

Effect of sintering temperature on the efficiency of clay ceramic water filter

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Abstract

Billion cases of diarrhea occur worldwide each year that result in million deaths because of lack of access to safe drinking water. Clean water is a key concern in many communities of developing countries. Household water treatment and safe storage methods, such as ceramic water filters, is one of the proven methods and have been shown to reduce diarrheal prevalence. The aim of this work was to develop a ceramic water filter with a better flow rate, which is capable to remove chemicals as well as microbial contaminants, by examining the effect of altering specific design variables. Ceramic water filters were developed from 60% of clay, 25% sawdust, and 15% grog by volume and sintered in 800-950°C range at 50°C intervals with 5°C per minute heating and cooling rate for 6 hours. The developed ceramic filters were characterized with FESEM, EDX, XRD, pH meter, BET and FTIR. The better ceramic water filter with total porosity of 32.95 ± 0.03 , average pore diameter of 50 nm, flow rate $1.97 \pm 0.23\%$ and *Escherichia coli* removal efficiency $99.36 \pm 0.55\%$ was developed from 60 % clay; sawdust 25%, 15% grog by volume and sintered at 900°C for 6 hours.

Keywords: Sintering, Ceramic filter, *Escherichia coli*, Microstructure, Porosity

1. Introduction

Safe drinking water is not available to the majority of the people living in the developing countries. Clean water is one of the most important public health measures in providing major controls against infectious diseases (UNICEF /WHO, 2009 & Clasen et al., 2004). Filtration, which is one of the proven Household water treatment and safe storage methods (HWTS) have been shown to reduce diarrheal prevalence by an average of 45% among users (Clasen and Boisson, 2006).

One of the low cost methods of water purification is the use of clay ceramic filters. Ceramic water filter is a porous ceramic medium to filter microbes or other contaminants from water. The pore size of the ceramic medium is engineered to trap anything bigger than a water molecule. The investigation of ceramic water filter to remove contaminants and microbes in water processing is becoming more effective and popular among ceramists (Zereffa and Bekalo, 2017). Ceramic filters have been proven

effective for improving water quality (Brown et al., 2007), users and implementers often express concern over their inability to produce a sufficient quantity of water due to their slow flow rate of approximately 1-2 liters per hour. If the flow rate could be increased by altering the current filter design, it would improve the ceramic filter's viability as a scalable HWTS option (Erhuanga et al., 2014). Water filtration is a process of removing suspended and colloidal particles in water. It is principally concerned with the removal of insoluble impurities, biological or physical. The contaminants are physically prevented from moving through the filter either by screening them out with very small pores and/or, in the case of carbon filters, by trapping them within the filter matrix by attracting them to the surface of carbon particles (the process of adsorption) (Ajayi and Lamidi, 2015).

One of the methods of water purification in this category is the use of ceramic filters (Sobsey et al., 2008). These filters may be produced with different materials and in various forms (Potter for Peace, 2006; Hagan et al., 2009). However, the most common ceramic filters are modified fired clay filters, which are supplied in candle, pot and frustum forms. These ceramic filters have become conventional in some parts of the world, such as India and Nepal (Mintz et al., 1995).

The ceramic filter designs developed from 15% saw dust, 80% clay and 5% grog that sintered at 950°C and 1000°C reported with *E. coli* removal efficiency

of $97.50 \pm 1.73\%$ and $95.94 \pm 1.73\%$ (Zereffa and Bekalo, 2017).

Therefore, the aim of the research was to develop a ceramic filter from 60% of clay soil, 25% sawdust and 15% grog that sintered at different temperature for household water treatment that is capable of removing microbial and chemical contaminants from water.

2. Methodology

The main raw materials used for the production of the filter are clay, water, grog and burnout materials (sawdust). Clay soil was collected from Kechene, Mariam River. Kechene Woreda is located in Addis Ababa administrations of Gullalle sub-city Woreda at 38°45'0"E and 9°3'30"N. Each of the basic materials was subjected to various physical treatments before use in the preparation of the filter materials. These treatments include sun drying, crushing and grinding (with piston and mortar), and sieving (using 75 μm sieve size). Grinding of the clay, grog, and sawdust to a finer texture was done after the materials had been sun-dried. Sieving was performed for all basic materials that is; clay, sawdust, and grog separately to obtain different particle sizes. Clay, saw dust and grog were mixed for more than one hour in dry. Then, water was added uniformly on a dry mixture of clay, saw dust and grog. The blends were again mixed in wet by wedging and rolling to get a homogenous uniform mixture and the wet mixture was divided into blocks. The blocked clay mixtures were molded in to frustum shape by using a 2-ton car

