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Developing Zeolite Ceramic Filters For Purifying Open Water Sources In Rural Communities

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Abstract
Access to safe drinking water has been a challenge for most rural dwellers in Sub-Sahara Africa who rely on open water sources for their consumption and domestic activities. In attempt to purify water from these open water sources, this study looked at producing zeolite from kaolin with NaOH concentrations through crystallization process, develop ceramic candle filters and subsequent zeolite filter unit to purify unsafe water. Two procedures were used for the synthesis; pre-treatment or thermal activation of the kaolin at 650^{0}C to get a dehydroxylated product called metakaolin and hydrothermal reaction of metakaolin in alkaline solutions for crystallization of the zeolite. The effect of the zeolite ceramic filter on the users was enormous. Results showed that properties such as turbidity removal, flow rate and coliform removal were within WHO standard after the purification. A pilot study of the zeolite ceramic filter showed improved health standards such as fewer diarrhea, nausea, cramps, vomiting and good skin growth.

Keywords: Zeolite, Ceramic filters, open water sources, safe water

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1.0 Introduction

Purified water plays significant role in the health of users. It provides the most essential components for a healthy prosperous life (Ravenga, et al, 2000; WHO/UNICEF, 2006). It is therefore a major challenge that in Sub-Saharan Africa, about 20% of the population lacks access to safe drinking water (Shiklomanov, 1999). In the rural and peri-urban settlements in Ghana, and other parts of the world, dwellers access water from streams and rivers. These untreated water bodies have harmful bacteria such as legionella which grows in them leading to high infant mortality rates (Grey and Sadoff, 2007; Petrik et al, 2012; Gleick, 1993; Harbawi, et al, 2010). It therefore means that the safety and quality of water for health, livelihoods, and production coupled with acceptable level of water related risks is imperative.

In Ghana, there are a number of river bodies that are accessed by both rural and peri-urban dwellers for consumption and other domestic purposes. Water drawn from these river bodies are used for cooking, drinking, washing clothes to mention a few, however, they are unsafe and pose many health challenges to users (UNICEF/WHO, 2012; Sekyere, 2013). In an attempt to treat water and make it safe for use, Von-Kiti (2012) and

Sekyere (2013) produced zeolite for the desalination of sea water. Zeolite have been found to be an alternative to the desalination and purification. Although their study was successful, their findings pointed to the fact that in subsequent studies, more ceramic materials should be developed in producing zeolite if a more favourable results is desired. Zeolites are used because of their capabilities of ions interest. Studies have shown that they have the abilities to function in pH values above 12.0 and temperature regions of 650^{0}C necessary for cost and energy effectiveness and this is due to the large surface structure and the porosity attribute which can encapsulate and adsorb (Belviso, et al, 2009; Sean, 2009).

Since zeolites were discovered, the industries such as the oil and gas, health, etc. have been employing zeolites for purification and desalination purposes. Even with flowing water from pipes, the Ghanaian Daily Graphic on May 16, 2016 published Centre for Scientific and Industrial Research report revealing that our water supplied by Ghana Water and Sewage Corporation is not totally safe. The cost of treating water according to Sekyere (2013) was expensive due to the worsening condition of the quality of raw water and the high cost of chemicals. The activities of "galamsey" (local parlance for illegal mining) has been one source of water pollution in Ghana (Mustapha, 2013).

Ghana is endowed with the availability of raw materials such as clay, kaolin that can be appropriately worked on in purifying these unsafe water sources. What is needed therefore is appropriate technology in design and the art to turn these raw materials in producing inventions that would be of immense help to the society. The study seeks to explore means of synthesizing zeolite from kaolin with NaOH concentrations through crystallization process, make a body to develop candle filters and subsequent zeolite filter unit and assess its effectiveness through purification of water from open sources.

