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Mesoporosity, thermochemical and probabilistic failure analysis of fired locally sourced kaolinitic clay

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Weibull distribution

Abstract A dense and mesoporous ceramic from locally sourced Nigerian clay under fracture-strength test were produced and the reliability analysis of the fractured strength was conducted using a three-parameter Weibull probability distribution. The samples were prepared by addition of starch (0–20wt%), pressed at 60 MPa and fired at 1300 °C. The as-received Nigerian clay, dense and porous ceramic were characterized using XRD, XRF, TGA/DTA, PSD, multi-point BET and FESEM. The fracture strength of the samples (33 each) was determined using a three-point bending test. The fracture strength data were analyzed using three-parameter Weibull probability distribution. From the characterization results, a mullite ceramic formed at a sintering temperature of 1300 °C. The threshold strength for the three-parameter Weibull provides the strength below which the dense and the porous ceramic will not fail. The Weibull moduli of the ceramics at different starch compositions show that failure modes in these materials are not identical. The Weibull modulus increases with increase in percentage starch from 0% to 15%. However, the value decreases with 20% starch addition. Reliability analysis provides a detailed interpretation and assessment of the fracture strength of the porous ceramics.

1. Introduction

Due to the rising cost of engineering ceramics and the limitations of polymeric membranes with regard to mechanical, chemical and thermal properties, researchers have resorted to using inexpensive clay minerals in ceramic membrane production for industrial processes such as reaction, separation and purification applications (Mauricio et al., 2011; Bose and Das, 2013; Emani et al., 2014). These clay minerals can be a cheap source of mullite ceramic, which can be obtained after sintering at the appropriate temperature (Bai, 2010). Despite the availability of literature on the strength data of these porous fired clay materials, a detailed analysis of the strength and

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reliability of these materials is important, since they operate under pressure-based driven process. However, the strength and reliability of these porous fired clays are not well understood. As a rule-of-thumb, brittle materials have variations in their fracture strength under the same fabrication conditions; this is due to the fact that brittle materials are prone to flaws/pores during the fabrication process, which become pronounced after sintering. Many researchers have reported single fracture strength at a particular sintering temperature and compaction pressure as the actual strength of these clay-based porous materials. For example, porous clay based membranes were produced at a distributed pressure of 2 Kg and fired at temperatures in the range of 850–1000 °C; the membranes produced have fracture strength values in the range 3–8 (Nandi et al., 2008). In addition, Jana et al., 2010 reported a fracture strength of 11.55 at a sintering temperature of 1000 °C while preparing porous microfiltration membranes by paste casting for chromate removal from wastewater. Several years later, Yakub et al. (2012) extended the research by reporting the deviation of the fracture strength of porous fired clay at a sintering temperature of 955 °C with a porosity in the range 36–47%; the fracture strength obtained are in the range 3.89 ± 0.09–7.14 ± 2.26 MPa. Moreover, Sahnoun and Baklouti, 2013 reported strength in the range 6.66–10.63 for porous fired kaolin clay prepared by compaction at compaction pressure in the range of 15–75 at sintering temperature in the range 800–1250 °C. Lastly, Emani et al. (2013) reported the fracture strength of kaolin-based porous fired membrane using compaction at a pressure range of 29–49, firing temperature of 900–1000 °C and porosity in the range 35–39%; the fracture strength obtained is in the range 7.81–11.

Conventional two-parameter Weibull assumes the strength below which all the materials will not fail (threshold strength) to be zero and the scale parameter to be the strength at 62.3% of the materials. In addition, the two-parameter Weibull probability distribution is suitable for a small sample size of 20 and below (Roos and Stawarczyk, 2012). However, in reliability statistics, higher Weibull modulus value is an indication that the threshold strength is larger than zero and should not be ignored (Han et al., 2009). Therefore, three-parameter Weibull probability distribution gives a detailed reliability of the strength below which all the tested materials will not fail (threshold strength) and uniformity of the strength data in the form of Weibull modulus. This is normally achieved by testing the strength of several samples (normally around 30) under the same fabrication condition and analyzing the data with three-parameter Weibull probability distribution (Han et al., 2009). Although, many studies have reported the strength of porous fired clays, there is limited research on the analysis of the strength data using three-parameter Weibull distribution. Therefore, we report the fabrication of dense and mesoporous fired Nigerian clay with starch as a pore former, characterization of the clay and the mesoporous fired clay and the analysis of the fracture strength using three-parameter Weibull probability distribution. The novelty of this work is to determine the strength below which these clay-based membranes will not fail, determine the uniformity of the data and the effect of porosity on the Weibull modulus. No attempt has been made previously on characterization and flexural strength analysis of dense and mesoporous fired Nigerian clay. The objective of the present research was to conduct XRF, XRD, PSD, FESEM and BET multi-point of as-received, dense and porous ceramics. The analysis of the fracture strength of the dense and porous ceramics was conducted by three-parameter Weibull probability distribution.

2. Materials and methods

2.1. Clay characterization

The as-received clay obtained locally from Kankara, Nigeria was screened through 50 μm sieve. Particle size analysis was carried out using Malvern particle mastersizer 2000. Mineralogical composition of the raw and fired clay (1000 and 1300 °C) was determined with Siemens Diffractometer D5000 equipment, Cu Kα (1.54056 nm) radiation, step size angle of 0.05°, scan rate of 2° in 2θ unit and scan range from 5 to 70°. The XRD data were analyzed using EVA software. The TGA/DTA analysis of the clay was carried out using PerkinElmer thermal analyzer at a heating temperature range of 50–1100 °C, heating rate of 10 °C/minute and flow rate of 20 ml/min under a nitrogen atmosphere.

2.2. Sample Preparation and characterization

The flexural strength samples were prepared from the raw clay and the clay mixed with 0, 10, 15 and 20% starch (label as A, B, C and D respectively in Table 1). The samples were compacted into a die of dimensions approximately 5 × 30 × 80 mm at a compaction pressure of 60 with an Instron 600 KN machine at 10 min holding time to attain perfect consolidation. Thirty three samples each were fabricated for the dense, 10, 15 and 20%. The samples were fired in a high temperature furnace at 1300 °C for a period of two hours. The flexural strength of samples A, B, C and D fired at 1300 °C (33 samples each) was determined using three point bending test. The specimens' width and breadth were measured and recorded and a span of 40 mm was used for the test. A load was applied on the specimen until fracture. The test was conducted using an Instron 100-KN electromechanical testing machine at a loading rate of 0.5 mm/min, based on the following equation (ASTM, 1999):

$$\sigma = \frac{3FL}{bd^2} \quad (1)$$

where σ is the flexural strength variable, F is the load, L is the span (40 mm), b is the width and d is the section thickness of specimen.

Multi-point BET was conducted on the dense porous ceramics at 1300 °C to determine the adsorption/desorption isotherm, average pore size and the pore size distribution on TriStarII 3020 surface area and pore analyzer with N_2 as adsorptive at −196 °C for 4 h in a vacuum. The morphology of the dense ceramic was recorded using Field Emission Scanning Electron Microscopy FESEM (ZEISS SUPRA35VP). For the porous ceramics, the samples were fractured, cold mounted, ground with 320–1200 grit of silicon carbide papers, polished using 1 μm diamond paste and cleaned with ultrasonic cleaner. The morphology was recorded with FESEM.

2.3. Three-parameter Weibull analysis

The fracture strength data were analyzed using three and two-parameter Weibull probability distribution with MINITAB 15