jack. The manufactured filter elements were 20 cm top diameter, 15.0 cm height, 10 cm bottom diameter, and 1.10cm wall thickness. The shaped filters were allowed to sundry for about two weeks. Once the filters were completely dried they were sintered in muffle furnace at 800, 850, 900 and 950°C temperatures for a period of 6 hours gradually starting from room temperature with an intermediate stay time 1hr at 500°C for combustible material removal in the initial heat treatment. Afterward, the filters were left to cool gradually until the temperature reached room temperature. Once cooled, the filters were soaked in water for 24 hours and tested for their clean water flux. The selected filters were washed with distilled water, dried in oven, packed properly in plastic bags to protect them from any contamination and made ready for the different tests. Each of the filter designs were replicated into 3. The contaminated river water sample was collected in sterilized containers from Modjo River, which is found in Modjo Town by purposive sampling techniques (Ilker et al., 2016). Modjo River is located 75 km East of Addis Ababa the capital city of Ethiopia which is geographically situated in between 39° 05' E – 39° 4'E longitudes and 8° 34' – 9° N latitudes. To evaluate the performance of each filter, the difference in water quality of influent and

effluent water was measured. The quality parameters determined were Escherichia coli (E.Coil), turbidity, Total Dissolved Solid (TDS), conductivity, and pH. Membrane filtration was used to determine the E. coli concentration of the influent and effluent samples of the filters. Samples were filtered in triplicate through 47 mm diameter and 0.45 µm pore size cellulose ester filters of Millipore. Influent samples were diluted before filtering through a membrane filter. The membranes were incubated on agar for 16–24 hours at 37°C. The log₁₀ reduction value is used to describe the removal efficiency in case the bacteria removal approaches 100%. The LRV can be calculated with:

$$LRV = \left\{ \frac{\text{no. } E. \text{ coli Influent}}{\text{no. } E. \text{ coli Effluent}} \right\} \log_{10}$$

Flow rates were estimated from the volume of measured filtrate and duration of flow through the filter medium. Porosity of the ceramic filters was determined using the water absorption test (direct) method. The filters were washed thoroughly and dried in oven. The dry mass of the filters was measured. The filters were again soaked in distilled water for 24 hours for saturation. The saturated masses of the filters were measured (D'ujanda, 2001). The total porosity (P) of the filter was calculated by the equation (1).

$$\text{Porosity of filter} = \frac{\text{Mass of saturated filter} - \text{Mass of dry filter}}{\text{Density of water} \times \text{volume of filter}} \quad (1)$$

Table 1: Composition of filter design in percentages and sintering temperature.

No	Filter Design	Clay(v/v)	Grog(v/v)	Sawdust(v/v)	Sintering Temperature (°C)
1	C800-60-25-15	60	15	25	800
2	C850-60-25-15	60	15	25	850
3	C900-60-25-15	60	15	25	900
4	C950-60-25-15	60	15	25	950

3. Result and Discussion

a) Chemical analysis

Table 2 shows the chemical composition of the raw and fired clay ceramic filter. The results revealed that the raw clay essentially consists of Si and Al. The amount of iron oxide is also noticeable while the contents of alkalis and alkaline-earth elements are low. The significant iron oxide content explains the red color of the raw clay sample. The composition of the fired clay is different from that of the raw clay, characterized by a significant increase of Al and Si due to the addition of grog (or fired clay without 3burnout). The relative high

amount of Al_2O_3 and low content of alkaline oxides, which serve as fluxes, indicate that the clay came from typical kaolinitic clay. The energy dispersive spectrometry (EDS) analysis revealed the presence of more elements like copper, tin and carbon in the ceramic filter surface that used for contaminated water analysis as indicated in figure 2. Furthermore, the SiO_2/Al_2O_3 ratio of 2.03 and 2.45 are above that of 1.18 associated with kaolinite, indicates an excess of SiO_2 in the form of free quartz.

Table 2: Chemical composition (%) of raw clay and fired clay ceramic filter.