2.0 Methodology

2.1 Processing of Materials and Reagents

The following reagents were also used for the study; Kaolin, Fosu clay, distilled water, sodium hydroxide (NaOH) pellets and starch. Kaolin was used as the main constituent of the zeolite synthesis. This was obtained from TelekuBokazzo in the Western Region, Ghana. It was mined in powdered form and subsequently sieved through fine mesh of 3 microns for finess and uniform particle size distribution. The Fosu clay was acquired in the raw rocky state from Fosu (a town in the Central region of Ghana) crushed with the primary jaw and the gyratory crushers and finally grinded into fine particles with the pulverizer. It was sieved through a fine silk mesh of 3microns and introduced into a batch as ball clay to give plasticity to the zeolite and kaolin which do not possess any plastic properties for plastic forming.

Distilled water under standard purification method was acquired from the Centre for Scientific and Industrial Research, Ghana and Sodium hydroxide in the form of pellets, was acquired from Pakus chemicals in Kumasi, Ashanti Region, Ghana. The pellets were grounded into powder with the aid of mortar and pestle to aid dissolution in water. Starch was obtained from cassava dough, which was mixed with water and heated to form a gel- like or thixotropic mixture. The thixotropic mixture was added to the batch as a combustible material to enhance the porosity of the final candle filter. The operating temperature for these processes was 1000^{0}C.

In order to produce the filter, Plaster of Paris (P.O.P) was used in the preparation of a one piece mould to create a model of the filter candle. The model was then used to produce the mould by mixing 1 part of P.O.P to 2 parts of water by sifting the P.O.P into the water until it breaks the surface of the water. It was stirred vigorously and poured slowly over the model which was well set in a cottle on a leveled table with all visible spaces blocked with clay to prevent any spillage.

2.2 Synthesis of Zeolite from Kaolin

The synthesis of zeolite from kaolin involves two processes. (i) The thermal pre-activation of kaolin to obtain dehydroxylated X-ray amorphous product called metakaolin (ii) hydrothermal reaction of metakaolin with aqueous alkali.

In the first process, 4kg of kaolin was weighed and sieved through a fine silk organdy mesh. The kaolin was hand mixed with a calculated amount of water and the mixture was made into noodles and left to dry for 48 hours. The formed kaolin noodles were placed in fired unglazed ceramic containers to facilitate even heat distribution during calcination. They were calcined at 650^0 C for 2 hours to loose structural water and reorganization of the molecular structure to obtain a more reactive phase (metakaolinite). The caked nature of the metakaolin was dismantled with the pulverizer after calcination to obtain fine particles.

During the second process, NaOH solution with 3M concentration was prepared by grinding NaOH pellets into powder with the mortar and pestle, and dissolved in 100ml of water in a volumetric flask fixed using the magnetic stirrer. 50g of metakaolin was weighed and added to the solution and stirred vigorously with the magnetic stirrer for an hour. The resulting mixture was allowed to age for 24 hours. Crystallization was carried out by hydrothermal synthesis under static conditions at 100°C for 4 hours in an oven.

Once the activation time was reached, the beaker containing the sample was removed from the oven and quenched in cold water to stop the reaction. The sample was grinded to loosen up its caked form after crystallization. The solid mixture was washed thoroughly with distilled water to remove excess alkaline present in the sample and filtered with the whatman filter paper and distilled water until the pH of the filtrate tested neutral or almost neutral. The sample was then dried in an oven at at temperature of 80^{0}C for 3 hours.

2.3 Characterization of the Zeolite Sample by X-Ray Diffraction (XRD)

XRD method was used for the identification of crystalline materials in the zeolite produced. It was used as means of characterization to identify and analyze the individual phases present in the zeolite sample. This was achieved by applying elastic scattering of X-rays on structures to acquire information on unit cell parameters of the product (Kulprathipanja, 2010). By measuring the angles and intensities of these diffracted X-ray beams, X-ray diffractometer produced a pictorial view of the density of electrons within the crystal. The two-dimensional image can be converted into a three-dimensional model of the density of electrons within the crystal using the mathematical method of Fourier transforms (Sekyere, 2013).

