Effects of sintering temperature on the filtration and mechanical properties of ceramic water filters

Oluwole A. Omoniyi1, Ali A. Salifu2, John D. Obayemi2, Oluwaseun K. Oyewole2, Pierre-Marie Nigay4, Omololu Akin-Ojo5,6 and Winston O. Soboyejo1,2,6,7

Abstract: This paper presents the results of a combined experimental and theoretical study of the effects of sintering temperature on the mechanical and filtration properties of clay ceramic water filters (CWFs). CWFs produced from 50:50 volume proportion mixtures of clay and sieved sawdust (porogen) were sintered at 850°C, 900°C and 950°C to produce micro- and nano-porous structures. The sintered clay structures were then characterized using X-ray diffraction and scanning electron microscopy. The flow and filtration characteristics of the filters were then elucidated using a combination of theory and fluid flow experiments, before exploring the effects of porosity on E. coli removal and flow rates through the filter. The E. coli removal efficiency is shown to be inversely proportional to the sintering temperature, with log removal values (LRV) of 4.89, 4.59 and 4.46 for the CWFs sintered at 850°C, 900°C and 950°C, respectively. The paper also examines the effects of sintering temperature on the compressive and flexural strengths, as well as the fracture toughness of the CWFs. The compressive strengths of the CWFs increased (between 6.55 and 7.02 MPa), whereas the flexural strengths (between 3.05 and 2.22 MPa) and the fracture toughness values (between 0.21 and 0.14 MPa m1/2) both decreased in response to the increased porosities induced by the increased sintering temperature. The underlying fracture and toughening mechanisms are then elucidated before discussing the implications of the results for the fabrication, transportation, and handling of robust ceramic water filters.

Subjects: Stress Analysis; Fracture & Damage Mechanics; Materials Processing; Water Science

Keywords: ceramic water filters; sintering temperature; E. coli removal; filter robustness; compressive/flexural strengths; fracture toughness; fracture; toughening mechanisms

1. Introduction

Waterborne diseases account for 3.6 million deaths every year (Brown et al., 2008; Tsao et al., 2016). In most cases, these deaths are due to microbial impurities (Brown, 2007) that can cause infectious diseases such as diarrhea and death (Van Halem, 2006; WHO/UNICEF, 2015; World Health Organisation (WHO), 2011). There is, therefore, a need for point-of-use water filtration systems that can remove microbial contaminants from water (Clasen et al., 2015; Hwang, 2003; Rayner et al., 2016, 2013). The need for potable water in rural/urban communities across the world has stimulated significant efforts to produce and distribute ceramic water filters (CWFs) to communities across the world (Potters for Peace (PFP), 2008, Soppe et al., 2015).
In most cases, the point-of-use CWFs are produced from locally available clays using well controlled mixtures of clay and saw dust (Potters for Peace (PFP), 2008). They are then transported, often through challenging road conditions, to rural/urban communities where they are used to filter contaminated water that often contains microbes like E. coli, which can cause water-borne diseases such as dysentery, diarrhea, meningitis, hepatitis A and E, cholera, and myocarditis (Mereder et al., 1998; Sullivan et al., 2017; WHO/UNICEF, 2017). There is a need to develop processing conditions that can reduce the likelihood of cracking during the transportation of CWFs.

Prior efforts to study the mechanical fracture properties of ceramic water filters have reported strength and fracture toughness values for filters that were produced under one sintering condition (Yakub et al., 2012; Annan et al., 2014; Nigay et al., 2017; Nigay et al. 2018, Nigay et al., 2019) using frustum shaped (flower pot) CWFs. However, they have not explored the effects of sintering temperature, which is a key variable in the control pore/defect size distributions and the mechanical properties of CWFs. There are different shapes of CWFs, such as candle, flowerpot, paraboloid, disc, and hemisphere shaped CWFs (Matthies et al., 2015). However, different stress states are induced on the clay matrix when pressed in various mold shapes. Thus, the mechanical properties of different shaped CWFs will vary, depending not only on sintering temperature but also on the underlying stress states associated with the formation of the filters. There is, therefore, a need for further studies of the effects of sintering temperature on the mechanical properties and the filtration characteristics of frustum shaped CWFs.

This paper presents the results of a combined experimental study of the effects of sintering temperature on the mechanical properties and the filtration characteristics of CWFs that are produced by the sintering of clay/sawdust mixtures with volume ratios of 50:50. The effects of sintering at 850°C, 900°C, and 950°C are explored in experimental and theoretical studies of the mechanical properties and filtration characteristics of the resulting micro-/nano-porous structures. This range of temperatures was used to avoid the thermal cracking of the CWFs. The underlying fracture and toughening mechanisms are elucidated before discussing the implications of the results for the fabrication, transportation, and use of CWFs for the purification of water in rural/urban communities.