Item	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	MnO	P ₂ O ₅	TiO ₂	H ₂ O	LOI
Fired	58.1	23.6	8.2	1.4	0.72	2.1	2.7	0.06	0.10	0.4	0.2	2.7
Raw	47.3	21.9	11.6	0.4	<0.01	4.2	1.5	<0.01	0.07	0.3	2.2	11.6

LOI: Loss of Ignition

Figure 2 shows the XRD pattern of the fired ceramic filter. The major peaks are associated with quartz (Q) in

agreement with the results in Table 2. Other peaks are related to kaolinite (K) and muscovite mica (M). The presence of

kaolinite can be attributed to the low-firing temperature, 600°C. It should be remembered that above this temperature kaolinite transforms to metakaolinite, which is an amorphous material and, consequently, displays no XRD peaks (Carty et al., 1998).

The porosity of the ceramic filter developed slightly increased with an increase in sintering temperature for the same composition of clay and burnout as it can be seen from the figure 3b. Moreover, figure 3b shows that the effect of firing on the porosity of the filter element that sintered at 900°C and 950°C are almost the same. The flow rate through the filters was found to vary with

change in sintering temperature. The average flow rate of filter designs: C800-50-15-35 (2.16 ± 0.32 L/h), C850-60-15-25 (2.56 ± 0.24 L/h), C900-60-15-25 (1.97 ± 0.23 L/h) & C950-60-15-25 (1.56 ± 0.25) as indicated in Table 3 were relatively good as per the potters for peace recommendation (Rayner, 2006). Filter that sintered at 850°C exhibited the fastest discharge. The flow rate of the ceramic filters was affected by the porosity of the filter elements as indicated on figure 3a. High flow rate, ~ 2.5 L/h, was obtained by low total porosity filter (C850-60-15-25) which might be due to the absence of dead and isolated pores in the filter body.

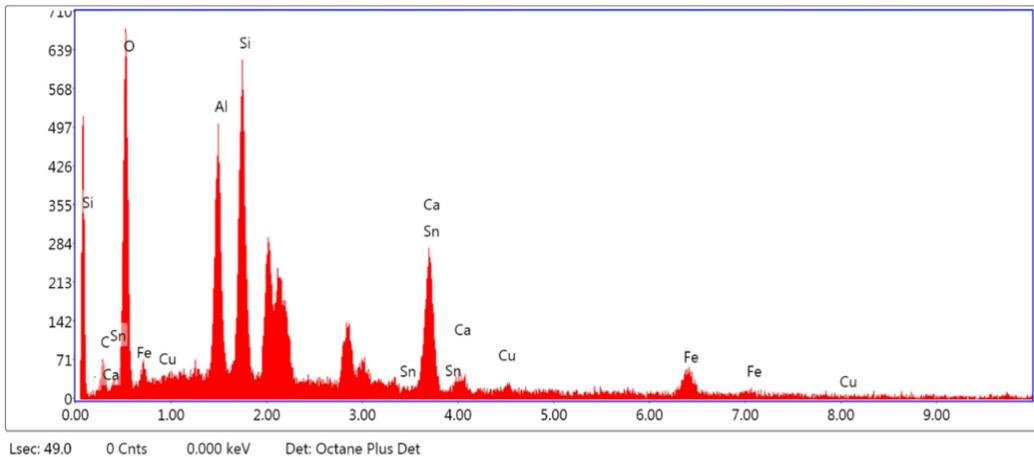


Figure 1. EDS spectrum of ceramic filter design (C900-60-25-15) after it is used for contaminated water filtration.

