due to the presence of other phase that might form through the process. This work looks at crystalizing combeite in 45S5 biocompatible glass through melting process at temperature 1100 ^oC using Bamboo leaves, turkey eggshell and sodium carbonate as source of silicon dioxide, calcium oxide and sodium oxide respectively. The XRD result showed that at temperature 1100 ^oC bioglass with a single phase of combeite was formed at 2 theta angle of 34.5^o. The SEM image confirms this result as it showed a compact consolidated material due to the crystallization and densification of the 45S5 bioglass. This result showed that bamboo leaves and turkey shells compositions favoured the one spot formation of single crystalline phase of combeite at 1100 ^oC which gives the bioglass its mechanical properties that makes it suitable for most medical, biomedical, and tissue engineering applications*

Keywords— Bioglass; Combeite; Bamboo leaves; Turkey eggshell; Melting process; SEM (Scanning electron microscopy); XRD (X-ray diffraction)

1. Introduction

In chemical, medical and biomedical fields humans are exposed to injuries such as cuts, bone fracture, defects, nerve breakdown, lung disease and other body harm. These injuries are often treated using synthetic chemicals and metals in the case of bone and tissue injuries. Synthetic chemicals and metals with time might be harmful to the human body and might not have the bioactive and mechanical properties needed to interact effectively with the human body fluids. This has led researchers to look at alternative materials that are biocompatible, non-toxic and possesses the mechanical features that can easily adapt with the human system. One of such material is bioglass ceramics with combeite phase. Combeite ($Na_2Ca_2Si_3O_9$) is the mechanical phase present in 45S5 bioglass during high temperature heat treatment and is known to possess unique mechanical properties that make bioglass applicable in bone treatment, tissue repair and scaffolding compared to its amorphous form [1]. Depending on the synthesis technique and heat treatment other phases such as devitrite, silico-rhenanite, wollastonite ($CaSiO_3$) etc., [2] are formed and this affect the application of the bioglass ceramics. Thus, it becomes important to synthesize pure bioglass ceramics with only combeite phase.

Bioglass is a material that is biocompatible and response to specific biological changes in living tissues and material interface by forming bond between the material and the tissue. Bioglass is composed of materials that occur naturally in the body. There are different types of bioglass based on the material compositions such as the conventional silicate glass (45S5 bioglass), phosphate based bioglass and Borate based bioglass [3]. Bioglass 45S5 is a bioglass that contain in weight percent silicon dioxide (45%), calcium oxide (24.5%), phosphorous oxide (6%) and sodium oxide (25%). The Silicon dioxide (SiO_2) component in bioglass is also known as silica, it is an oxide of silicon and is white or colorless crystalline commonly found in quartz. It is usually used in the manufacturing of glass, ceramics and abrasives. Calcium Oxide (CaO) is a colorless, cubic crystalline or white amorphous substance, commonly known as lime. It is widely used in industry for sugar purification, preparing bleach powder, glass, cements etc. Sodium oxide (Na_2O) is an alkali metal oxide and is often used for optic, glass and ceramic applications. Research has shown that phosphorus and calcium oxides molecular proportions in bioglass are similar to those found in bones [4].

Bioglass can be synthesized through melting and sol-gel processes. Melting method involves heating the mixture of starting precursors at high temperature. Sol-gel method involves the formation of metal salt precursors and organic metal solution and the formation of gel through chemical reaction. The gel is made to undergo different treatment steps such as such aging, drying, stabilizing and calcining at about 700^{o}C [5]. Bioglass has been used in implant devices in human body for repairing, replacing and filling damaged bones, it acts as bone material and aid the formation of new bone materials, heal diseases and is used as substrate for stem cell [4-6].

Most bioglass ceramics developed over the years are from synthetic chemical materials that might be harmful to the

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human body over a period of time and with the increasing demand of bioglass in medical, tissue and engineering applications. The fabrication of bioglass that are biocompatible with desired mechanical properties that are less harmful to human body and devices over a period of time becomes inevitable. This has made researchers to look at recycling of bio-waste materials that have the required composition for bioglass synthesis [7].

Bio-waste or biodegraded waste consist of mainly organic materials such as green waste, food waste and even biodegradable plastics. Recycling of such waste can naturally benefit the environment. Research has shown that the use of bio-synthesized calcium and silicon oxide as alternative to synthetic forms in producing biomaterials for medical applications has lots of beneficial effects as well as cost reduction [7]. This research work makes use of Bamboo leaves waste as source of silica, turkey eggshell waste as a source for calcium oxide and sodium carbonate as source for sodium oxide for the synthesis of 45S5 bioglass ceramics through melting process. Bamboo leaves is known for its high silicon dioxide composition and other elements [8] and turkey eggshell is known to contain calcium oxide and other components [9].