2. Materials and methods

2.1. Materials and processing of ceramic water filters

Redart clay (Cedar Heights, Pittsburgh, PA) and sawdust containing 75% oak and 25% Spanish cedar obtained from JB Sawmill, Hopkinton, MA, were used for the processing of the ceramic water filters. To vary their porosity after sintering, three 50:50 (volume %) of clay to sawdust mixture was molded and fired at 850°C, 900°C and 950°C to yield the CWFs. The sawdust was sieved using a 500 micron or 35 mesh wire sieves. The clay and sieved sawdust were then mixed in an industrial mixer (Avonco-Mix 20, The Hobart Manufacturing Company, Troy, OH), with intermittent addition of deionized water, until the mixture rolled up into a big lump (Iyasara et al., 2016). The mixture was then manually shaped before inserting it between the male and female molds in a hydraulic press. Between 1.8 and 2.0 L of water was used in the blending.

Prior to the hydraulic pressing, the male and female molds were covered with plastic bags that were coated with non-stick cooking spray. The blended mixture was placed between the female mold and the male mold. A pressure of 140 kPa (20 psi) was then applied to the male mold. This was used to press out the filter using a 50-tonne hydraulic press (TM Torin Big Red Jacks, Inc, Ontario, Canada). After pressing, the filters were labeled for identification and air-dried for 6 days in the laboratory at room temperature (25°C) and 40% relative humidity.

The air-dried CWFs were fired in a gas kiln (Paragon Professional Series Dragon Kiln with Sentry 2.0 Microprocessor, Paragon Industries, Mesquite, Texas, USA). They were then pre-heated to 450–550°C for three hours (50°C/h) to burn off the combustible (sawdust), prior to subsequent heating.
at a rate of 100°C/h to sintering temperatures of 850°C, 900°C, and 950°C for 5 hours. The filters were then furnace-cooled in the air to room temperature (25°C). The dimensions of the resulting frustum-shaped CWFs obtained (Figure 1) were: Top diameter = 30 cm, height = 25 cm, and base diameter = 22 cm with a capacity of 10 L. A total of 9 CWFs were used for this study; 3 filters per sintering temperature (n = 3), unless otherwise stated.

The sintering temperature is very important in this study, hence the need to ensure that the stated temperature is maintained for the specified CWFs. This was achieved by using a thermocouple inside the kiln to detect the temperature for the controller to maintain the temperatures within a range of ±20 F. In addition, a digital pyrometer was used to verify the temperatures generated by the internal thermocouples. Also, pyrometric cones were used to measure the heat work, and to ensure that consistent temperatures were maintained during the firing.

2.2. Material characterization
Scanning Electron Microscopy (SEM) (Model XL 30, Philips, Amsterdam, the Netherlands) and Energy Dispersive X-Ray spectroscopy (EDX) were used to study the microstructure and local variations in chemistry of the porous structures that were produced. This was done using an Oxford EDS system (Oxford Instruments, Oxford, UK) in an Environmental Scanning Electron Microscope (ESEM) (Quanta 200 Field Emission FE-ESEM, FEI, Hillsboro, OR, USA).

2.3. Determination of porosity and permeability of the CWFs
The open porosities of the filters were determined via a vacuum immersion method. Samples with dimensions of 3 mm x 3 mm x 3 mm were cut from four different locations on each of the three CWFs, i.e., the base and lower, middle, upper part of the side of filters. The samples’ weights are taken when dry in air as initial weight, \( W_1 \), before submerging the sample in distilled water within a beaker. The beaker was then placed in a vacuum of 29 inches (i.e., a pressure of 25 mm) for 3 hours at 26°C. The soaked samples were weighed under distilled water as \( W_2 \), thereafter, the samples were removed, and their surfaces were wiped with tissue or cloth and weighed out of distilled water as \( W_3 \).

The weights \( W_3 \) and \( W_2 \) of the saturated CWFs are assumed to be measured when pores in them have been filled with water. \( W_3 \) is expected to be lesser than \( W_2 \) because of the upthrust on the weight inside water.

The open porosity \( P_{open} \) was, thus, expressed as:

\[
P_{open} = \frac{W_2 - W_1}{W_2 - W_3}
\]  

The permeability \( \mu \) (m²) of the CWFs was also determined under laminar flow in a fluid (nitrogen). This was evaluated using the Darcy and Hagen Poiseuille laws, which are expressed as:
\[ \mu = \frac{n \delta}{A} \times \frac{q}{p_1 - p_0} \times \frac{2p_1}{p_1 + p_0} \]  

(2)

where \( \mu \) is the dynamic viscosity of nitrogen at ambient temperature (25°C), \( \delta \) is the thickness of CWF piece, \( A \) is the cross section of the sample, \( q \) is the fluid volume moved through the sample, and \( p_1 \) and \( p_0 \) are the respective constant pressures at the inlet and outlet of the sample. (D. Lantagne, 2002). Prior to this, the three CWFs were saturated by total immersion in a tub containing distilled water (model D8611, Barnstead/Thermolyne, Hampton, NH, USA). The soaking was maintained for 12 hours.

2.4. Determination of flow rates

The water flow rates (through the porous membranes of the filters) were determined for the three types of CWFs that were fired at 850°C, 900°C, and 950°C. A reference Potters for Peace filter (composed of 60:40 clay: sawdust and colloidal silver overlay) also fired at 850°C, 900°C and 950°C was subjected to flow rate determination to serve as a comparison. In each case, the volume of water discharged from the CWFs per time was used to estimate the flow rate (Annan et al., 2014; Farrotul et al., 2018; D. Lantagne, 2002).

