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BIOACTIVE GLASS BONE IMPLANT MATERIALS FROM CANE WASTE AND EGG SHELL WASTE

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Abstract :
Sugarcane bagasse and eggshell waste are common types of waste found in Indonesia. This article introduces an innovation to utilize these two waste materials as bone implant materials, offering a way to increase their economic value. Bone implant materials primarily consist of SiO_2 and CaO, both of which can be derived from sugarcane bagasse and eggshell waste. The process of making bone implants begins with the purification of sugarcane bagasse and eggshell waste to produce SiO_2 and CaO compounds. Characterizations conducted at each stage of the reaction process in this research include FTIR, XRD, and SEM.The purification of sugarcane bagasse into SiO_2 is carried out using a hydrothermal reaction, while the purification of eggshell waste is performed using the calcination method. Once the high-purity raw materials are obtained, the process continues with the production of bioactive glass. Bioactive glass is a biomatrix material used as a bone implant material. Its composition requires SiO_2 = 45%, Na_2O = 25%, CaO = 25%, and P_2O_5 = 5% (weight/weight). The sol-gel method is employed to produce the bioactive glass.After the bioactive glass material is produced, it is further characterized to evaluate its structure and properties. Additionally, bioactivity testing is conducted using SBF (Simulated Body Fluid) to ensure its safety when used as a bone implant and its interaction with body tissues.

Keywords: Bioactive Glass, Egg Shell, Sugarcane Bagasse

INTRODUCTION

The SDGs (Sustainable Development Goals) are an international sustainable development plan aimed at addressing global issues, agreed upon by leaders worldwide, including Indonesia, with 17 main goals, one of which is Health. Bone health issues remain a common health problem in Indonesia. According to Kemenristek (2017), the need for bone implants in Indonesia reaches 80,000-100,000 pieces per year due to the high number of accidents and osteoporosis-related bone problems. The high number of traffic accidents from 2014 to 2019 is estimated to reach 107,500 cases, many of which involve bone fractures, leading to an increasing demand for bone implants. However, this rising demand is not matched by the availability of domestically produced bone implants, resulting in a market flooded with high-priced imported products, reaching up to 400 USD, making it difficult for the public to afford them (Kemenristekdikti, 2017).

Waste issues always pose significant concerns, one of which includes bagasse and eggshell waste, commonly found in daily life. Bagasse waste is abundant from the sugar production process, generating approximately 0.8 million tons of waste annually (Iqbal et al., 2016). Bagasse contains a high level of silica (SiO_2). In 2018, Indonesia produced 1.64 million tons of chicken eggs, with about 12% being shells, resulting in approximately 196,800 tons of eggshell waste (Nurlaela et al., 2014). Eggshell waste can be utilized due to its high calcium content. These two types of waste can be used to produce Hydroxyapatite (HAp). Hydroxyapatite (HAp), with the chemical formula $Ca_{10}(PO_4)_6(OH)_2$, is the main inorganic component of bone and teeth. However, the hydroxyapatite used in the medical field in Indonesia is still imported, necessitating the development of hydroxyapatite synthesis research (Puspita et al., 2017).

Currently, many studies are developing research on bone implants. One material used in bone implants is synthetic biomaterial, namely Hydroxyapatite (HAp) (Dumitrescu, 2011). Bioactive glass 45S5 (SiO_2-Na_2O-CaO-P_2O_5) influences and causes responses from living tissues, organisms, and cells in the formation of Hydroxyapatite, which can react with body fluids, resulting in the formation of a hydroxycarbonate apatite (HCA) layer, and has osteoconductive properties to grow bone in pores and bond to the host bone surface (Kamalian et al., 2012) (Hidayat, 2017). Hydroxycarbonate apatite (HCA) has a similar chemical composition to bone and can interact with damaged bone collagen fibrils for bonding. Protein adsorption, collagen fibril incorporation, bone progenitor cell attachment, cell differentiation, extracellular bone matrix excretion, and mineralization are involved in the formation of the HAp bonding bone layer (Abbasi et al., 2015). The bioactivity properties of BG occur by forming a strong bond with the bone through a hydroxyapatite (HAp) and hydroxycarbonate apatite (HCA) layer when in contact with body fluids (Hidayat, 2017). The osteoconductive properties of BG allow for bone growth within pores and bonding to the matrix surface (Kamalian et al., 2012). The osteogenic (osteoinductive) properties mean that soluble silica and calcium can stimulate osteogenic cells to produce bone matrix (Jones, 2015; Jones et al., 2016) (Radandima, 2020). Based on the above explanations, it is evident that utilizing bagasse and eggshell waste is highly beneficial and offers promising opportunities.

