Comparison of a native clay soil and an engineered clay used in experimental ceramic pot filter fabrication

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ABSTRACT

Ceramic pot filters (CPFs) have been shown to be an effective means of household water treatment in the developing world. The filters are typically made using local labor and locally available materials including clay soils and various burn out materials used to create porosity. Artisanal approaches may be used to manufacture the filters, and there have been efforts to improve CPF performance through laboratory studies. The importation of soil to make the filters may be highly regulated and could be cost prohibitive, so some researchers use commercially available clay to fabricate experimental filters. However, such efforts typically do not include a comparison of the engineered clay to native clay soil, nor do most studies compare the performance of experimental CPFs fabricated from engineered clay to CPFs made from native clay. This study compares mineralogical and geotechnical properties of a clay soil from Rabinal, Guatemala, used to produce CPFs in that country to an engineered clay developed for use in laboratory experiments. Flowrate is the primary quality control parameter used in CPF production, and performance testing indicated that experimental CPF flowrates were not necessarily a function of clay composition. However, engineered clay could be used as a surrogate for native soil with some limitations.

Key words | ceramic pot filters, clay, drinking water, flowrate, Guatemala

INTRODUCTION

The World Health Organization (WHO) and United Nations Children’s Fund (UNICEF) (2013a, b) update on drinking-water and sanitation indicates that approximately 768 million people did not have access to an improved drinking water source in 2011. One way in which residents can obtain clean drinking water in developing areas is through household water treatment (HWT) systems. Sobsey et al. (2008) and Hunter (2009) have identified ceramic pot filters (CPFs) as one of the most effective types of HWT. CPFs are porous clay pots with or without silver nitrate or colloidal silver that are placed into water containers (usually five gallon buckets). The CPF is periodically filled with raw water by the consumer, and the treated water is collected in the bottom of the bucket, where it is available for use via a spigot. CPFs are made by mixing local clay with sawdust or some other burnout material and water, and then forming it into the shape of a pot. The pots are then dried and fired in a kiln where the sawdust burns out leaving pore spaces for filtering water (Van Halem et al. 2007). There are several factories throughout the world that produce CPFs and each operation has different quality control measurements, including visual inspections at various stages and auditory inspections after firing; however, nearly all manufacturers rely on flowrate measurements as the final quality control criterion (CMWG 2011). For example, Clark & Elmore (2011) state that CPFs from a factory in Antigua, Guatemala are released to consumers if flowrates are between 1 and 2 L/hour. There have been several studies conducted on filters from production plants worldwide, including those conducted by Van Halem et al. (2007, 2008), Lantange et al. (2010), Clark & Elmore (2011), Archer et al. (2011), and others. Production CPFs are relatively delicate to ship and others have fabricated filters in their laboratories for CPF-related research.
(Oyanedel-Craver & Smith 2008; Plappally et al. 2011; Elmore et al. 2011). However, it is expensive and administratively difficult to import large quantities of clay for experimental CPF fabrication, and US Department of Agriculture (USDA) permits are required for importation. For example, approximately 9 months were required to obtain the USDA import permit for this study: the authors had to travel to Guatemala to package the clay according to the permit requirements, a customs broker was retained in the USA to assist with the import, the shipping costs exceeded US$1,000, and the permit-required sterilization of the clay required approximately 2 weeks in the US laboratory to complete. A common alternative to native clay is to fabricate experimental CPFs from engineered clays composed of commercially available material. However, studies using the engineered clays typically do not comment on the potential physical and performance differences between those surrogate materials and the native clays used for production CPFs. This work describes the development of an engineered clay that can serve as an experimental substitute for the native clay used to produce CPFs in the factory in Antigua, Guatemala. The engineered clay is compared to the Guatemalan clay in terms of mineralogy and geotechnical characteristics, and a performance study is performed using experimental CPFs fabricated from the two materials.