The pre-soaked CWFs were filled with 10 L of deionized water. The flow of water through the porous ceramic membranes was then measured by collecting the discharged water in a receptacle that was mounted on a PGL 20001 weighing balance (Adam Equipment, Oxford, CT, USA) that was instrumented with the Adam DU Software Package (Adam Equipment, Oxford, CT, USA), as shown in Figure 2, that was used to monitor the weight changes due to the collection of filtered water. The volume of water discharged was obtained by dividing the weight of the water by the product of the acceleration due to gravity and the density of water (1 g cm\(^{-3}\)). The flow rates (in the first hour), the mean flow rates and estimated values of permeability were subjected to statistical analysis.

2.5. Statistical experimental study

The flow experiments were carried out 20 times to ascertain the variation in the flow parameters. These were fitted using the modified Darcy equation for frustum-shaped CWFs. Through this method, values were obtained for permeabilities and standard deviations, together with the established statistical distributions which may characterize the flow rates obtained.

2.6. Bacterial removal efficiency

The bacteria removal efficiencies of the CWFs sintered at different temperatures of (850°C, 900°C, and 950°C) were determined using Escherichia coli (E. coli) bacteria (C-300 Strain, ATCC 15597). The E. coli was cultured in nutrient broth at 37°C for 24 hours in a shaker incubator (Model G25, New Brunswick Scientific Edison, NJ) at 100 rpm to reach the stationary growth phase. Subsequently, 4 mL of the stationary phase culture was mixed with 4 L of sterile deionized water to produce pre-filtrate suspensions containing approximately 10\(^5\) to 10\(^7\) colony forming units (cfu/mL; Bielefeldt et al., 2009; Brown & Sobsey, 2010; Venis & Basu, 2020). All the 4 L of pre-filtrate was poured quickly into the CWF. The filtrate was then collected into a covered plastic bucket (Figure 2).

The numbers of viable cells (concentration of E. coli) in the pre-filtrate (\(C_{pi}\)) and filtrate (\(C_{f}\)) suspensions were determined by serially diluting the suspensions and plating 1 ml aliquots onto E. coli count plates (Petrifilm, 3 M, St. Paul, MN). The plates were then incubated at 37°C for 18–24 hours, before counting the number of colonies on the plates. The concentrations of the E. coli in the suspensions were then determined as the number of colony forming units per ml (cfu/mL). These were used to calculate the percentage removal and log reduction values (LRVs) (yield filtration efficiency) from Equations 1 and 2:
\[
\% \text{ Removal} = \frac{C_{pf} - C_f}{C_{pf}} \times 100
\]  

(3)

\[
LRV = -\log_{10} \frac{C_{pf}}{C_f}
\]

(4)

where \(C_{pf}\) is the concentration of \(E. \ coli\) in the pre-filtrate suspension (cfu/mL), and \(C_f\) is the concentration of \(E. \ coli\) in the filtrate suspension (cfu/mL).

### 2.7. Mechanical testing

The mechanical properties of the CWFs were determined under compression and three-point bending using an electromechanical testing machine (Instron Model 3366, Instron, Canton, MA) equipped with a 500 N load cell. The specimens were prepared from the three sections of the CWF (top, middle, and bottom) using a wet tile saw. They were rectangular in shape, with straight edges to give uniformity during testing, 15 specimens were tested. The compressive strengths of the CWFs were estimated by compressive loading of rectangular specimens with length, \(H\), of 7.0 cm, breadth, \(B\), of 2.5 cm, and thickness, \(H\), of 2.5 cm. The samples were loaded monotonically to failure at a rate of 0.1 N/s. The three-point bending tests were carried out on specimens with dimensions of 17.5 cm x 2.5 cm x 1 cm at a constant displacement rate of 1 mm/min until the onset of fracture with a loading span of 50 mm.

Fracture toughness was studied using Single Edge Notch Bend (SENB) specimens. SENB specimens with dimensions of 17.5 cm x 2.5 cm x 1 cm and notch depths of 2 mm (0.40–0.45 notch to width ratio) were tested using the Instron 3366 electromechanical testing machine. The specimens
were loaded at a displacement rate of 0.5 mm/min until a pop-in was attained at the notch (Nigay et al., 2017; Yakub et al., 2012). The fracture surfaces of the SENB specimens were then analyzed using SEM.

2.8. Modeling of water flow through the ceramic water filters

A hydrodynamic model (Figure 1) was used to model the flow of water through the filters (Yakub et al., 2012). Flow through the frustum shaped filter was modeled using Darcy’s law. This gives:

\[ Q = \frac{kA\Delta p}{\gamma L} \]  

(5)

where \( k \) is the permeability of the materials, \( A \) is the surface area, \( L \) is the thickness of the material, \( \gamma \) is the dynamic viscosity of the water and \( \Delta p \) is the difference between the top and bottom of the surface (Soni et al., 2008). The corners in this regard, are negligible, the flow rate across the bottom is \( Q_b \), and the flow rate across the sides is \( Q_s \).

