Manufacture of a low-cost ceramic microporous filter for the elimination of microorganisms causing common diseases
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ABSTRACT
Africa is one of the most water-scarce continents on earth and the lack of potable water is responsible for the death of approximately 4,900 children every day. An effective way of making sure that water is of good quality is by decontaminating it by means of a household ceramic water filter. The low-cost water filters suitable for the removal of suspended solids, pathogenic bacteria and other toxins from drinking water were developed using the traditional slip casting process. The locally produced filter has the advantage of low cost due to the usage of locally available raw materials, labour and expertise. Furthermore, the project provides opportunities for local financing and innovation. The product was tested using water contaminated with high concentrations of selected bacterial cultures as well as with water from local polluted streams. The ceramic filter was found to be highly effective in removing the bacteria and suspended solids from the contaminated water. With correct cleaning and basic maintenance, this filter can effectively provide clean drinking water for rural families affected by polluted surface water sources. This could provide a low-cost solution for the more than 250 million people without access to clean drinking water in Africa.

Key words | bacteria, ceramic filter, low cost, micro-porous, slip casting, water filtration

INTRODUCTION
Water purification methods applied in high income countries are neither cost effective nor technically adaptable to low-income countries (Clopeck et al. 2006). In developing countries, household-scale ceramic filters are being used as a better treatment option for both unpurified and insufficiently disinfected water at household level (Lantagne 2001; Clasen & Boisson 2006). Their use is becoming widespread, especially with involvement of governments and international non-governmental organisation (NGO) efforts such as Potters for Peace with 35 independent factories in 18 countries (Potters for Peace 2012).

Three common forming processes can be used for the shaping of porous ceramics: pressing, extrusion and slip casting. Each fabrication method results in a specific range of pore sizes, pore size distribution, and porosity, as well as varying levels of interconnectedness among the pores (Dobrovolskiy 1977). The amount, size and shape of defects in the ceramic are also dependent on the processing method used. Finally, each fabrication method differs in terms of overall investment cost and cost per product and quality requirement. Oyanedel-Craver & Smith (2008) and Fahlin (2003) indicate that significant potential variability in pore structure is possible in local ceramic filters tested by them, with the bulk of pore sizes in test samples being 20 μm. The pore size distribution in locally produced ceramic filters is therefore also likely to vary widely because of the various methods and materials used in the local manufacturing of ceramic water filters (CWFs).

This paper describes a method of manufacturing porous ceramic using the traditional slip casting process originating from developed countries. A number of important slip casting criteria were examined before selecting the best raw material:
(a) material should require limited further processing
(b) rheological property should include the ability to form a stable suspension with minimum segregation
(c) it should enable quick release from the mould without damaging the shaped body
(d) pore former has to enhance or match the slip rheological properties for inclusion into the mixture
(e) pore former should be able to release volatiles and fuse together with the raw material during sintering stage to give a strong product with a maximum degree of porosity (Chi 2004; Almeida 2009)
(f) body should not be temperature sensitive (minimum thermal expansion and thermal shock) over the firing range (Mandal 2007).

Slip casting requires only plaster moulds for the shaping process and the process is therefore more cost effective, especially for small-scale operations.

Each ceramic filter type was tested using sterile distilled water inoculated with three different bacterial cultures as well as three natural stream water sources (tested for indicator organisms – coliform bacteria) to verify the microbiological quality of the water produced from the micro-porous ceramic filters. The major limiting factor of the microbiological testing was however its short duration when compared with other studies (Van Halem et al. (2008): 1 to 2 years and Lantagne et al. (2010): 6 to 8 weeks).

Table 1 shows a summary of worldwide testing done on low cost ceramic water filters (developing countries) for the period 2000 to 2010. The average benchmark standard for *Escherichia coli* effectiveness was 97 ± 3%, whereas virus testing effectiveness was limited (Simonis & Basson 2011). Table 1 illustrates the difficulty for low cost ceramic filter producers in achieving the WHO (2011) recommended standards for household water treatment systems.