**METHODS**

**Characterization of Rabinal clay**

CPF are produced at multiple locations in Guatemala, and a CPF factory located in Antigua, Guatemala has collaborated in several CPF studies including Archer et al. (2011) and Elmore et al. (2011). The clay used at the Antigua factory is mined from a pit in Rabinal, Alta Verapaz, Guatemala, which is a municipality known for the production of roof tiles, domestic pottery, and other ceramic products. The clay is batch homogenized using a skid loader in Antigua and stockpiled as CPF feedstock. The clay used for this study was collected from the homogenized stockpile, transported to the ceramic engineering laboratories at the Missouri University of Science and Technology, and heat sterilized at 121°C for at least 2 hours according to the terms of the USDA soil importation permit obtained for this study.

Clay characterization is a multi-step process which involves both chemical (mineralogy) and physical properties. Terrones et al. (2008) conducted a study to characterize the nature and morphology of clay from Campo dos Goytacazes, Brazil. The methods used to identify the clay’s chemical and physical properties included grain size distribution, X-ray fluorescence (XRF), and X-ray diffraction (XRD). XRF is used to give an elemental breakdown of the material while XRD is used to determine the mineralogical composition, or phase identification. Bianchini et al. (2002) used XRF analysis and other techniques to determine the chemical and mineralogical composition of clay sediments from Ferrara, Italy. CMWG (2011) found that clay properties that can impact CPF production and CPF flowrate are the shrinkage and workability or plasticity of the clay. This study used tests similar to those described in the literature above to characterize the Rabinal clay and to develop a reasonable surrogate material.

Geotechnical testing of the Rabinal clay included grain size analysis and Atterberg limits. Grain size analysis was used to quantitatively identify the sand, silt, and clay fractions because those components influence the workability of the material. Grain size analysis of the sand fraction (particles larger than 0.075 mm or a US Standard No. 200 sieve) was conducted by soaking the sample for at least 12 hours, washing the material through a series of sieves (US Standard Nos 10, 20, 40, 60, 100, and 200) and drying the sample in an oven for at least 12 hours at a minimum of 75°C. Washing was conducted following ASTM C117, and five separate washes were performed to obtain an average value of sand content. Hydrometer testing was conducted to determine the grain size distribution below the No. 200 sieve size. The hydrometer analysis was generally conducted following ASTM D422; however, data were collected beyond the standard 24 hours to obtain a more complete gradation curve. Atterberg limit testing on the Rabinal clay was completed according to ASTM D4318. The results from three samples were averaged to determine the liquid limit, plastic limit, and plasticity index of the clay.

The physical characteristics of the clay when fired in a kiln were characterized by hydrating the clay for 12 hours with deionized water to produce a formable body. The
The analysis of the chemical and mineralogical composition of the clay was conducted using XRF and XRD analyses. XRF analysis was performed by Acme Analytical Laboratories Ltd (Vancouver, British Columbia, Canada) using a PANalytical Axios XRF machine. XRF analysis was conducted on five samples of the Rabinal clay. XRF analysis evaluates the percent by weight of several oxides including silica (SiO2) and alumina (Al2O3) as well as loss on ignition (LOI). LOI is important to porosity because this indicates the amount of organic material that naturally occurs in the clay and will be burned out upon firing. XRD analysis was conducted at the Missouri University of Science and Technology Materials Research Center using a PANalytical X’Pert Pro Multi-Purpose Diffractometer. XRD analysis evaluates the mineralogical composition of a sample.

**Development of engineered clay**

The components of the engineered clay were selected to represent the principal Rabinal clay mineralogy; however, commercial clays could not reproduce the relatively high percentage of mica found in the Rabinal samples. Pacer Corporation Custer feldspar was selected as the primary component of the engineered clay. US Silica F-65 sand was included in a percentage equal to the sand content of the Rabinal samples. CMWG (2011) found that CPF production and CPF flowrate could be impacted by the shrinkage and workability (plasticity) of the clay material. Data from Izdebska-Mucha & Wojcik (2013) were used to semi-qualitatively characterize shrinkage as a function of either liquid limit, plasticity index, or plastic limit. The review of those data suggested that liquid limit was the better predictor of shrinkage. Therefore, the percentage of Pure Wyoming bentonite was varied in the engineered clay to change the liquid limit of each formulation. The final product contained an amount of bentonite such that the liquid limit of the engineered clay was equal to that of the Guatemala clay. Other clays were used in smaller percentages to offset the low plasticity of the Custer feldspar. These additional clays also provided chemical and mineralogical variation.