The pressure applied to the side surfaces is equal to the hydrostatic pressure of water. The pressure that is applied to the filter membrane is given by:

\[ \Delta p = \rho gh(t) \]  

(6)

Substituting Equation 4 into Equation 3, and noting that the area of the bottom of the filter is given by \( A = \pi r_o^2 \), the thickness of the porous material \( L = t_o \), while the permeability is constant, the flow rate through the bottom of the filter \( Q_b \) is then given by:

\[ Q_b = \frac{k \pi r_o^2 \rho gh(t)}{\mu t_o} \]  

(7)

On the sides however, the pressure depends on position \( y \), hence expressed as \( \Delta p = \rho g(h(t) - y) \). The area of the filter equally is a function of \( y \). The radius alters along the filter height and is given by

The permeability coefficient is taken to be constant and equal to that on the bottom of the filter. Therefore, the flow rate across the side is expressed as:

\[ Q_s = \int_0^{h(t)} \frac{k \pi \rho g h(t) - y}{\mu} \left[ \frac{2}{3} \tan \theta \right] dy \]  

(8)

Integrating equation 6 gives:

\[ Q_s = \frac{k \pi \rho g 2h^2(t)}{\mu} \left[ \frac{r_o}{2} \cdot \frac{h(t)}{3} \tan \theta \right] \]  

(9)

Adding equations 6 and 8, i.e., bottom and side flows, the total flow rate \( Q \) for the CWF is given by:

\[ Q = \frac{k \pi \rho g h(t)}{\mu} \left[ \frac{r_o^2}{t_o} + \frac{r_o h(t)}{t_o} - \frac{2h^2(t)\tan \theta}{3t_o} \right] \]  

(10)

The values of \( h(t) \) is obtained from the expression for volume of water \( V(t) \) present in the frustum shaped filter. The time-dependent volume is given by:
\[ V(t) = \pi \left[ R^2 h(t) + R h(t^2) \tan \theta + \frac{h(t) \tan^2 \theta}{2} \right] \]  

(11)

The values of \( h(t) \) were obtained by fitting the experimental data to (10), using flow rate data obtained from this study (Plappally et al., 2009; Schweitzer et al., 2013; Yakub et al., 2012).

3. Results and discussion

3.1. Structure of the porous ceramic water filters

The SEM images of the sintered clays (Figure 3) revealed that residual pores are distributed within the sintered samples in which the saw dust particles have been broken down during the thermal decomposition of organic matter, leaving behind a porous ceramic structure (CMWG, 2011; Mikelonis et al., 2020; Oyanadel-Craver & Smith, 2008). No significant differences were observed in the SEM images obtained after sintering at different temperatures.

The surface morphologies of the combustible “saw dust” and that of the clay ceramics were imaged with the SEM machine. This was done using samples that were embedded in epoxy resin, polished, and viewed under back-scattered imaging conditions (Plappally et al., 2009; Sobsey et al., 2008).

Figure 3. SEM micrographs of the ceramic water filters showing the pores in the sintered clay-sawdust microstructures.
The phases present in the ceramic powders and the sintered clays are presented in Table 1. The Redart clay powder contains kaolinite, silica and illite, while the fired clays contained mostly aluminum and potassium silicate hydroxide.

3.2. Porosity
The porosimetry (vacuum immersion method, using Archimedes immersion technique) revealed the pore size distributions associated with the CWFs that were produced by firing at 850°C, 900°C and 950°C (Tables 2 and 3). From Tables 2 and 3, it was observed that the CWFs sintered at 850°C had mean and median pore diameters of 0.73 ± 0.03 and 0.86 ± 0.04 µm, respectively. CWFs sintered at 900°C also had mean and median pore diameters of 0.84 ± 0.04 and 0.98 ± 0.03 µm, respectively, while CWFs sintered at 950°C had mean and median pore diameters of 1.07 ± 0.05 and 3.16 ± 0.19 µm, respectively. In general, the porosity increased with increasing sintering temperature, as shown in Table 4 and Figure 4.

The highest mean and median pore sizes were obtained from CWFs that were sintered at 950°C, while the lowest mean and median pore sizes were found with CWF sintered at 850°C. Conversely, the experiments revealed that the CWFs sintered at 850°C had the lowest mean and median pore sizes, and the best capacity for bacteria removal (Figure 3). Thus, the higher the porosity, the lower the capacity for bacterial removal. This suggests that the porosity is inversely related to the capacity for bacteria removal (Table 5). Similarly, in the case of CWFs sintered at 900°C, medium sizes of mean and median pore sizes had a balance of slower flow rates/porosity and bacteria removal efficiency (Schijven et al., 2003).