2.4 Development Of The Zeolite Candle Filter (ZCF)

The raw materials used were in the following percentages for sample A and B:

Table 1:

Percentage batch compositions of Zeolite Candle Filters

Materials	Sample A (%)	Sample B (%)
Zeolite	40	30
Kaolin	35	30
Fosu clay	15	30
Starch	10	10
Total	100	100

Mixing of the raw materials of both samples were done thoroughly by hand with appropriate PPE such as nose mask, gloves and overall coat. The materials were mixed with water and starch gradually until a homogenous mixture was obtained. The mixture had little plasticity as a result of the zeolite and kaolin which have little or no plasticity. The half dried mixture of both samples were hand kneaded to remove all air bubbles. The mixture was pressed and scooped to get the desired shape using a Plaster of Paris (P.O.P) mould. The formed shape was left in the mould for 4 hours until the candle separated from the mould. The formed candles from both samples were air dried at room temperature to remove most of the physical water. After sufficient drying, the samples were fired to a temperature of 900^{0} C which was enough for the combustible material to burn off and also improve the strength and durability of the candles. After firing, the kiln was cooled slowly until the candle could be handled.

2.5 Assembling and Operation of the Filter Unit

The filter unit is made up of upper bucket with a lid, a ceramic candle, a candle spout, a lower bucket with a lid and a water spout (outlet). The candle was glued to the candle spout and then screwed to the upper container through the holes made in the upper bucket and the lid of the lower bucket as assembled in Fig. 1. Contaminated water fetched from Weweriver (a river that runs through the KNUST Campus and accessed by surrounding towns) was poured into the top container where the candles are attached. The water passes through the pores in the candle and drains into the lower container through the candle spout. The filtered water is stored in the lower container protecting it from recontamination.

Fig. 1: Assembled Filter Unit

3.0 Results And Discussion

3.1 Synthesis and Characterization of the Zeolite

Fig 2 shows the synthesis and the crystallization pattern of the zeolite. The synthesized zeolite was very fine in size and cream in colour as seen in Fig 2. The crystallization pattern was observed for the sample and was identified as zeolite type A. With a crystallization time of 4 hours and 3M concentration of NaOH, zeolite was produced in huge quantities for the development of the ceramic filter candles. XRD analysis was carried out on the synthesized zeolitic sample to monitor the phase purity and crystallization. The XRD pattern revealed Zeolite Linde Type A (LTA) with a cubic crystal system. The XRD pattern were in accordance with International Center for Diffraction Data (ICDD), a standard reference pattern as cited in Kwakye-Awuah (2008). High reflection peaks was observed in the samples' diffraction pattern as shown in Fig 3.

