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Fabrication and characterization of bone china using synthetic bone powder as raw materials

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ABSTRACT

The synthetic bone powder was studied as a raw materials for bone china, completely replacing natural bone ash raw materials. The physical and thermal properties of samples obtained by the two bone powders were tested and comparatively studied. Performance tests included pyroplastic deformation, flexural strength, bulk density, sintering shrinkage, water absorption, transmittance, thermal expansion coefficient and the thermal shock resistance. The phase composition and morphology were investigated by X-ray diffraction (XRD) and scanning electron microscopy (SEM), respectively. The results indicated that using synthetic bone powder could shorten the preparation time, reduce the sintering temperature and result in high-quality bone china. The pyroplastic deformation decreased from 9.14% to 7.92%, the three-point flexural strength increased from 123 MPa to 191 MPa, the light transmittance (at a 2-mm thickness) increased from 6.7% to 11.2%, the thermal expansion coefficient decreased from $8.24 \times 10^{-6}\,°C^{-1}$ to $7.69 \times 10^{-6}\,°C^{-1}$, and the thermal shock resistance increased from 140 °C to 180 °C. A continuous interface layer without cracks was produced by using the synthetic bone powder.
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1. Introduction

Bone china is a highly specialized product in terms of its appearance. Being exceptionally white and translucent makes it the world's most expensive type of tableware [1]. Bone china shares some advantages with delicate porcelain, including glazed smoothness and soft gloss appearance. In addition, bone china is a highly specialized product whose properties critically depend on the quality of the raw materials used. This is also to be kept in mind that selection of raw materials and their proportions particularly the bone ash, processing parameters particularly firing atmosphere, maximum temperature attained and the soaking time have strong influence on the development of microstructure which in turn influence the ultimate thermo-mechanical properties of the products [2].

The typical composition of a modern commercial bone china is 25 wt% clay, 25 wt% fluxing material and 50 wt% bone ash [3–5]. Natural bone ash is made of cattle processed bones and was calcined at approximately 1000 °C. It may be noted that poorly calcined bone impairs the rheological properties of the slip and may lead to both a reduced firing range and increased fired porosity [2,6]. Cattle bone is a complex material containing protein, additional organic and inorganic content and moisture which is a limited resource. In cattle bone calcination, the organics are burned off to leave bone ash, such processing is harmful to the environment. Therefore, due to the recent awareness of environmental preservation, the production of bone china is given a new interest, which should further boost its development and consumption [7]. Although the technical quality of bone china is sufficient to justify its high market acceptance, the objective of this study is to further emphasise the aspect of environmental and the limited nature of the raw material.

In the present work, synthetic bone powder was used completely to replace natural bone ash, the traditional raw materials used in bone china formulation. The aim of this study is to evaluate the possibility of using synthetic bone powder based material as bone china. The behavior properties and the microstructure of synthetic bone powder based material have been investigated using sintering study, pyroplastic deformation, flexural strength measurement, light-transmission rate, X-ray diffraction (XRD) and scanning electron microscopy (SEM) techniques. The results showed that the synthetic bone powder can provide bone china with excellent performance.

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2. Experimental

2.1. Sample preparation

A mixture of calcium hydrogen phosphate di-hydrate ($CaHPO_4 \cdot 2H_2O$) and calcite ($CaCO_3$) with a Ca/P molar ratio of 3/2–5/3 was added to 90 wt% water to create a slurry. The calcium hydrogen phosphate di-hydrate was feed grade with a median particle size of 50.0 μm. The calcite was a coarsely agglomerated powder with a median particle size of ~50.0 μm. The slurry was agitated using cobblestone balls in a ball mill at 50 rpm for 24 h at room temperature. The reacted slurry was dried under ambient conditions and then heat-treated in air at 1200 °C for 2 h to produce the synthetic bone powder.

The materials used in this study were synthetic bone powder and natural bone ash, K-feldspar, calcite, kaolin, silica, clay and scrap. The composition of the bone china was 31.0 wt% clay (*Longyan* kaolin 13.0 wt%, *Suzhou* kaolin 4.0 wt%, *Datong* kaolin 5.0 wt%, K-18 kaolin 4.0 wt%, GF-88 kaolin 3.0 wt%, *Yixian* kaolin 2.0 wt%), 14.0 wt% K-feldspar, 5.0 wt% silica, 5.0 wt% calcite, 41.0 wt% bone powder (the same contents of synthetic bone powder and natural bone ash) and 5.0 wt% scrap porcelain powder of calcined waste bone china body.

The raw materials and chemical reagents used throughout this work were kindly supplied by Nanjing Haoqi Advanced Materials Co., Ltd. The composition and impurity content of the starting materials were reported in a previous study [8]. The quartz and K-feldspar were finely ground with a median particle size of 3.0 μm. The clays used were manufactured using the following general processing procedure. The clay was wet grinded and the milled clay was then sieved, passed over magnets, and washed with water, followed by filtering, drying and forming a mud cake with a median particle size of 3.0 μm. The scrap porcelain powder of calcined waste bone china body was a coarsely agglomerated powder with a median particle size of ~1.0 mm.