| Table 1. Elemental composition of the Redart clay obtained by X-ray Fluorescence (XRF). Analysis |
| Composition | Analysis (wt. %) |
| MgO | 1.61 |
| CaO | 0.25 |
| Al₂O₃ | 15.53 |
| SiO₂ | 64.93 |
| K₂O | 4.16 |
| Fe₂O₃ | 7.07 |
| TiO₂ | 1.10 |
| Na₂O | 0.36 |
| P₂O₅ | 0.21 |
| Loss on Ignition (LOI) | 4.74 |
| Total | 96.6 |

| Table 2. Average pore diameters of the ceramic water filters sintered at 850°C, 900°C and 950°C |
| Sintering Temperature °C | Average Pore Diameter (µm) | Average |
| | Base | Lower | Middle | Upper | |
| 850 | 0.65 ± 0.03 | 0.78 ± 0.04 | 0.75 ± 0.03 | 0.74 ± 0.03 | 0.73 ± 0.03 |
| 900 | 0.82 ± 0.04 | 0.91 ± 0.04 | 0.85 ± 0.04 | 0.79 ± 0.03 | 0.84 ± 0.04 |
| 950 | 1.04 ± 0.04 | 1.11 ± 0.05 | 1.09 ± 0.05 | 1.02 ± 0.04 | 1.07 ± 0.05 |
Table 3. Median pore diameters of the ceramic water filters sintered at 850°C, 900°C and 950°C

| Sintering Temperature °C | Median Pore Diameter (μm) | Average |
|--------------------------|---------------------------|---------|
|                          | Base          | Lower     | Middle    | Upper     |
| 850                      | 0.86 ± 0.03   | 0.78 ± 0.04 | 0.94 ± 0.04 | 0.86 ± 0.04 |
| 900                      | 1.00 ± 0.04   | 0.98 ± 0.02 | 1.03 ± 0.04 | 0.92 ± 0.03  |
| 950                      | 3.10 ± 0.18   | 3.01 ± 0.21 | 3.24 ± 0.18 | 3.28 ± 0.19  |

Table 4. Percentage porosities of the ceramic water filters sintered at 850°C, 900°C and 950°C

| Sintering temperature °C | Porosity (%) | Average Porosity (%) |
|--------------------------|--------------|----------------------|
|                          | Base          | Lower     | Middle    | Upper     |
| 850                      | 35.30         | 37.91     | 35.16     | 33.15     | 35.38 ± 1.95 |
| 900                      | 38.75         | 38.15     | 42.65     | 40.58     | 40.03 ± 2.03 |
| 950                      | 45.80         | 43.90     | 44.40     | 41.50     | 43.90 ± 1.79 |

Finally, it is important to compare the measured pore sizes to the diameters of bacteria and non-viral pathogens, which range from 1–3 μm. Thus, the measured pore diameters were less than typical sizes of bacteria that are generally of the order of about 1–10 μm (Haiyan & Shangping, 2020; D. Lantagne, 2002; Mwabi et al., 2012).

3.3. Fluid flow through the filters

The flow rates through the CWFs sintered at the three temperatures (850, 900 and 950°C) are presented in Figure 7. The initial flow rates for the first hour were between 1.2 and 2.0 L per hour, for their first hour of flow. These are consistent with prior results (1–2 L per hour) obtained for Potters for Peace (PFP) Filters in prior work (Yakub et al., 2012). These flow rates also increased with increasing sintering temperature (Figure 5a), while the bacteria removal (LRV) over 24 hours decreased with increasing sintering temperature (Figure 5b). The flow rates started to decline after 20 hours of filtration (Figure 6). This is attributed to the reduction in the flow due to the lower pressure heads associated with the reduced water levels at different stages of the fluid flow through the filters.

The mean flow rate of the discharged water for the six filters was 1.7 ± 0.2 L/h during the first hour of the discharge. The effective permeability of each CWF was equally obtained by fitting the measured data for the flow rate into the modified Darcy equation for frustum-shaped CWFs (Figure 7).

Their standard deviations were also taken as well as the statistical distributions that may characterize the measured flow rates. The permeability values for the filters ranged between 0.41 × 10⁻¹⁴ m² and 2.52 × 10⁻¹⁴ m², with an average value of 1.16 × 10⁻¹⁴ m² with hydraulic conductivity k value of 1.41 × 10⁻⁷ m/s. This average effective permeability value is comparable to the permeability values obtained for CWFs in Cambodia (1.37 × 10⁻⁷ m²) and Ghana (1.3 × 10⁻⁷ m²; Van Halem, 2006) and Nigeria (1.19 × 10⁻¹⁴ m²; Annan et al. 2016). The average effective permeability estimated fell within the range of the values of hydraulic conductivity of 1.15 × 10⁻⁷ m/s to 5.01 × 10⁻⁷ m/s obtained by Oyanadel-Craver and Smith (2008) for ceramic water filters. Darcy fits to the flow data are also presented in Figure 8. This shows very good fits between the measured data and the fitted flow rates, suggesting that the Darcy fits provide estimates of the average permeabilities that best characterize the flow through the filters.
This work also showed that there is a linear relationship between flow rate and permeability which suggests that the effective permeability approach follows the trends in the flow rate data.

The variations in the flow rates were typified by normal distributions that fitted into the experimental data obtained (Figure 9).

The distribution of flow rate (in the first hour and mean) and permeabilities (in the first hour and mean) attained for the 20 runs are approx. normally distributed and this was confirmed using Kolmogorov-Smirnov Normality test.