### EXPERIMENTAL PROCEDURE

**Slip preparation**

Ceramic products based on lithium alumina-silicates maintain steady dimensions in a wide temperature interval and can withstand thermal shock without damage. For this reason it was selected as starting material. Lithium alumina-silicate slip with 55 weight % loading was prepared by ball milling for 15 h and 30 h using spherical alumina grinding media (Ø = 15 to 30 mm). This allowed for a shorter firing cycle as well as the advantage of faster cooling rates without product damage (cooling cracks). The material required no deflocculants or any further additions for obtaining the required flow rates. In Figure 1 the impact of milling on the particle size distribution for raw material before (−150 μm) and after milling (15 h and for 30 h), is indicated.

Starch was selected as the pore former because of its dilatant rheological properties (Gilbert 2001; Gregorova & Pabst 2007; Chryss & Pullum 2007). The starch particle size distribution complimented that of the lithium alumina-silicate making allowance for the dense packing during the casting process. Based on a starch density of 1.43 g cm⁻³ and a lithium alumina-silicate density of 2.46 g cm⁻³, the dry volume percentage of starch added giving the best green strengths were 33, 36, 39 and 42 percent (standard, low, medium and high flow filters respectively). The starch was added to the ball milled slip followed by ball milling for 2 hours to improve slip homogenisation and conditioning.

The slip flow properties and specific gravity (SG) were then tested.

**Flow properties**

A 100 cm³ slip sample was poured into a funnel and the timed release through a 3 mm funnel opening was determined. This flow time reading (seconds) is used as a viscosity indicator. The flow measurements were performed at a constant temperature of 25 °C. Slip from the funnel filled a 100 cm³ measuring cylinder for the determination of the slip specific gravity (SG). Density and flow measurements were kept within pre-determined limits to avoid variations that can result in casting defects (Ergun 2004).

**Forming**

The candle filter moulds were kept full of slip until the desired wall thickness had been achieved. The forming process was then stopped by inverting the moulds to remove the excess slip (drain casting). After a short drying period
in the mould the shaped piece was stripped and then dried at 99°C overnight before further testing and evaluation. The final shape of the filter after stripping is shown in Figure 2.

| Table 1 | Summary of bacterial and viral testing for low-cost ceramic water filters (adapted from Brown & Sobsey (2010) and Simonis & Basson (2011)) |
| Reference | Bacterial Type | Reduction |
| | | % | LRV (log₁₀) | Remarks |
| Benchmark standards for low cost CWF | | | |
| Dies (2000) | *Escherichia coli* | > 98 | Katadyn filter |
| Sagara (2000) | *Escherichia coli* | | Nepalese CWF |
| Lantagne (2001) | *Giardia lamblia* | 4.6 | |
| | *Cryptosporidium parvum* | 4.3 | |
| Dies (2003) | *Escherichia coli* | > 98 | White Clay CWF |
| | *Escherichia coli* | > 98 | Hong Phuc CWF |
| Coulbert (2005) | *Escherichia coli* | 99.8 | Pozzani CWF tested 5 types of commercial filters |
| Franz (2005) | *Escherichia coli* | 92 – 100 | |
| McAllister (2005) | Bacteria | 99 | Potters for Peace with CS |
| | Viruses | 20 | |
| Clasen et al. (2006) | | | mean reduction of 39 – 44% in diarrheal disease |
| Van Halem (2006) | *Escherichia coli* | 3 – 6.8 | |
| | *Clostridium spores* | 3.3 – 4.9 | |
| Baumgartner (2007) | *Escherichia coli* | 99.8 | Filtron with CS |
| | Total coliforms | 99.4 | |
| WSP (2007) | *Escherichia coli* | UP TO 98 | 50% reduction in diarrheal disease |
| | *Escherichia coli* | Boiling: 99 | |
| Oyanedel-Craver & Smith (2008) | *Escherichia coli* | ≥ 97.8 | |
| Brown (2010) | Viruses | < 90 | |
| | *Escherichia coli* | Mean 99 | 2.1 – 2.9 |
| | Bacteriophages MS2 | 90 – 99 | 1.2 – 4.1 |
| USA and other standards for producing drinking water (USEPA 1987; NSF 2003) | Bacteria | 6 | Specification for filters |
| | Viruses | 4 | |
| | Protozoa | 3 | |
| Brazil ABNTNBR 14908 | *Escherichia coli* | 2 | No reduction required |
| | Viruses | | |
| WQA ORD9001 | *Escherichia coli* | 3 | |
| | Bacteriophages MS3 | 3 | |