The raw materials were mixed in a wet mill. Balls and water were added as necessary to achieve a proportion of material: ball: water = 1:2:0.36. Then, three types of dispersants consisting of sodium humate (0.35 wt%), sodium silicate (0.15 wt%) and sodium tripolyphosphate (0.08 wt%) were added into the wet mill.

At present, the preparation process of bone china using calcined cattle bone as raw material is as follows: ball milling, sieving, water washing, iron removal, mud pressing, slurry preparation, slip casting, drying, bisque firing and glaze sintering in an electric furnace. However, the manufacturing process of bone china using synthetic bone powder can eliminates three steps: water washing, mud pressing and slurry preparation. The synthetic bone powders can simplify the process and shorten preparation time, thus increasing productivity compared with natural bone ash.

The manufacturing processes were as follows. The raw materials were ball-milled for 16.0 h and passed through a 200-mesh sieve. Next, the iron in the slurry was removed by an iron stick. Shaping was carried out in this study by slip casting to produce two types of shapes: cylindrical rod (Φ 6 × 95 mm) and rectangular prism (95 × 30 × 5 mm). The pieces were naturally dried for 72 h or in an oven at 60 °C for 24 h. The dried green bodies were then sintered at temperatures between 1200 °C and 1265 °C for 2.0 h in an electric furnace.

2.2. Characterization

Upon the completion of firing, cylindrical rod specimens (Φ 6 × 95 mm) were cast to quantify changes in flexural strength. The rectangular specimens (95 × 30 × 5 mm) were cast to quantify changes in pyroplastic deformation, sinter shrinkage, bulk density, transmission properties and other properties; 6 samples were prepared under each condition in our study. The chemical composition of the raw materials and the fired samples was analyzed by X-ray fluorescence (XRF model Shimadzu-1800). The particle size distribution of the milled mixture was determined using a Malvern Particle Size Analyzer, (Model 2602 LC) via a laser diffraction technique. All of the experiments were carried out using a 200-mesh sieve. The rheological properties of the slip were characterized using a digital viscometer (Brookfield DV-II+). To correlate the pyroplastic deformation with the crystallization phases developed in the microstructure, the sintered samples were analyzed by XRD and SEM. Phase analysis was conducted using an X-ray diffractometer (Thermo Electron Corporation ARLXTRA). The samples were scanned over the range $2\theta = 10–80°$ at a scanning speed of $0.5° \min^{-1}$ using Cu·Kα radiation at 50 kV and 30 mA. The crystalline phase compositions of the mature bodies were analyzed using the Search Match software program and were compared with the PDF2 database. The content of each phase was calculated using the Rietveld whole pattern fitting quantitative analysis method with Jade 7.0 and Fullprof software. For SEM, specimens were polished using 20, 10 and 5.0 μm diamond pastes after grinding with silicon carbide powders as abrasives and lubricated with water. The polished surface of the samples was etched in a 5.0 wt% HF solution for 3.0 min and coated with gold/carbon. A JEOL S-4800 FE-SEM (operating at 5 kV) was used to examine the microstructure of the samples, with secondary electron images (SEIs) being predominantly used.

The amount of pyroplastic deformation of a bar was measured by determining the value of d, which is a measure of how the bar has sagged, as shown in Fig. 3. The pyroplastic deformation percentage was then calculated as follows [9]:

$$A = \left(d/D\right) \times 100\% \tag{1}$$

where D is the unsupported length of the bar.

To determine d, a metal bar was placed against the sagged ceramic bar on the concave side so that its ends were equidistant from those of the ceramic bar, and d was measured using a vernier caliper.

Sintering shrinkage is described by the following expression:

$$\lambda = \frac{(L_0 - L)}{L_0} \times 100\% \tag{2}$$

where λ is the shrinkage of the body after being fired, L_0 is the length between the two baselines before firing and L is the length of the two lines after firing.

Both the bulk density and water absorption of the bone china samples were measured by the Archimedes method using distilled water as the medium according to the ASTM C373-88 (2006) standard [10]. The specimens were 20 × 20 × 5 mm in size and were immersed in boiling water for 1.0 h and then cooled to room temperature before their wet weight was measured. In the study, six samples were used to measure the properties.

The relative density in this study was the ratio of bulk density to true density of the samples, and the true density of the samples was calculated using the simple rule of mixtures, which was consistent with the relative proportions of the phases.

The mechanical properties of bone china were measured on an electronic universal material testing machine (CMT 6203, Shenzhen Shiji Tianyuan Instrument Co., Ltd., Guangdong, China). Three-point bending tests were carried out using cylindrical rod specimens according to the ASTM D143-94 (2007) standard [11]. The flexural specimen size was 6 mm (diameter) × 95 mm (length), with a span of 50 mm and a crosshead velocity of 0.5 mm/min.

The flexural strength was calculated using the following expression:

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