**Formation of filters**

After the formulation of the engineered clay composition was completed, experimental CPFs were fabricated in order to perform flowrate comparisons. Other than the clay composition, either Rabinal clay or engineered clay, all of the other fabrication factors were the same.

CMWG (2011) summarized best practice recommendations for CPF manufacturing based on data collected from CPF factories in several countries. That document lists several types of burn out material including rice husks, sawdust, and peanut shells. The CPF factory in Antigua uses pine wood sawdust as burn out material, so yellow pine sawdust was used in this study. The clay to sawdust ratio was selected as 6:1 (14%) by weight to fabricate experimental CPFs from both the Rabinal clay and the engineered clay. That weight fraction was close to the center of the typical factory-used range of 5 to 25% reported by CMWG (2011). CMWG (2011) reported that some CPF factories sieved burn out material using sieves ranging between No. 8 and No. 48, while other factories did not sieve the burn out material. The sawdust for this study was sieved to remove particles larger than a No. 8 sieve which was consistent with both the practice reportedly employed at the Antigua factory and the CMWG observations. The percent water and percent clay of each bucket of extruded clay was determined using ASTM D2216 and the sawdust and clay were mixed using hand wedging. The standard production practice is to form CPFs using molds and a hydraulic press (CMWG 2011). However, commercial...
ceramics are formed using a rotational process called jollying described by Kingery (1958). The jollying process was used to form the experimental CPFs because the process yielded more consistent shapes relative to pressing. Greenware was allowed to cure initially at ambient conditions up to 10 days prior to drying in an oven at 35 to 50 °C for at least 8 hours prior to firing.

The experimental pots had a bottom inside diameter of 121 mm (4.75 inches), an inside diameter at the top of 133 mm (5.25 inches) with an overall wall thickness of 12.7 mm (0.5 inches) and an internal height of 114 mm (4.5 inches). A saturated filter could hold a maximum volume of approximately 1.25 L. The experimental filters were smaller than the production CPFs from Guatemala, which were measured to have a maximum volume of approximately 9 L. The smaller size permitted a maximum of 14 experimental filters to be fired simultaneously in the experimental kiln, while it was observed that as many as 200 production CPFs could be fired simultaneously in the Guatemala kiln.

Test firings were performed with coupons made from the Rabinal clay and the engineered clay. The porosity of the test pieces was characterized using the Archimedes test. The kiln firing of the experimental CPFs was conducted using a custom sequence to allow for complete burn out of the sawdust. The kiln temperature was slowly increased to 230 °C where it was maintained for 1 hour. The temperature was then slowly ramped up to 530 °C where it was again maintained for an hour before increasing the temperature to the final 993 °C value. Seven filters were fabricated from the engineered clay, and seven filters were fabricated from the Rabinal clay. However, two of the Rabinal filters were damaged during fabrication, so flowrate testing was performed with the remaining five Rabinal filters.

Flowrate testing

The 12 experimental CPFs were saturated in tap water for 24 hours prior to flowrate testing, which was consistent with the quality control procedures reported for the Antigua CPF factory. Flowrates were measured by suspending each CPF over a bucket, filling the CPF with approximately 960 mL of tap water, and measuring the filtered water retained in the bucket after 90 minutes. The CPFs were refilled with tap water and the cycle was repeated over a 12-hour time period. Due to time constraints and limited scope of the flowrate analysis, data were collected every 90 minutes (1.5 hours) over three 12 hours cycles for a total of 24 data points per CPF.