Note: References cited in Table 1 can be found in Brown & Sobsey (2010) and Simonis & Basson (2011).

After final firing, a sample was randomly selected for each flow type; three samples were cut for each filter from the top (casting side), middle and bottom sections and tested for segregation (Figure 2).
Pyrolysing, burnout and sintering

The pyrolysis process was carried out separately from the sintering process to reduce cracking that results from stresses generated during the pyrolysis process in the low strength ceramic matrix. The following first stage firing factors were used to avoid cracking in the final product:

(a) packing product on an open steel grid for maximum volatile release
(b) rate-controlled firing with temperature increase set at 1 °C/min
(c) limited oxygen kiln environment (sealed doors and closed flue)
(d) limiting the product weight loss during pyrolysis phase
(e) product re-packing onto high temperature kiln ware and inspection stage.

The starch pore former was first pyrolised (removal of volatiles leaving a carbon structure) at a temperature of 220 °C to 240 °C before burning off the carbon structure in an oxidising kiln with a slow temperature increase (1 °C per minute) up to 600 °C. Figure 5 shows the carbon structure within the ceramic matrix after pyrolysing. Sintering was completed at 1,250 °C for 1h to minimise shrinkage. Total dry and fired shrinkage of 3% was obtained.

The coated filter was prepared by dipping a standard filter in a finely ground lithium alumina-silicate slip, drying the filter and then re-firing the filter to 1,000 °C.

Product testing

The determination of the sintered pore structures in terms of porosity was done using three complementary methods: water immersion, Hg porosimetry and scanning electron microscope (SEM) investigations. Water immersion and Hg porosimetry were used to identify both the smaller pores caused by the contact areas, or necks, between the larger spherical pores left by starch particles as well as the larger pores caused by the starch agglomerates. SEM, on the other hand, visually showed the larger pore structures left by starch particles. For use as a bacterial filter, the smaller connected pores at the neck points will be the determining factor, whereas the larger pores will ensure increased hydraulic conductivity ensuring adequate filtration rates during use.
Porosity and density measurements

The density and porosity of the sintered product was determined by the water immersion (Archimedes) method. Apparent specific gravity (ASG), apparent porosity (AP) and water absorption (WA) were obtained from the three weights (dry, saturated and suspended weights) using appropriate equations.

Pore size distribution

The bi-modal pore size distribution of the sintered product was determined using a mercury porosimeter (Micromeritics, USA). The pore size distribution characterised the interconnected necks as well as the pores left behind by the starch particles (Tari 1999).

SEM investigation

The overall pore size and structure formed by the burned-off starch particles were studied using SEM.

Bacteriological analysis

Three different bacterial cultures were tested as well as three natural local water sources. The culture selection was based on the following characteristics and morphological properties:

(a) *Escherichia coli* effectiveness as a faecal indicator organism

(b) *Staphylococcus aureus*‘ tendency to form clusters

(c) *Bacillus cereus*‘ ability to form endospores.