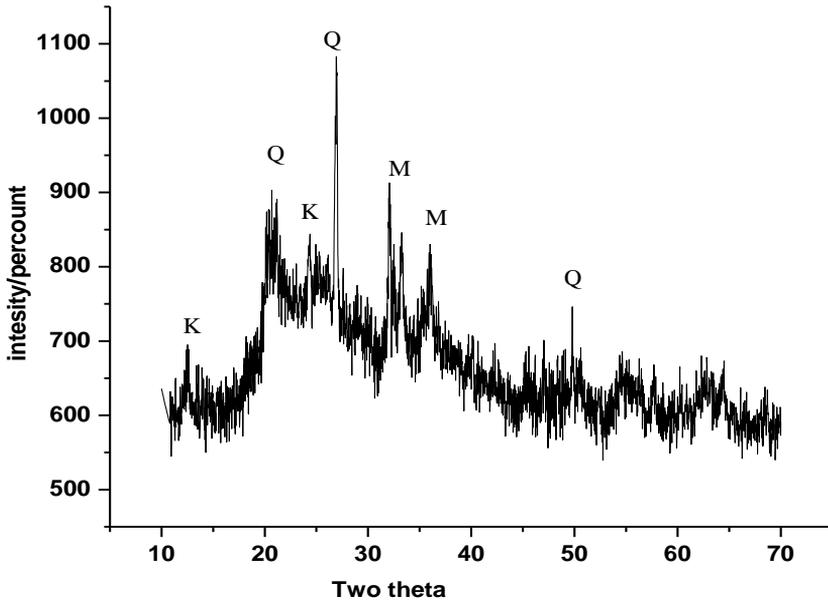


Figure 2a. X-ray diffraction pattern of raw clay.

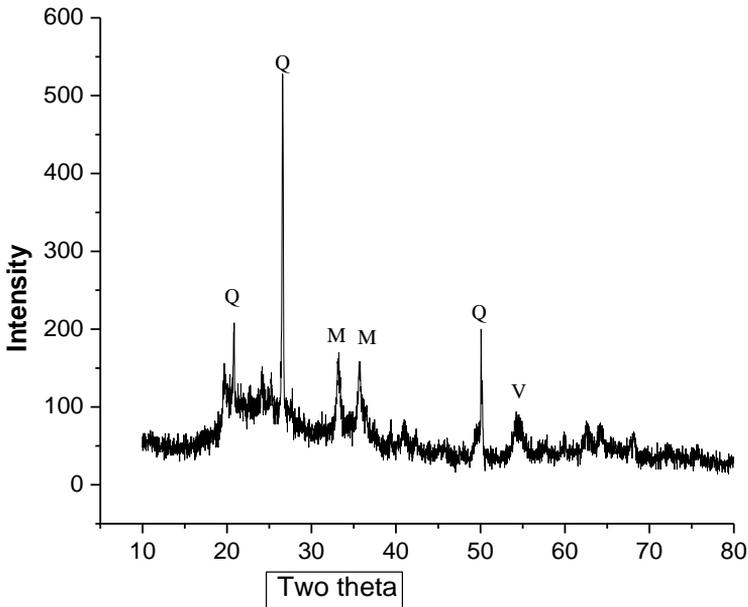


Figure 2b. X-ray diffraction pattern of C900-60-15-25

Table 3: Ceramic filter design with: average flow rate, total porosity, and E Coil removal efficiency and other quality parameters.

No	Filter Design	Flow rate (L/hr)	Porosity (%)	E-Coil (%)	TDS (ppm)	Turbidity (%)	E.C., μ s	pH
1	C800-60-15-25	2.16 \pm 0.32	31.07 \pm 0.02	98.97 \pm 1.22	288	100	138 \pm 0.23	7.4 \pm 0.13
2	C850-60-15-25	2.56 \pm 0.24	30.37 \pm 0.02	90.33 \pm 3.36	261	98.9	101 \pm 0.30	7.2 \pm 0.02
3	C900-60-15-25	1.97 \pm 0.23	32.95 \pm 0.03	99.05 \pm 1.79	250	93.5	98 \pm 0.38	7.0 \pm 0.01
4	C950-60-15-25	1.56 \pm 0.25	32.67 \pm 0.01	98.99 \pm 1.90	270	99.6	130 \pm 0.29	6.9 \pm 0.12

TDS = Total dissolved solid, E.C = Electrical conductivity,

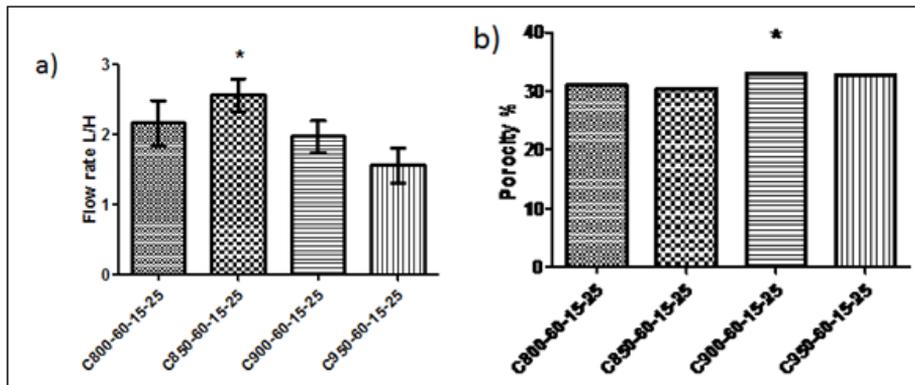


Figure 3. a) Flow rates and b) porosity of ceramic filters: C60-25-15 sintered at 800, 850, 900 and 950°C.