### 3.4. Bacteria removal efficiency

The percentage of E. coli removal and the log removal value LRV of E. coli trapped by each CWF are presented for both pre-filtered (stock) and the post-filtered (filtrate) in Table 5. The CWFs had log removal values (LRV) of 4.46, 4.59, and 4.89, respectively, for the filters fired at 950°C, 900°C and 850°C, respectively.

![Figure 4. Variations in the porosities of the ceramic water filters at different sintering temperatures, highlighting the effects of sintering temperature on (a) the porosity of the base of the CWFs and (b) the porosity of the sides (lower, middle and upper) of the CWFs. (c) Particle size distributions of the sawdust and Redart clay used for making the ceramic water filters.](image)

| Sintering Temperature °C | Percentage Removal of E. coli | Log Reduction Value of E. coli (LRV) |
|--------------------------|-------------------------------|-------------------------------------|
| 850                      | 99.995                        | 4.89 ± 0.18                         |
| 900                      | 99.996                        | 4.59 ± 0.17                         |
| 950                      | 99.998                        | 4.46 ± 0.15                         |
Figure 5. The effects of sintering temperature on the: (a) flow rates of the ceramic water filters, and (b) log reduction values (LRV) of the removal of E. coli from contaminated water by the CWFs.

Figure 6. The water flow rates through the ceramic water filters sintered at different temperatures: (a) flow rates as a function of time; (b) flow rates as a function of porosity.

850°C (Figure 5). The corresponding bacteria removal efficiencies were 99.995%, 99.996% and 99.998%, respectively. Thus, the effectiveness of the CWFs in bacteria removal increases with reducing sintering temperature. Hence, the CWFs sintered at 850°C had the highest bacteria removal efficiency. Similar trends were also observed after multiple filtration cycles.

3.5. Compressive and flexural properties

The flexural strengths, $\sigma$, were obtained from the following expressions:

$$\sigma = \frac{3F_{\text{max}}L}{2BH^2}$$  \hspace{1cm} (12)

where $F_{\text{max}}$ is the applied force; $L$ is the loading span; $B$ is the specimen breadth, and $H$ is the sample height (ASTM International, 2017), while the compressive strengths of the CWFs were estimated from:
Figure 7. Flow rates of water through the ceramic water filters: (a)-(f) Experimental and analytical flow rate plots for various CWFs (a),(b) sintered at 850°C; (c),(d) sintered at 900°C, and (e) (f) sintered at 950°C.
\[ \sigma_C = \frac{P}{BW} \]  

(13)

where \( P \) is the failure load; \( B \) is the specimen breadth, and \( W \) is the specimen width, for specimens with rectangular cross-sections.

The measured compressive and flexural strengths are presented in Figures 10a,b and 10c,d, respectively, and summarized in Table 6. These show that compressive strengths increased with increasing sintering temperature from 850 to 950°C (Figures 10a and 10b). On the other hand, the flexural strengths decreased with increasing sintering temperature (Figures 10c and 10d), attributed to reductions in defect sizes with increasing sintering temperature. The compressive and flexural strengths also increased and decreased, respectively, almost linearly with increasing porosity. Furthermore, filters with porosities around 35% had higher compressive strengths and lower flexural strengths. Filters with increased porosities ~ 44% had the highest compressive strengths and the least flexural strengths. This is consistent with similar trends have been reported by Yakub et al. (2012) and Wagh et al. (1991).
Figure 9. Statistical distributions of the (a) percentage porosity, (b) flow rates, (c) log reduction values (LRV) of E. coli removal, (d) compressive strength, (e) flexural strength, and (f) fracture toughness of the ceramic water filters.
Figure 10. The mechanical properties of the ceramic water filters showing the variation of: (a) the compressive strength with porosity; (b) the compressive strength with sintering temperature; (c) the flexural strength with porosity; (d) the flexural strength with sintering temperature; (e) the fracture toughness with porosity, and (f) the fracture toughness with sintering temperature.

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3.6. Fracture toughness and toughening mechanisms

The fracture toughness of the three CWFs (sintered at 850, 900 and 950°C) were found to decrease with increasing porosity due to the increasing sintering temperatures, as shown in Table 6 and Figures 10e and 10f.

This is consistent with the results presented in earlier work by (Yakub et al., 2012), who reported fracture toughness data for CWF clay structures with different porosity levels that were controlled by varying the volume fractions of sawdust that were used in the processing of the filters. The fracture toughness, $K_{ic}$, values were estimated from (ASTM International, 2018).

$$K_{ic} = f \left( \frac{a}{W} \right) x_f x \sqrt{\pi a}$$

(14)

where $a$ is crack length, $f \left( \frac{a}{W} \right)$ is the compliance function, and $\sigma_f$ is the stress at the peak load.

Evidence of the underlying toughening mechanisms was obtained from ESEM micrographs of the side profiles of the fracture toughness specimens, as shown in Figure 11a,d.

From these SEM images, the microstructural details of crack bridges (bridge length, width, depth and separation) were measured using image J and summarized in Table 8a,c for the different CWFs.