Two bacteriological methods were used for the preparation and testing of influent and effluent from the ceramic filters:

Method 1: For the three different bacterial cultures, sterile water was inoculated with known concentrations of microbes with different morphologies and filtered through the ceramic filters. Both the influent and effluent water were assayed to determine the microbial reduction (WHO 2011). The three cultures were prepared overnight in 100 ml of nutrient broth (Merck Cat. No. 1.05443.0500) and inoculated in 1,000 ml sterile distilled water as influent water. Ten-fold dilutions were used for the influent water. The spread plate technique was used to count the colony forming units (CFU) on nutrient agar plates (Merck Cat. No. 1.05450.0500) where 0.1 ml of water samples were pipetted aseptically on the different agar plates and spread with a sterile glass spreader. All plates were incubated at 37 °C for 24 hours. Samples were prepared in triplicate and CFU averages were calculated (Standard Methods, APHA et al. 2006).

The ceramic filters for the standard and coated filters were tested in parallel. Filters were first flushed using 250 ml of sterile distilled water (at least 10 volume displacements). To simulate actual household use conditions, 20 litres of influent was completely filtered (WHO 2011).

Method 2: For the three natural water sources, three local water sources (a lake and two streams) in close proximity to the University of Zululand (KwaZulu-Natal, Republic of South Africa) were used as influent water for the filters. The three water sources are used as drinking water supply whenever there is a breakdown of the centralised water distribution system.

Three ceramic water filters for each filter grade (permeability: standard, low, medium and high) filters were also tested in parallel. Filters were first flushed using 250 ml of sterile distilled water (at least 10 volume displacements). To again simulate actual household use conditions, 20 l of influent was completely filtered.

Total coliforms, faecal coliforms and heterotrophic bacteria were enumerated through use of the standard membrane filtration method as described in the Standard Methods (APHA et al. 2006). Influent and effluent water samples were filtered aseptically through sterile 0.45-μm filters (Millipore Cat. No. HAWG047S31) using a vacuum aspirator. Using sterile forceps the filters were transferred onto the following prepared media for microbiological testing Standard Methods (APHA et al. 2006):

(a) nutrient agar (MERCK Cat. No. HG0000C1.500) for the heterotrophic count

(b) Eosin blue methylene blue agar (MERCK Cat. No.1.01347.0500) for the total coliforms count

(c) ChromoCult agar (MERCK Cat. No.1.10426.0500) for faecal coliforms count.

Samples were prepared in triplicate. All plates were incubated at 37 °C for 24 hours except for the ChromoCult medium for faecal coliforms, which was incubated at 44 °C for 24 hours. CFU averages were calculated for the respective determinations (Simonis & Basson 2011).
Hydraulic conductivity

The hydraulic conductivity for the assembled filters was tested using a constant head test. The filter was first fully saturated prior to testing.

RESULTS

Rheology

The lithium alumina-silicate required limited ball milling for obtaining a sufficient – 1 μm fraction, dispersed properly without deflocculation and matched perfectly with the rheology of the starch. The slip therefore showed dilatant behavior, which is characterised by an increase in viscosity as the shear rate increased.

Shaping and stripping

Although high viscosity slips were prepared, the slip was easy to pour and filled the mould quickly without leaving pouring marks on the outer surface. After consolidation in the mould the product stripped cleanly from the mould indicating that little or no segregation had occurred and that water had been removed from the product into the plaster mould.

The segregation was tested using immersion testing. The results from the various positions for each filter type are indicated in Table 2.

Burn-out and sintering

The burn-out stage allowed for improved de-gassing and made it possible for rejects to be removed at an early juncture and at a lower firing temperature (230 °C) than the more expensive firing stage, where a higher temperature (1,200 °C) is used.

X-ray powder diffraction (XRD) analysis

XRD analysis carried out on the sintered product revealed diffraction curve patterns, from which two crystalline phases could be identified:

- LiAlSi3O8 forming the dominant phase
- SiO2 in trace quantities but acting as internal standard.

The dominant phase obtained compares best with that for the tetragonal high temperature phase of LiAlSi3O8 (International Index Reference Pattern: 35–794) and forms the end member of a complete solid solution series with beta-spodumene.

Pore structure

Figure 4 indicates the pore size distribution obtained from Hg porosimetry for the porous sample. The maximum incremental pore volume was intruded into the 2–3 μm pore size. The bimodal distribution indicates that a smaller micro pore size of 0.01–0.02 μm (necking between pores) also occurred.