2. MATERIALS AND METHOD

The following materials and equipment were used for this research work. Bamboo leaves, Turkey eggshells, Na_2CO_3 (99.9%), 0.5M hydrochloric acid, Distilled water, Muffle furnace, platinum crucible, mortar and pestle, air oven, brass molds, bowl, pipette, and beakers.

2.1 Synthesis of Silicon Dioxide

Bamboo leaves were collected and washed thoroughly with water to remove impurities and cut into smaller bits. The washed leaves were placed in a beaker containing 0.5 M hydrochloric acid at 60°C for 30 minutes. It was removed from the acid solution and rinsed severally with distilled water. The treated bamboo leaves were air-dried and placed in an oven at 110°C for 24 hours to dry. The dried bamboo leave were placed in a muffle furnace at 600°C for 4h.

2.2 Synthesis of Calcium Oxide

Waste turkey eggs were collected and washed thoroughly with water without its membrane. It was placed in an oven and dried at 120°C for 2 hours. The eggshells were ground into fine particles after drying. The powdered eggshells were treated in a two-step thermal treatment. Firstly, the powdered eggshells were placed in an oven at 450°C for 2 hours at rate 5°C/min to destroy organic residues in the powder. Secondly, the powdered eggshells were placed in a furnace at 900°C for 2 hours at rate 0.50°C to convert the eggshell powder to calcium oxide by releasing carbon dioxide.

2.3 Crystallization of Combeite in 45S5 bioglass

The mixture of 45 % silicon dioxide, 25% CaO and 25% Na_2O by weight powders was placed in a crucible and melted at 1100°C for 3 hours inside a furnace. The resulting molten mixture was quenched in water. Followed by grinding and sieving the glass pieces to micrometer particle sizes below 30 um.

3. RESULTS AND DISCUSSION

Fig. 3.1 shows the SiO_2 synthesized from bamboo leaves through calcination. Fig. 3.2 shows powder eggshell after grinding and powder gotten after heating the eggshell at 450°C for 2 hours. At 450°C organic residues were destroyed and powder turns dark ash in colour. Fig. 3.3 shows calcium oxide gotten from eggshell after heating eggshell powder at 900°C for 2 hours in muffle furnace and the powder turns whitish in colour.

Fig. 3.1 SiO_2 synthesized from bamboo leave

Fig. 3.2 Powdered eggshells after grinding and Eggshell powder after heating at 450°C for 2 hours

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Fig. 3.3 Calcium oxide from eggshell at 900°C for 2 hours

Fig. 3.4 is the XRD diffraction pattern result for the synthesized 45S5 bioglass synthesized. The XRD was carried out on SiO_2–CaO–Na_2O bio-glass to determine the bioglass amorphous nature and its crystalline phases. In Fig. 3.4, the XRD showed the absence of a broad hump

around 30° with sharp peaks indicating that the characteristics amorphous nature of glass had disappeared due to the high temperature sintering resulting in the crystallization of the bioglass powder. Bioglass powders are known to crystallize and compact at higher temperatures from 1000°C and above and forms dense materials such as scaffolds with porosity [10]. 45S5 bio-glass particles are known to start crystallization in the range 550°C-750°C, with densification step taking place within this range after glass transition temperature is reached, and the particles connected through sintering necks [11-13]. Fig. 3.4 showed that after sintering and quenching, the XRD pattern showed a single sharp peak identifying combeite ($Na_2Ca_2Si_3O_9$) denoted by the symbol (x) at 2 theta angle (34.5°) as the crystalline phase present in the sample according to the standard PDF #22.1455. The presence of $Na_2Ca_2Si_3O_9$ in bioglass improves the mechanical properties of Na-containing glasses which are required in medical and engineering applications.