RESULTS AND DISCUSSION

The grain size analysis yielded the following results: the average sand content of the Rabinal clay was found to be 7.4%, the average silt content was found to be 58.2%, and the average clay content was found to be 34.4%. The Atterberg Limits testing was used to determine the liquid limit, plastic limit and plasticity index. The average liquid limit was found to be 49.7% with values ranging from 49.4 to 50.0%. The plastic limit was estimated to be 31.1% with values ranging from 31.8 to 30.4%. The average plasticity index of the clay was estimated to be 18.6% with values ranging from 17.6 to 19.4%.

The XRF results from five samples were averaged to determine the chemical components of the Rabinal clay. The dominant components were found to be silica (SiO₂) at 59.14% and alumina (Al₂O₃) at 19.67%. There were also significant (greater than 1%) fractions of iron oxide (Fe₂O₃) at 6.694%, potassium oxide (K₂O) at 3.574%, sodium oxide (Na₂O) at 1.602%, magnesium oxide (MgO) at 1.53%, and calcium oxide (CaO) at 1.198%. These are all commonly found in several minerals including micas and feldspars according to Nesse (2000). The XRF analyses also characterized the percent of LOI at 5.23%. These results are congruent with XRF analysis conducted on several raw clayey materials by Montana et al. (2011). Many of the clays studied in Montana et al. (2011) contained alumina (14.86–24.35%) and silica (54.91–59.49%) contents similar to that of the Rabinal clay.

A number of these clays were found to contain quartz, feldspars, and mica. XRD analysis was used to identify the primary minerals present in the Rabinal clay. The XRD analysis showed that the Rabinal samples contained mostly quartz and muscovite with albite, which is the sodium-rich end member of plagioclase feldspar.

The porosity results from the Archimedes test were analyzed and the final firing temperature was found to be the value where the porosity of the coupons was approximately the same for both compositions. That temperature was identified as 993 °C (cone 06).
The engineered clay was developed using a number of commercial clays. Pacer Corporation Custer feldspar was the primary component of the engineered clay at 48.4% because feldspar was the primary component in Rabinal samples. US Silica F-65 sand was included at 7.4% to be consistent with the Rabinal samples. A bentonite fraction between 9 and 10% resulted in liquid limit values approximating the Rabinal value of 50. Other clays were used in smaller percentages to offset the low plasticity of the Custer feldspar. These additional clays also provided chemical and mineralogical variation and included Edgar Minerals EPK kaolin (20% by weight), Resco Cedar Heights GoldArt (10%), and KT Clays Old Mine #4 (5%). The final bentonite percentage was 9.2.

The Digital Fire Reference Database (2008) was used to estimate the chemical composition of the engineered clay given the component ratios listed in the paragraph above. That estimate, along with the results of the XRF analysis of the Rabinal clay, is listed in Table 1. The matches between the two major components, silicon oxide and aluminum are relatively close because the percentage difference between the individual compounds is 11% or less. The combined weight fraction of the minor components including the Mg, Ca, Na, and K cations are within 3% for the two clays. However, the iron oxide content of the Rabinal clay is approximately an order of magnitude greater than the estimated content of the engineered clay. Resco Cedar Heights RedArt is the only commercially available clay in the USA that has a significant iron content, and a composition analysis indicated that using RedArt to increase iron would have reduced the alumina fraction in the engineered clay. Given that iron oxide was less than 10% of the Rabinal clay, RedArt was not included in the final engineered clay mixture. The DigitalFire database was also used to estimate the LOI for the engineered clay, and the resulting value of 5.19% was essentially the same as the Rabinal LOI.

The difference in chemical composition between the XRF results of the Rabinal clay and the composition of the engineered clay is shown in Table 1. The values are ranked from largest component to smallest according to the Rabinal clay, and there are relatively small differences in the percentages of the top six compounds with the exception of iron oxide.

A comparison of the physical properties of the two sets of experimental CPFs is shown in Table 2. The goal of closely approximating the liquid limit of the Rabinal clay was achieved with a relative percent difference of less than 1% with the engineered clay, but that close match did not result in the desired approximation of shrinkage as described by the linear change values. However, both clay mixtures were sufficiently workable, and the experimental CPFs were formed without significant difficulty.