b) The micro-structure of ceramic filters

In this section, the microstructures of C60-25-15 ceramic filters sintered in the range of 800 - 950°C with different performance were investigated. The porosity in microstructure is one of the physical properties of ceramic materials and one of the significant factors that affect the mechanical properties. The desired flow rate and microbial removal efficiency value can be achieved due to the defects such as porosity and crack occurred during production of ceramic

materials and development of microstructure after firing. From the analysis, there were strong correlations between flow rate and sintering temperature of the clay filter. This might be due to the formation networked pores when the combustible materials burnout with the increasing in sintering temperature. Images obtained by electron microscopy techniques, which now can be considered not only as research tools, but also as very valuable instruments for quality control of microstructures. As indicated on the SEM images (Figure 4a) the

average pore size increased with the increasing sintering temperature due to the overlapping of small pores large pores at higher sintering temperature were created. Hence, the SEM

micrograph images of the mentioned composite ceramic filters confirmed as sintering temperature highly influence the performance of the filters.

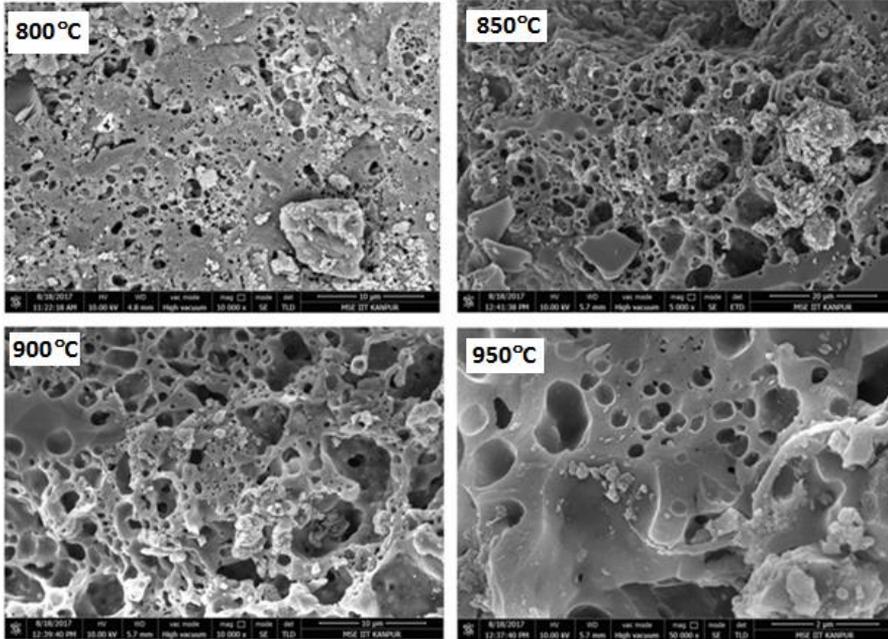


Figure 4a. SEM micrographs of the fractured surface of C60-25-15 sintered at 800, 850, 900 and 950°C.



Figure 4b. SEM micrographs of the fractured surface of C60-25-15 sintered at 850°C.

c) FT-IR Analysis

FTIR studies of the filters help in the identification of various forms of the minerals present in the sintered filter elements before and after usage. In the IR studies, the coupled vibrations are appreciable due to the availability of various groups. Most of the bands such as 3696cm^{-1} , 3622cm^{-1} , 3450cm^{-1} , 1033cm^{-1} , 917cm^{-1} , 795cm^{-1} , 692cm^{-1} , 533cm^{-1} , 466cm^{-1} show the presence of kaolinite (Franco, 2004). As indicated on figure 5a, the vibrations observed at $\sim 917\text{cm}^{-1}$ indicates the possibility of the presence of hematite (Farmer, 1974). Whereas 3622cm^{-1} , $\sim 1631\text{cm}^{-1}$, 1033cm^{-1} are also indicative of gypsum and $\sim 693\text{cm}^{-1}$ shows the possibility of the presence of calcite (Farmer, 1974). A

strong band at 3696cm^{-1} , 3622cm^{-1} and 3450cm^{-1} indicate the possibility of the hydroxyl linkage. However, a broadband at 3450cm^{-1} and a band at 1633cm^{-1} in the spectra of clay ceramic filter suggests the possibility of water of hydration in the sample. FT-IR spectrum (figure 5a and b) revealed the difference between the functional groups between the filter elements before and after usage. FT-IR spectrum revealed that the hydroxyl groups on the adsorbent surface were involved in the sorption of ions. Anion exchange and electrostatic interaction were suggested as the main mechanisms involved in the sorption of ions on the adsorbent. The changes of stretching frequency of treated filter material compared to untreated filter material confirm the chemical modification.

