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Efficiency of Ceramic Composite Filter Produced Using Nano Particulate Carbonaceous Material

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Abstract: *Millions of people have no access to safe water. This has led to continued incidence of waterborne diseases with severe cases of complications and sometimes death. Attempts to use silica sand-based materials for the production of filters by researchers are still evolving. In this study a novel hybrid clay-nanoparticulate agro-waste blend water filter with appropriate pore size and flow rate for contaminant-free water filtration was produced. The composite filters were produced using hydraulic pressing and sintering process. Four different ratios of clay, activated carbon and nanoparticle coconut shell (60:30:10, 60:20:20, 60:10:30, and 60:0:40 wt %) were mixed and fired at 700 °C, 750 °C, 800 °C, 850°C, 900 °C, 950 °C, and 1000 °C respectively at the rate of 75 °C/hr. E. Coli, Total Coliform, and turbidity tests were carried out on the influent water collected from Lagos Lagoon, and effluent water samples were filtered using the produced samples. The result of water tests revealed that all the filters produced removed between 86.81 % to 99% Escherichia Coliform (E. coli), and 81.81% to 93.31%% total coliform in the water sample. The blend of 60:10:30 fired at 850 °C and 900 °C showed improved properties with a flow rate value of 2.83 l/hr and 2.77 l/hr. In conclusion, the study established that synthesis of nanoparticle coconut shell and activated carbon is a suitable material for producing clay composite filters that can purify water to Nigerian Standards for Drinking Water Quality (NSDWQ) acceptable level.*

Keywords: *Ceramic Composite, Coconut Shell, Nano-particle, Water Filter, Water Treatment*

1. INTRODUCTION

Eight out of every ten people lacking access to supply of basic drinking water lives in rural areas and sub-Saharan Africa has 70 % of its population still in need of a safe drinking water supply [1]. To this extent, research around water treatment and purification is required, particularly in rural areas of developing countries like Nigeria having more than 63 % of its population residing in rural areas and only about 4 % of that population has access to safe and affordable drinking water supply [2,3]. Among the less than 4% that can assess safe drinking water, most must transport their water from the source to their various households and in the process the water can become contaminated [4,5,6] which reveals the need to consider and evaluate clean and safe drinking water source especially at household level. Most agricultural activities are carried out in the rural areas. These activities involve the use of fertilizers, pesticides and other additives which can find their way to the water source(s), and they are harmful to humans [7,8,9]. Rural areas in Nigeria have bigger issues when it comes to availability of drinkable water due to the rate of poverty and lack of supporting infrastructures like electricity. Identifying effective and reliable technologies to eliminate faecal contamination from water remains a significant challenge in ensuring safe drinking water [10,11]. While modern drinking water systems, such as community tap water or in-house piped water, are ideal for long-term solutions, there are difficulties [12] in establishing and maintaining these systems, particularly in rural areas.

Methods of treating water at the point-of-use (POU) have proven to be effective to a good extent in making water available at household level. Presently, there are lots of promising POU water treatment methods, amongst which are; CCWF [13,14], chlorination [15,16], bio sand filters [17], flocculation [18]. The above-mentioned methods of water treatment have been tested and proven effective in laboratory environment, but their real effectiveness is put to test in household conditions where they are subjected to consumer use conditions (maintenance, hygiene, operation, and so on) [19]. For instance, the use of disinfectant powders has a short lifespan, use of flocculent is cumbersome as it requires lots of steps [20], chlorination affects the odor and taste of the water [15,16,18]. Other POU water treatment technologies are ultraviolet irradiation/ solar disinfection, membrane filtration, ion exchange, biological filtration, and candle filters among others [21-24], most of which are gradually dating out except for chlorination and candle filters.

Point-of-use (POU) filtration systems provide a viable solution for delivering safe drinking water to rural communities [25]. These systems are designed to prevent contamination during water transit. One notable example of a POU filtration system is the composite clay-based water filter (CCWF) [14,26,27,28,29], which is the focus of this study. CCWFs are produced using locally available materials such as clay, sand, and burnout materials, making them an effective means to reduce waterborne diseases. The materials used in ceramic water filters can also include water, grog, laterite (soil rich in iron oxide), and bone char, along with the primary components (clay and carbonaceous material). Clay in its plastic state, can be moulded easily without breaking [30]. Ceramic filters can be crafted in various shapes, such as flowerpots, discs, and candles. They are affordable, accessible, and practical technologies that enable households, schools, and workplaces to manage their drinking water quality and are effective in treating common drinking water contaminants, including biological pathogens and general macro contaminants [31,32,33].