Figure 5 shows the SEM images at two scales for the microstructure of two sintered samples. The large spherical-shaped pores correspond well to the shape and size of the original starch particles.

The SEM images show the following textural features:

(a) Sintered fine grained equant particles 0.2 to 2 μm in diameter in a glassy matrix that tends to fill all the

Table 2 | Determination of segregation by using the immersion test (n – 3) for the low flow filter

<table>
<thead>
<tr>
<th>Sample position</th>
<th>Result</th>
<th>AP %</th>
<th>WA %</th>
<th>ASG g.cm⁻³</th>
</tr>
</thead>
<tbody>
<tr>
<td>China</td>
<td>x</td>
<td>67</td>
<td>80</td>
<td>2.65</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>1</td>
<td>2</td>
<td>0.01</td>
</tr>
<tr>
<td>Top</td>
<td>x</td>
<td>67</td>
<td>85</td>
<td>2.42</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>0</td>
<td>0</td>
<td>0.03</td>
</tr>
<tr>
<td>Middle</td>
<td>x</td>
<td>73</td>
<td>109</td>
<td>2.41</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>0</td>
<td>0</td>
<td>0.05</td>
</tr>
<tr>
<td>Bottom</td>
<td>x</td>
<td>73</td>
<td>113</td>
<td>2.44</td>
</tr>
<tr>
<td></td>
<td>s</td>
<td>0</td>
<td>1</td>
<td>0.01</td>
</tr>
</tbody>
</table>
spaces between the individual particles with very few micro-pores of submicron size.

(b) Course vesicular structure where the rounded pore spaces are mostly larger than 2 to 4 μm. These pores appear to provide the main interconnecting channel ways.

Hydraulic conductivity ($k$) results

Table 3 shows the $k$ values for all three flow types. The low $k$ value obtained for the porous filter translates to a 1–21 h$^{-1}$ filtration rate, making the filters suitable for use by a small family when fitted into the BACSTER bucket filter.

Table 4 shows the results from Archimedes immersion tests. The results correspond with the increasing $k$ values as the concentration of starch was increased from 36% to 42%.

Microbiological results

Table 5 shows the results from the bacteriological analysis. The results show that these micro-porous ceramic filters are eminently suitable for eliminating bacteria from polluted drinking water when compared with other low cost filters shown in Table 1.

DISCUSSION AND CONCLUSION

The historically tried and tested method of slip casting for the manufacture of porous ceramic used in this study has been shown to be very successful. The excellent flow properties and dispersability of the starch when mixed with water negated the use of any dispersants or flocculants, while ensuring high solid load density. This ensured minimum segregation (Table 2) and maximum casting densities, providing a sufficiently strong green product that could be stripped and handled without breakage. The overall pore size identified
from the SEM images and the pore size distribution from Hg porosimetry agreed with each other. Limited dimensional change (shrinkage) gives accurate control of the ultimate filter size after sintering. The hydraulic conductivity \( k \) could be classified as low to medium (soil permeability scale \( \text{– Lambe (1951)} \)) depending on the starch addition.

Bacteriological testing exceeded all expectations. The WHO (2011) recommends a performance requirement for
household water treatment (HWT) technologies and associated log$_{10}$ reduction criteria as interim, protective and highly protective for reference microbes (virus, bacteria and protozoa). The log$_{10}$ reduction for bacteria specifies a $\geq 4$ requirement. The low cost filters developed and tested in this study exceeded the extreme performance criteria for bacteria. Further testing is required, as recommended by WHO (2011), for correct, consistent and continuous use as well as for certification. Future filter testing will include virus and protozoa reference microbes.

This low cost method and inexpensive equipment, makes this one of the more promising techniques for producing ceramic filters in a rural setting.

REFERENCES


Mandal, S. 2007 Synthesis of low expansion ceramics in Lithium-alumina-silica system with zirconia additive using the power precursor in the form of hydroxyhydrogel. Ceramics International 33, 123–132.


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