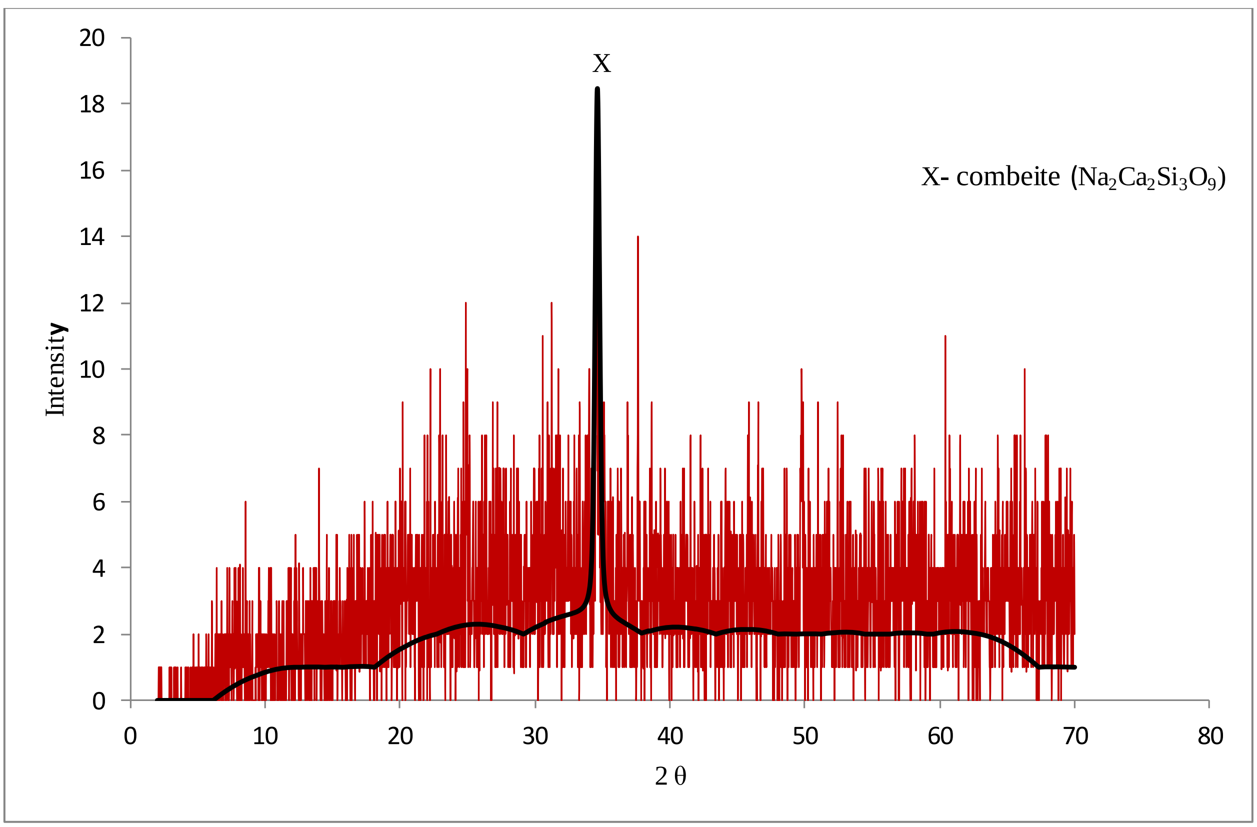

Figure 3.4 XRD pattern for the synthesized 45S5 bio-glass at high temperature

Fig. 3.5 shows the SEM image of the crystallized 45S5 bioglass. The image showed a compact consolidated material due to crystallization and densification of the 45S5 bioglass at high temperature.

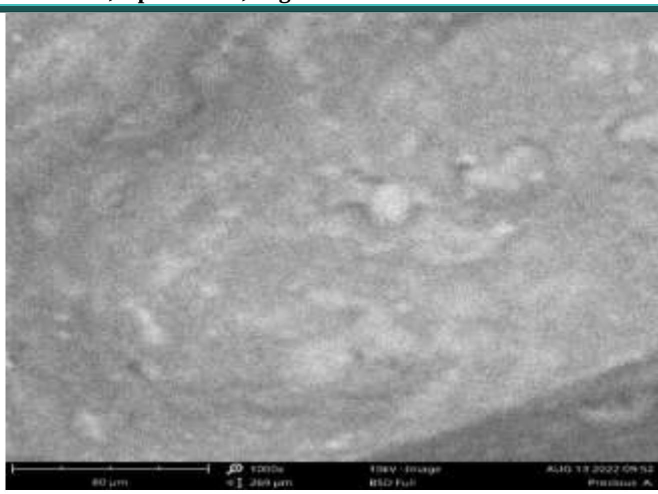

Fig. 3.5 SEM image of the synthesized 45S5 bioglass at high temperature

4. CONCLUSION

Bioglass 45S5 was successfully synthesized by using bamboo leaves and turkey eggshells as sources of silicon dioxide and calcium oxide. Sintering the bioglass at 1100ºC resulted in the formation of single crystalline phase of combeite ($Na_2Ca_2Si_3O_9$) at 2 theta angle of 34.5º with no other phase present. This shows that bamboo leaves and turkey shells compositions favoured the one spot formation of single crystalline phase of combeite which gives the bioglass its mechanical properties that makes it suitable for most medical, biomedical, and tissue engineering applications..

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