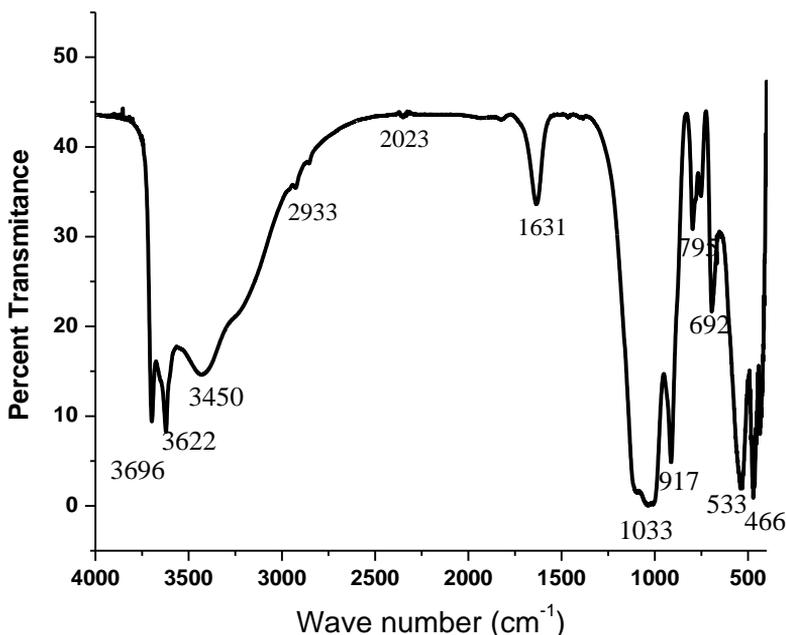


Figure 5a. Untreated FT-IR spectrum of C900-60-25-15

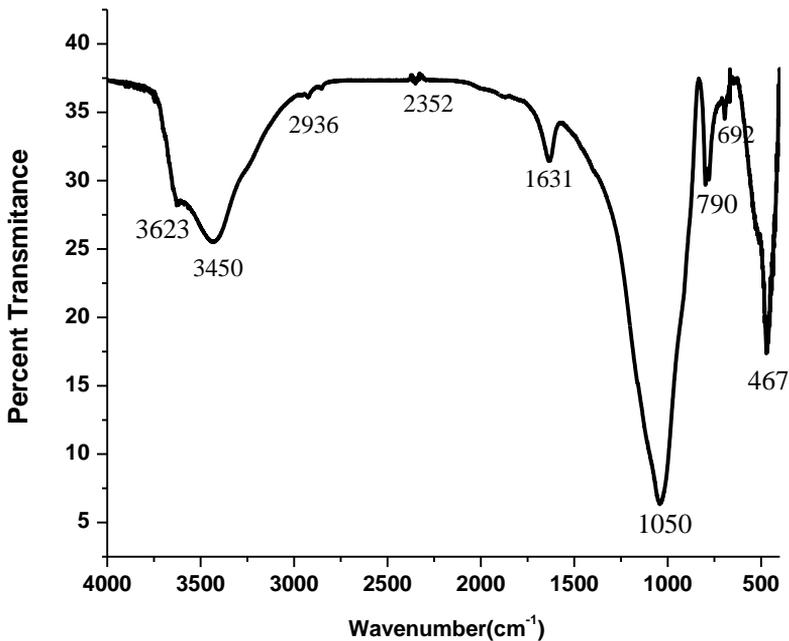


Figure 5b. Used FT-IR spectrum of C900-60-25-15

4. Conclusion

The quality of drinking water, especially in developing countries can be enormously improved by the use of the clay-sawdust composite filter. From the obtained results within the limit of this research, it can be concluded that the sintering temperature of the filter element influences the microstructure, porosity, flow rate and E. coli removal efficiency of the ceramic filters.

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