Two of the potentially more critical clay parameters governing the formation of porosity in a ceramic are LOI (because of the combustion of the organic material) and sand content (because it remains unchanged during firing). There were small differences (less than 10%) in these values for the Rabinal and engineered clays. In fact, the difference in the porosities of the two materials was also small. These three small differences imply that the resulting flowrates have a good potential to be similar; however, inspection of the Figure 1 flowrate time series shows that the Rabinal flowrates appear to be systemically higher and the engineered clay flowrates appear to be more variable.

<table>
<thead>
<tr>
<th>Compound</th>
<th>XRF-measured percent weight of Rabinal clay</th>
<th>Engineered clay percent weight</th>
</tr>
</thead>
<tbody>
<tr>
<td>SiO₂</td>
<td>59.1</td>
<td>64.0</td>
</tr>
<tr>
<td>Al₂O₃</td>
<td>19.7</td>
<td>21.8</td>
</tr>
<tr>
<td>Fe₂O₃</td>
<td>6.69</td>
<td>0.765</td>
</tr>
<tr>
<td>K₂O</td>
<td>3.57</td>
<td>5.25</td>
</tr>
<tr>
<td>Na₂O</td>
<td>1.60</td>
<td>1.78</td>
</tr>
<tr>
<td>MgO</td>
<td>1.53</td>
<td>0.273</td>
</tr>
<tr>
<td>CaO</td>
<td>1.20</td>
<td>0.305</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Property</th>
<th>Rabinal clay</th>
<th>Engineered clay</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOI (%)</td>
<td>5.23</td>
<td>5.19</td>
</tr>
<tr>
<td>Linear Change (%)</td>
<td>−1.37</td>
<td>−0.32</td>
</tr>
<tr>
<td>Porosity (%)</td>
<td>26.56</td>
<td>24.37</td>
</tr>
<tr>
<td>Plastic limit (water content, %)</td>
<td>18.6</td>
<td>30.8</td>
</tr>
<tr>
<td>Liquid limit (water content, %)</td>
<td>49.7</td>
<td>49.6</td>
</tr>
<tr>
<td>Plasticity index</td>
<td>31.1</td>
<td>18.8</td>
</tr>
<tr>
<td>Sand content (% by wt.)</td>
<td>7.4</td>
<td>7.4</td>
</tr>
</tbody>
</table>
The values shown in Table 3 for minimum, maximum, mean, and median flow also support the observation that the Rabinal flowrates were higher compared to the engineered clay flowrates. The better agreement between the Rabinal mean and median flowrates compared to the engineered clay mean and median values as well as the fact that the Rabinal coefficient of variation (COV) is closer to unity than the engineered clay COV supports the observation that the Rabinal flowrates were less variable than the engineered clay flowrates.

Inspection of the time series graph also suggests that the measured flowrates for both sets of experimental CPFs increased with time (or the volume of water filtered). This alternative was tested by using the median flowrates measured during each time period as the summary statistic, and Figure 2 includes histograms of the median flowrates for each clay mixture. The non-parametric two-sample Wilcoxon rank-sum test (Mann–Whitney test) described by Moore (2007) was used to evaluate the hypotheses based on the differences between the two population medians.

**Table 3 | Statistical summary of flowrate data**

<table>
<thead>
<tr>
<th>Statistic</th>
<th>Rabinal clay</th>
<th>Engineered clay</th>
</tr>
</thead>
<tbody>
<tr>
<td>N</td>
<td>120</td>
<td>168</td>
</tr>
<tr>
<td>Minimum (mL/hr)</td>
<td>233.3</td>
<td>171.7</td>
</tr>
<tr>
<td>Maximum (mL/hr)</td>
<td>350.0</td>
<td>305.0</td>
</tr>
<tr>
<td>Median (mL/hr)</td>
<td>282.5</td>
<td>263.3</td>
</tr>
<tr>
<td>Mean (mL/hr)</td>
<td>284.6</td>
<td>252.6</td>
</tr>
<tr>
<td>Standard deviation (mL/hr)</td>
<td>23.88</td>
<td>33.17</td>
</tr>
<tr>
<td>Coefficient of variation</td>
<td>0.8391</td>
<td>0.1513</td>
</tr>
</tbody>
</table>