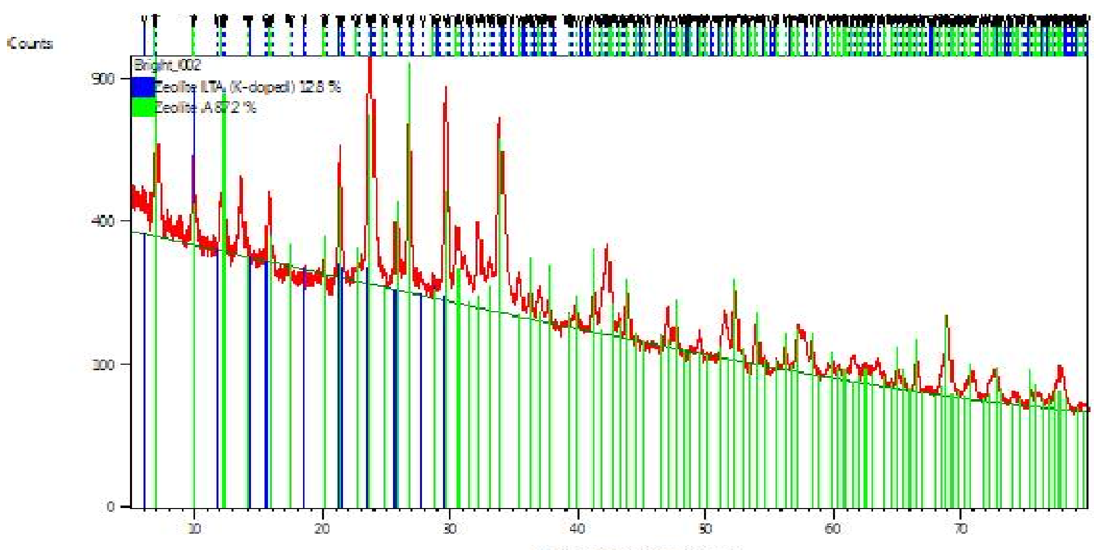

Fig. 2: Standard reference pattern

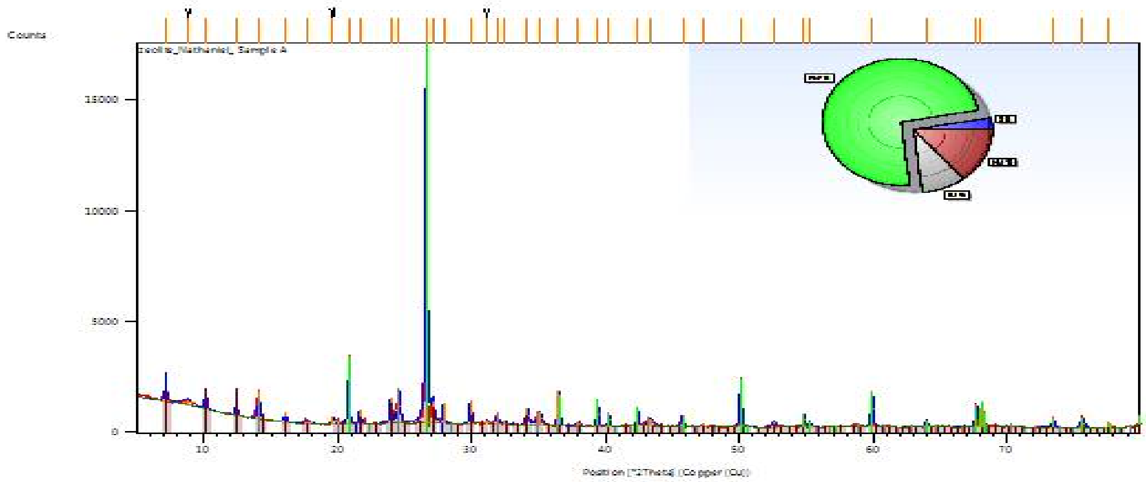

Fig. 3: Diffraction pattern for Zeolite sample

Tests on Zeolite Candle Filter

Series of physical and microbial tests were performed on the developed candle to test for its efficiency as a filter and the results presented on Table 2. These tests were performed before filtration and after filtration of the polluted water through the developed zeolite filters.

For the turbidity test, the unfiltered and filtered turbidity readings were compared to determine turbidity removal. Turbidity reading for the unfiltered water was 22.13 NTU whilst turbidity reading for the filtered water was 1.96 NTU. For the pH test, the reading for the unfiltered water was 7.16 compared to 7.6 after filtration through the zeolite candle.

Table 2:

Water Quality Test Results for Unfiltered and Filtered Water using the Zeolite Candle Filter

WEWE RIVER – TEST	BEFORE FILTRATION	AFTER FILTRATION	WHO STANDARDS
PH	7.16	7.6	6.5 – 8.5
Total Dissolved Solutes	155ppm	140ppm	<500
Electricity Conductivity	310u/cm	281u/cm	<600
Turbidity	22.13 NTU	1.96 NTU	1 NTU
Colour	60	40	<15
Calcium Hardness	4.2	1.7	<75
Total Hardness	3.5	2	<300
Chloride	37.99	59.87	<250
E-Coli	0	0	0
Salmonella	2^{10}	6×10^{2}	0

The colour test reading of the unfiltered water was 60 and after filtration through the candle, the colour was reduced to 40. Calcium hardness for the unfiltered water was 4.2 and after filtration the calcium hardness was reduced to 1.7 whereas the total hardness reading for the unfiltered water was 3.5 and reduced to 2 after filtration of polluted water through the developed candle.