The SEM images show evidence of toughening of crack bridging by uncracked ligaments and crack trapping. Thus, the resistance-curve behavior can be estimated from the sum of initiation fracture toughness, $K_i$, and the toughening due to the observed crack tip shielding mechanisms (Figures 12a,d).

Since the crack tip shielding mechanisms included small/large scale bridging ($K_b$) and the crack trapping ($K_t$), the points along the resistance-curve can be estimated from the following equation:

$$K_R = K_i + \Delta K_b + \Delta K_t$$

(15)

Table 6. A summary of the mechanical properties of the ceramic water filters at different sintering temperatures. Data are presented as mean ± standard deviation

| Sintering Temperature °C | Pore Diameter (µm) | Porosity (%) | Compressive Strength (MPa) | Flexural Strength (MPa) | Fracture Toughness (MPa √m) |
|--------------------------|--------------------|--------------|-----------------------------|-------------------------|-----------------------------|
| 850                      | 0.73 ± 0.03        | 35.38 ± 1.95 | 6.55 ± 0.30                 | 3.05 ± 0.31             | 0.21 ± 0.02                 |
| 900                      | 0.84 ± 0.03        | 40.03 ± 2.03 | 6.71 ± 0.36                 | 2.70 ± 0.28             | 0.17 ± 0.01                 |
| 950                      | 1.07 ± 0.04        | 43.90 ± 1.79 | 7.02 ± 0.32                 | 2.22 ± 0.26             | 0.14 ± 0.03                 |

Table 7. A summary of Fett and Munz (1994) parameters for Single Edge Notched Bend Specimen subjected to weighted crack bridging fractions

| µ  | 0     | 1    | 2    | 3    | 4    |
|----|-------|------|------|------|------|
| 0  | 0.4980 | 2.4463 | 0.0700 | 1.3187 | −3.067 |
| 1  | 0.5416 | −5.0806 | 24.3447 | −32.7208 | 18.1214 |
| 2  | −0.19277 | 2.55863 | −12.6415 | 19.7630 | 10.986 |
Figure 11. The fracture and toughening mechanisms of the ceramic water filters. Environmental SEM micrographs of the porous ceramic water filters showing evidence of (a),(b) crack trapping and (c), (d) crack bridging. (e) displays the resistance-curve behavior of the clay-ceramic and clay-ceramic with 50 wt.% sawdust at different sintering temperatures.
Table 8: Microstructural details of crack bridges for specimens sintered at (a) 850°C, (b) 900°C, and (c) 950°C

| (a) 850°C | Cold shock cycles (N) | Bridge length p (µm) | Bridge width w (µm) | Bridge depth h (µm) | Bridge separation @ (µm) |
|-----------|-----------------------|----------------------|---------------------|---------------------|--------------------------|
| 5         | 52.20                 | 0.76                 | 0.56                | 0.52                | 0.27                     |
| 10        | 35.21                 | 0.66                 | 0.56                | 0.52                | 0.27                     |
| 15        | 28.35                 | 0.56                 | 0.52                | 0.27                |                          |
| 20        | 20.02                 | 0.52                 | 0.27                |                     |                          |

| (b) 900°C | Cold shock cycles (N) | Bridge length p (µm) | Bridge width w (µm) | Bridge depth h (µm) | Bridge separation @ (µm) |
|-----------|-----------------------|----------------------|---------------------|---------------------|--------------------------|
| 5         | 0.72                  | 0.92                 | 1.16                | 1.60                |                          |
| 10        | 0.57                  | 0.45                 | 0.41                | 0.17                |                          |
| 15        | 0.33                  | 0.28                 | 0.09                |                     |                          |
| 20        | 0.28                  |                      |                     |                     |                          |

| (c) 950°C | Cold shock cycles (N) | Bridge length p (µm) | Bridge width w (µm) | Bridge depth h (µm) | Bridge separation @ (µm) |
|-----------|-----------------------|----------------------|---------------------|---------------------|--------------------------|
| 5         | 0.57                  | 0.85                 | 1.06                | 1.60                |                          |
| 10        | 0.32                  | 0.45                 | 0.33                | 0.17                |                          |
| 15        | 0.21                  | 0.09                 |                     |                     |                          |
| 20        | 0.07                  |                      |                     |                     |                          |

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where $K_a$ is any value of $K$ beyond the initiation toughness, $\Delta K_a$ is the toughening due to crack bridging, and $\Delta K_f$ is the toughening due to crack trapping. The toughening due to crack bridging was predicted by a small-scale bridging approach, for crack extensions smaller than 0.5 mm and via a large scale approach (Nigay et al., 2017; Yakub et al., 2012) using material parameters that are summarized in Table 7. The results are presented in Figure 8e. These show that the shielding models predict the trends in the measured resistance-curve behavior for the clay ceramic membranes that were sintered at temperatures of 850°C, 900°C and 950°C.