**Figure 1 | Flowrate time series.**
That test yielded a p-value of less than 0.001 with a median difference of 13.3, and that low p-value provides the basis for rejecting the null hypothesis. The results of the Wilcoxon rank-sum test were consistent in indicating that the Rabinal and engineered data are not comparable and the flowrates are not the same over time. The non-parametric Mann–Kendall test for trend (Gilbert 1987) was used to check for homogeneous trend direction. The null hypothesis of no trend in the flowrate time series for each clay mixture was tested against the alternative hypothesis that the flowrates increased with time. The S-statistics were positive for both clays. The Rabinal data had an S-value of 156, and the engineered data had an S-value of 149.

So the result of the Mann–Kendall test was that both sets of flowrates had a statistically significant positive trend over time. Sen’s non-parametric estimator of slope (Gilbert 1987) was used to estimate the true slope of the linear trend over time for the summary statistics for each type of filter. The Rabinal data yielded a positive slope of 0.74 with a 90% confidence interval of 0.46 to 0.97 and the engineered data yielded a positive slope of 0.92 with a 90% confidence interval of 0.60–1.19. The results of the statistical analyses indicate that the Rabinal filter flowrates are systemically greater than the engineered filter flowrates, but that the incremental differences timewise are similar. That is, the slopes of the straight lines fit to each time series of flowrates are similar. This increase in flowrate at early times is consistent with flowrate observations of both experimental and native CPFs described by Hubbel & Elmore (2022).

In a study conducted by Oyanedel-Craver & Smith (2008) two native clays and one laboratory clay were compared using hydraulic conductivity, which was used to calculate flowrate. In this study the same amount of burn out material by weight and sand by weight was used for each CPF formation mixture and the calculated flowrates varied from 0.5 and 1.2 L/hour for the native clays and 2.1 L/hour for the laboratory clay. This indicates the significant influence of the clay material on flowrate and further supports the need for a more uniform laboratory produced clay for the purpose of studying CPFs. The percent difference in mean flowrates between the engineered and Rabinal clay CPFs was 4.9%, a relatively close match when compared to the flowrates presented in Oyanedel-Craver & Smith (2008).

**CONCLUSIONS**

Two sets of experimental CPFs were fabricated in an identical manner with the exception of composition of the clay body. Key compositional and geotechnical parameters were measured in the native clay, and the engineered clay was formulated to match those parameters. However, the flowrates observed in the two sets of CPFs were significantly different, and the engineered clay flowrate variation was observed to be greater compared to the native clay. This was a counter-intuitive result given that it is reasonable to
expect that the inherently more uniform nature of engineered clay should produce less variable flowrates. Considering the controlled nature of the experimental CPF fabrication process, the size distribution of the pore forming material and the distribution of that material within the CPF body may be the source of the flowrate variability, because no mechanism is employed to manage those variables (with the exception of a single sieving effort). This work shows that replicating the clay composition alone is not sufficient in terms of flowrates, and the characterization of porosity parameters may be a data gap.

However, the experimental results indicated that there were similarities in the flowrate data. The principal similarity was that the flowrates for the two sets of CPFs increased at about the same rate over time or over the initial quantity of water filtered. Given that the typical acceptable production CPF flowrate range of 1 to 2 L/hour is not theoretically founded, studies using experimental CPFs may be insensitive to flowrate magnitude. Therefore an engineered clay with a composition similar to the one used in this study may be appropriate for use in CPF experiments given the noted flowrate limitation.

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Clay Science

Centers for Disease Control and Prevention, Atlanta, GA, USA.


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