RESEARCH METHOD

Preparation of SiO_2 and CaO

The process to produce SiO_2 from bagasse begins with burning the bagasse to turn it into ash, which helps to break down water, carbon, and other components. According to various studies reviewed by the author, the burning of bagasse can be conducted at different temperatures and durations: at 300°C, 400°C, 500°C, 600°C for 2 hours (Hasan and Karya, 2017), 500°C for 30 minutes (Purnawan et al., 2018), 600°C for 3 hours (Megawati et al., 2018), 600°C for 6 hours (Soleh et al., 2020), 700°C for 4 hours (Azzahra, 2021), 900°C for 2 hours (Hidayat, 2017), 1000°C for 4 hours (Bimo, 2019), and 1500°C (Khoirianti, 2019). According to Hasan and Karya (2017), increasing the temperature above 700 or 800°C will increase the quantity of silica.

Afterward, the bagasse is cleaned of SiO_2 impurities using 0.1 M HCl (Azzahra, 2021) (Adibah, 2017), 1 M HCl (Hasan and Karya, 2017), 2 M HCl (Radandima, 2021), 0.5 M HCl (Gomez et al., 2019), 2.5 N HCl (Indrasti, 2020), and 6 M HCl (Purnawan, 2018). After cleaning the ash with HCl, it is washed with distilled water until a neutral pH is achieved (Rifani, 2017) (Khoirianti, 2019). The neutralized ash is then reacted with 2 N NaOH (Azzahra, 2021) (Yensya, 2019), 1 M NaOH (Hasan and Karya, 2017), 2.5 N NaOH (Indrasti, 2020), 0.5 M NaOH, 1 M NaOH, 1.5 M NaOH, and 2 M NaOH (Megawati et al., 2018). The concentration of NaOH is crucial as it influences the amount of silica produced. According to Zuwanna et al. (2021), higher NaOH concentrations result in higher silica yields. The reaction between NaOH and the bagasse ash is carried out using a magnetic stirrer at 200 rpm until boiling (Megawati et al., 2018). The mixture is then filtered with filter paper to obtain a clean solution. The resulting solution is titrated with 2.5 M HCl (Chindaprasirt & Ubolluk, 2020), 2 M HCl (Radandima et al., 2021), or 1 M HCl and 1 M H2SO4 until pH 7 (Sapawe et al., 2018). The titrated product is dried at 120°C for 24 hours (Channoy et al., 2018). Once dry, it is ground with a mortar to produce SiO2 powder.

For processing CaO from eggshell waste, the first step is to clean the impurities from the eggshells. According to Nurlaela et al. (2014), this is done by boiling the eggshells in boiling distilled water for 30 minutes. After cleaning, the eggshells are milled to a 200 mesh size (Radandima, 2021) before being calcined at 900°C (Prabakaran et al., 2005) as stated by Nurlaela et al. (2014).

Making Bioactiveglass Composites

The next process is the production of Bioactive Glass. The production of Bioactive Glass uses the sol-gel method with the compound ratio of SiO_2 : Na2O : CaO : P2O5 being 45% : 25% : 25% : 5% (Radandima, 2020). The first reaction involves mixing SiO_2 with NaOH for 30 minutes, stirred with a magnetic stirrer at 300 rpm at room temperature. The ratio between SiO_2 : NaOH : H2O is 2.7:1.94:50 in grams (Adams et al., 2013). Next, 0.1 M HNO3 is added and stirred with a magnetic stirrer at 450 rpm for 45 minutes at room temperature (Arofah, 2020). Then, CaO and TEP are added, each stirred for 45 minutes, and finally, an additional hour for finishing (Durgalakshmi, 2020). The obtained sample is left to stand at room temperature in an airtight condition for 72 hours. After that, it is dried for 72 hours at 60°C, then heated at 120°C for 40 hours, and finally calcined at 700°C for 2 hours (Randandima, 2020).

RESULTS AND DISCUSSION

The characterizations used in the bioactive glass research are XRF, XRD, FTIR, SEM, and bioactivity testing. According to Radandima (2020), XRF characterization is used to determine the elemental composition of the sample. When characterizing bagasse using XRF, the SiO_2 content is particularly important. Hasan and Karya (2017) found that the higher the initial burning temperature of the bagasse, the higher the SiO_2 yield, emphasizing the importance of temperature control. For instance, SiO_2 content at 600°C is 36.24%, significantly higher than at 300°C, which is 57.39%. Sholeh et al. (2020) reported that burning at 600°C for 6 hours produced 93.295% SiO_2, while Megawati et al. (2018) reported 54% SiO_2 at 600°C for 3 hours, indicating that both temperature and time influence SiO_2 purity. The concentration of HCl used also affects the amount of silica produced, with higher HCl concentrations yielding more silica (Meidinaryasti et al., 2020). Additionally, the NaOH concentration affects silica content, with higher concentrations resulting in higher silica yields (Nurlina et al., 2017). Different sources and types of bagasse also influence SiO_2 yield. For egg shell-derived CaO, XRF characterization showed the highest purity of 99.97% (Radandima, 2020), compared to 55.02% CaO (Nurlaela et al., 2014) when milling to 200 mesh was included.