For total coliform removal, the membrane filter technique was applied for the removal of the total coliform. After 18 hours of incubation, the number of salmon to red colonies was recorded as coliforms by visual examinations and no E.coli was present in the unfiltered water. The numbers of coliforms present in both the unfiltered and filtered water were compared to assess the microbial effectiveness of the zeolite filters. The result showed a high concentration of coliform present in the unfiltered water. The filtered water showed a reduction in the number of coliforms present prior to filtration. However, Dies (2003) revealed that the incorporation of colloidal silver in ceramic filters such zeolite filters can aid in the total removal of coliforms. Coloration of the membrane filter paper used for the filtered sample was observed after 18 hours.

3.2 Pilot Study on the Zeolite Ceramic Filter

The zeolite ceramic filter was piloted in order to confirm results from the filtered and unfiltered water. Using random sampling technique, three semi-detached house units in a rural setting were selected for this pilot study. The household of these housing units were trained on how to set up the zeolite ceramic filters. The zeolite ceramic filters were set up on the compounds of these housing units who fetch water from the river and stream around them. The pilot study was up to 12months. The following were the outcomes from the pilot study of the user of the zeolite ceramic filter invention:

1. Results showed that the users of the filtered water from the zeolite had less cases of diarrhea. Between 2009 - 2013, 51,131 cases of diarrhea were reported with 55.2% being females over the five year period. The highest episode of diarrhea by age-group occurred in children under-five during the study period (Peprah, et al, 2016).
2. Users of filtered water from the zeolite candle had fewer cases of cramps, nausea and vomiting.
3. Physical observational on the users of the zeolite candle showed good skin growth and limited skin rashes and other dermatological diseases.

3.3 Synthesis of Zeolite

As noted in this study, the synthesized zeolite after characterization showed a type A which proved effective in the river water filtration test. However, different types of zeolite exist, each with a unique structure as well as composition controlling properties. During metakaolinization, the kaolinite structure is destroyed and the organic content is reduced to a great extent (Sekyere, 2013). During the calcination, the samples were fired at 650^0 C and soaked for 2 hours within which the individual phases present in the sample were stabilized. The iron content got oxidized leading to coloured ferric compounds, thus reducing the brightness of the metakaolin.

It was observed that the synthesized sample became slippery when it was wet and it could be due to the release of OH into the system from the NaOH solution added during the crystallization process. The slipperiness of the sample was due to the presence of NaOH and contributed to the high pH value of around 10. This was reduced to 8 by washing the sample several times using distilled water and Whatman filter paper for filtration.

3.4 Characterization

The results from XRD indicated diffraction patterns of the sample which compared to findings from Sekyere (2013), Von-Kiti (2012) and Smaihi et al (2003) confirmed a corresponding diffraction pattern of Zeolite A. The test also revealed that the sample had a cubic crystal system or structure. Sharp peaks which were observed confirmed full crystallization and the nature of the peaks also confirmed the sample as polycrystalline materials. The diffraction pattern of the sample in Fig 3 indicates high peak which shows the full rate of crystallization and other dislocations. It was recognized as a type of zeolite Linde Type A (LTA) which was crystallized in the oven for 4 hours. According to Smaihi et al, (2003), further hydrothermal treatment does not influence the morphology nor the crystallinity of the product and hence, a zeolite material cannot be further synthesized hydrothermally to change or alter the morphology.