### 3.7. Implications

First, the results show that mechanical properties (strengths and fracture toughness) of the porous clay CWFs depend strongly on porosity and the underlying toughening mechanisms associated with interactions of cracks with microstructural features. These are affected by sintering temperatures between 850°C and 950°C. Sintering at higher temperatures also gives rise to higher porosity and lower fracture toughness/flexural strengths, as well as higher compressive strengths. Thus, intermediate temperatures (between 850°C and 900°C) are needed for the sintering of CWFs with a balance of fluid flow and filtration characteristics that are needed to engineer filters that can remove bacteria effectively during fluid flow at reasonable rates. Furthermore, the sintering of filters at temperatures between 850°C and 900°C is most likely to result in filters with the best balance of compressive/flexural strengths and fracture toughness/resistance-curve behaviour due to crack bridging and crack trapping mechanisms.

Moreover, the filtration properties depend strongly on the sintering temperatures between 850°C and 950°C. The mechanical properties of porous CWFs also depend on sintering temperature/porosity, with the flexural strengths/fracture toughness values decreasing with increasing sintering temperature, while the compressive strengths increase with increasing temperature (Figures 9, 10 and 11). The trends in the fracture toughness values are associated with crack/microstructure interactions that give rise to crack-tip shielding by crack bridging and crack-trapping. Hence, more robust filters can be produced by sintering at higher temperatures between 850°C and 950°C.

However, sintering at higher temperatures (900°C—950°C) also increases the flow rates of water through the filters. This is associated with a significant increase in the porosity, with small changes in the log removal of *E. coli* or the filtration efficiency. This suggests that the best balance of filtration characteristics and mechanical robustness can be achieved by the use of filters that are sintered at temperatures in the range between 850°C—900°C where the resulting filters have reduced porosity, lower flow rates, attractive filtration characteristics, and attractive combinations of compressive/flexural strengths and fracture toughness/resistance-curve behavior.

The study also shows that fluid flow through CWFs is characterized by normal distributions at the first hour of flow. Effective permeability for 20 consecutive flows through the filter are characterized by normal distributions (Figure 7). It was also found that there is a correlation between the flow rate and permeability in the first hour. This can be used in field conditions for the testing of filter quality.

### 4. Conclusions

1. The porosity of CWFs increases with increasing sintering temperature between 850° and 950°C. Sintering in this temperature range also results in bigger pore sizes and increasing porosity. Also, the porosity and pore sizes increase with increasing sintering temperatures from 900° and 950°C. The average flow rates attained for the tested CWFs were found to increase as the permeability values increase. At the 0.05 level of significance, all data obtained for flow rate (in the first four) and their permeabilities were normally distributed.

2. The average pore sizes (1–3 µm) in the sintered CWFs are in a range that is comparable to the typical sizes of *E. coli*. Thus, the sintering of CWFs at temperatures between 850° and 950°C results in similar geometrical occlusion of *E. coli* from biologically contaminated water. Thus, the *E. coli* removal is similar in the CWFs that were sintered at 850°C, 900°C and 950°C.
(3) The compressive strengths of the CWFs increase with increasing sintering temperature. However, both the flexural strengths and the fracture toughness values of the CWFs decrease with increasing sintering temperature between 850°C and 950°C. The trends in the fracture toughness are associated with the effects of toughening by crack trapping and crack bridging. The measured fracture toughness and resistance curves are also well predicted by toughening by crack bridging and crack trapping.

(4) The sintering of robust ceramic water filters requires balanced considerations of the effects of sintering on filtration characteristics and mechanical properties. In general, the filtration characteristics (high bacteria removal and low fluid flow rates) can be optimized by sintering between 850°C and 900°C. However, although the compressive strengths increase with increasing sintering temperature, the flexural strengths and fracture toughness of the CWFs decrease with increasing sintering temperature between 850°C and 950°C.

(5) Toughening of the clay CWFs occurs largely by crack trapping and crack bridging mechanisms. These result in overall toughening levels that predict the trends in the measured resistance-curves of the CWF membranes.

Thus, the best balance of mechanical properties and filtration characteristics can be achieved by sintering of clays at temperatures between 850°C and 900°C. Within this range, the resulting fired clay ceramics have attractive combinations of fluid flow characteristics, bacteria removal, and compressive/flexural strength, and fracture toughness/resistance-curve behavior.

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Author details
Oluwole A. Omoniyi
Ali A. Salifu
John D. Obayemi
Oluwaseun K. Oyewole
Pierre-Marie Nigay
Omololu Akin-Ojo
Winston O. Soboyejo
E-mail: wsoboyejo@wpi.edu
ORCID ID: https://orcid.org/0000-0002-0209-1079

1 Department of Materials Science and Engineering, African University of Science and Technology (AUST), Abuja, Nigeria.
2 Materials Science and Engineering Program, Department of Mechanical Engineering, Worcester Polytechnic Institute (WPI), Worcester, MA, USA.
3 Department of Physics, University of Ibadan, Ibadan, Nigeria.
4 East African Institute of Fundamental Research (EAIFR), University of Rwanda, Kigali, Rwanda.
5 Department of Theoretical and Applied Physics, African University of Science and Technology (AUST), Abuja, Nigeria.
6 Department of Biomedical Engineering, Worcester Polytechnic Institute (WPI), Worcester, MA 01609, USA.

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