XRD characterization is used to determine the crystal structure of a sample (Aji, 2020). For bioactive glass, XRD characterization at 700°C showed major crystal phases such as Na4.5Ca3.5Si6O18, indicating reactions between Na_2O, CaO, and SiO_2 during sintering (Adams and Essien, 2016). Combite and tricalcium silicate phases were detected at angles 29.40° and 32.48°, respectively, with combite's presence being significant for selecting an ideal scaffold for new bone growth (Yang et al., 2017).

FTIR analysis of BG45S5 (SiO_2-NaO-CaO-P_2O_5) compared to previous studies on BGQ (SiO_2-CaO-P_2O_5) and BG4 (SiO_2-CaO-P_2O_5) indicated significant differences due to the higher SiO_2 content in BG45S5 (Rahmawati, 2020). BG45S5 showed deep absorption peaks for Si-O-Si bonds, which were absent in the other compositions, indicating its potential for HA formation and enhanced bioactivity due to the presence of silicon atoms as catalysts (Ravarian et al., 2010). The calcination temperature also influenced the reactive properties, with BG45S5 at 700°C showing higher reactivity compared to BGQ and BG4 at 250°C (Ravarian et al., 2010).

SEM characterization of bioactive glass calcined at 700°C revealed a random structure with a heterogeneous surface, flaky particles, and some crystalline areas, providing a porous structure suitable for nanobiocomposites (Essien et al., 2012). Higher temperatures above 700°C caused particle fusion and sintering into larger aggregates, while shorter calcination times were preferable to avoid densification (Luz & Joao, 2011). BG particles exhibited irregular morphology with many nanoparticles forming a porous structure due to interfacial tension during drying (Chen et al., 2017).

Bioactivity testing can be done by immersing the bioactive glass in SBF to observe HA or HCA layer formation on its surface (Radandima, 2020). The carbon and hydrogen content in the BG affects

HA and CHA formation during SBF immersion (Azami et al., 2012; Yazdanpanah et al., 2012). The in vitro bioactivity of BG material is predicted by apatite formation on its surface in SBF. Complete dissolution of compounds during SBF preparation indicates the formation of silanol ($Si(OH)_4$) with ion exchange (Na^+ and Ca^{2+} with H^+ or H_3O^+ from SBF) leading to HCA formation (Adams and Essien, 2016). Prolonged SBF immersion shows increasing HA or CHA formation, indicated by rising pH and widening absorption bands for phosphate and carbonate ions, confirming continuous ion exchange and hydrolysis during the 28-day period (Jones, 2015).

The formation of carbonated hydroxyapatite (CHA) layers has significant implications for various clinical applications, particularly in the fields of bone regeneration, dental implants, and tissue engineering. CHA, characterized by its lower crystallinity and increased solubility compared to traditional hydroxyapatite (HA), enhances bioactivity and biocompatibility, making it a preferred material for biomedical applications (Sari et al., 2021). In bone tissue engineering, CHA coatings have been shown to improve osseointegration, which is crucial for the success of implants. For instance, studies indicate that CHA coatings with hierarchical micro-/nanorod topography optimize the interaction between the implant and surrounding biological tissues, facilitating better cellular attachment and proliferation (Li et al., 2018). The presence of carbonate ions in CHA not only enhances its solubility but also promotes the adsorption of proteins, which play a pivotal role in mediating cellular responses and guiding tissue regeneration (Ohta et al., 2017). This protein layer is essential as it influences the behavior of osteoblasts and other cells involved in bone healing, thereby enhancing the overall effectiveness of the implant (Li et al., 2018).

Moreover, the application of CHA in periodontal therapy has garnered attention due to its ability to promote wound healing and tissue regeneration. Research has demonstrated that CHA can upregulate the expression of genes associated with proliferation and migration in periodontal ligament cells, which is vital for periodontal regeneration following surgical interventions (Zhu et al., 2023). The positive biological effects of CHA in periodontal applications have been supported by histological evidence, showcasing its potential in treating intrabony defects and enhancing healing outcomes. In addition to its applications in bone and dental implants, CHA's properties are being explored in drug delivery systems and regenerative medicine. The porous structure of CHA allows for the encapsulation of therapeutic agents, which can be released in a controlled manner, thereby enhancing the efficacy of treatments (Yao et al., 2021). This characteristic is particularly beneficial in the context of injectable bone repair materials, where CHA microspheres can serve as scaffolds that support cell growth and tissue integration.

CONCLUSION

Based on the discussion above, the utilization of bagasse and eggshells as materials for Bioactive Glass is appropriate and cost-effective since they are waste materials. The factors affecting the purity of silica and CaO in XRF characterization include the initial furnace temperature, HCl concentration, and NaOH concentration. In FTIR and XRD characterization of Bioactive Glass, the calcination temperature is crucial, with 700°C being identified as the optimal temperature in most studies. SEM characterization indicates that temperatures above 700°C in a ternary system lead to the fusion and sintering of particles into large aggregates. For bioactivity characterization, SBF can be used, where the appearance of HA on Bioactive Glass signifies its compatibility with the human body. Based on the discussion above, further research is needed to apply bone implant materials to living cells so that they can eventually be used in the human body.

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