3.5 Development of the Zeolite Candle Filter

The synthesized zeolite had little or no plasticity and as such, other clays (binders) were added to enhance the plasticity to help in the forming and shaping of the candle. Two test candle samples A and B were produced using different batch as indicated in Table. 1. Sample A had little plasticity when compared to the sample B due to the use of more non-plastic materials precisely kaolin and zeolite. The increment in the non-plastic materials for sample A resulted in increment in porosity when compared to sample B. Starch which is a combustible material was added to aid in the binding of the materials and also to improve the porosity of the candle. The mould developed from the model was used in producing the candle. The wet batch was pressed manually into the P.O.P. mould and scooped to obtain the candle. This manual pressing and scooping is easy and cost effective compared to other forming methods such as jolleying which require the use of electricity and special templates. The presence of the zeolite makes the pores uniform and reduces their sizes from micro to nanometer hence increasing filtration rate whilst effectively sieving contaminated water sample (Sekyere, 2013).

3.6 Performance of Zeolite Candle Filters

Series of physical and microbial tests were performed using the developed candle filters. River water was tested before filtration and after filtration. The tests were performed so that comparison could be made between the unfiltered water, and after it had been filtered using the developed filters to assess how effective the filters were in the removal of bacteria and physical parameters such as colour and pH.

The pHs for the unfiltered and filtered river samples were recorded to be 7.16 and 7.6 respectively. As such, the value of the pH after filtration falls within the standard pH range by World Health Organization (W.H.O.). Total Dissolved Solutes (T.D.S.) reading for the unfiltered water was 155ppm and after filtration using the developed candles reduced to 140ppm. Electrical conductivity (E.C.) also reduced from 310u/cm to 281u/cm before filtration and after filtration using the developed zeolite candle filters. T.D.S. and E.C. values after filtration fell within the standard values set by the W.H.O.

There was improvement in colour as the reading for the unfiltered river sample reduced from 60 to 40 after filtration using the developed candles. Though there was improvement in the colour after filtration it did not meet the standard by W.H.O. Calcium hardness was 4.2 before filtration and this was reduced to 1.7 after filtration. Total hardness value for the unfiltered river sample was 3.5 and reduced to 2 after filtration. There were significant improvement in the calcium and total hardness of the water. In percentages, they are 56% and 43% respectively.
Test that are very critical and important when it comes to the certification of safe water for consumption such as turbidity, e-coli, salmonella and total coliforms were also tested to assess the performance of the filters in the bacteria removal. Turbidity reading for unfiltered river sample was 22.13 NTU and was reduced drastically to 1.92 NTU after filtration. The recorded value could not meet the standard but there was a significant improvement in turbidity removal. This improvement as expressed in percentage was 96%. The high turbidity reading for the unfiltered sample indicated that the river sample was very muddy or cloudy and as such the developed candle would almost be 100% effective in turbidity removal when a less muddy or cloudy sample is used. E-coli was absent in the unfiltered water and as such there was none present in the filtered water. Salmonella value for the unfiltered river sample was 2^{10} and was reduced significantly to 6×10^{2} indicating the zeolite filter's effectiveness in reducing salmonella.
The numbers of coliforms present in both the unfiltered and filtered water were compared to assess microbial effectiveness of the zeolite filters. The results showed a high concentration of coliform present in the unfiltered water. There was a significant reduction in the number of coliforms present after filtration using the developed zeolite filters. However, Dies (2003) laid bare the fact that the incorporation of colloidal silver in ceramic filters such as zeolite candles can aid in the total removal of coliforms.

4.0 Conclusions

The main focus of the study was to synthesize zeolite to produce water filters to purify unsafe water for domestic and consumption purposes. The study has successfully produced a Zeolite LTA and capable of purifying unsafe water for rural dwellers. The production was been done with locally available material and its effectiveness assessed by the purification of Wewe river and piloted in a rural settings. The following conclusions were drawn:

1. There was significant improvement in physical test such as colour, pH, total hardness and total dissolved solutes.
2. Even though turbidity was reduced by 96%, it could not meet the WHO standard.
3. The zeolite water filter produced is capable of improving the quality of water used by rural dwellers.

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