Given the poor quality of available drinking water sources and the lack of centralized water treatment systems in Nigeria, it is essential to explore low-cost and efficient household water treatment methods. CCWFs show promising potential in this area. Consequently, this paper aims to explore novel material blends for the production of CCWFs under various conditions and their resulting water treatment efficiency level.

2. EXPERIMENTAL PROCEDURE

2.1 Materials Sourcing and Sample Preparation

Clay materials (Figure 1a) was sourced from Clay Company, Oregun and dried in the oven at 150 °C to remove all moisture before being pulverized. The coconut shell was obtained from Lagos market, it was then washed, sundried, crushed, pulverized and milled (Figure 1b). Lemon was bought from the market and it was manually juiced and mixed with water in the ratio 1:3 (Figure 1c).

(a) (b) (c)

Figure 1: (a) clay material (b) Pulverized coconut shell (c) lemon juice mixed with water.

The pulverized clay was sieved and sizes below 70 µm were kept for further work. The coconut shell was milled to nanoparticle size with obtained average size of 28.16 nm after 16 hours of milling using a planetary ball mill (JC – QM – 2; JC2021 - 072367). Some of the pulverized coconut shells were carbonized at 800 ^{o}C, for 4 hours [34] as shown in Figure 2a and was allowed to cool in the furnace. Carbonization was carried out in a heat treatment furnace (SXL – 1208; 1905741). The carbonized coconut shell was then mixed with the lemon water juice and kept in an air-tight container for 24 hours (Figure 2b) before being washed dried and sealed in a container for use.

(a) (b)

Figure 2: (a) Carbonized coconut shell in a lined steel box (b) Carbonized coconut shell soaked in lime juice for 24 hrs

The Clay, activated carbon, and nano particulate coconut shell blend was mixed in four different blends; 60:30:10, 60:20:20, 6:10:30, and 60:0:40 wt%. A typical 60:30:10 mix will contain 60 g of clay, 30 g of activated carbon and 10g coconut shell. Water added to the blends is 10% of the total composition in g. The mixed blends were then weighed into 50g for each sample and rammed using a rammer. The rammed samples have diameter of 5.0 cm and height of about 1.6

cm. The rammed samples were sintered in the furnace at different temperatures of 700 ^oC, 750 ^oC, 800 ^oC, 850 ^oC, 900 ^oC, 950 ^oC, and 1000 ^oC respectively. The sintering process caused the burning out of carbonaceous material in the blend (that is NPCS) leaving pores in the samples. After firing the sample diameter became 4.8 cm and height was 1.5 cm with varying weights between 38.80 g to 39.90 g (Figure 3).

Figure 3: Sintered filter samples

Table 1: Composition of clay composite filter samples produced

			Temperature (°C)						
			1000	**950**	**900**	**850**	**800**	**750**	**700**
			1	**2**	**3**	**4**	**5**	**6**	**7**
Composition	**A**	60Cl:30AC:10NPCS		60:30:10	60:30:10	60:30:10	60:30:10	60:30:10	60:30:10
	B	60Cl:20AC:20 NPCS		60:20:20	60:20:20	60:20:20	60:20:20	60:20:20	60:20:20
	C	60Cl:10AC:30 NPCS		60:10:30	60:10:30	60:10:30	60:10:30	60:10:30	60:10:30
	D	60Cl:0AC:40 NPCS		60:0:40	60:0:40	60:0:40	60:0:40	60:0:40	60:0:40

NB: Cl is clay; AC is Activated carbon; NPCS is Nano Particle coconut shell

2.2 Characterization of Material

Malvern PANanalytical XRF spectrometer with software version 10.3.0.159 was used at 20kV and 25 mA to determine the elements and their corresponding concentration in the clay sample. Using CuKα radiation (α = 1.5418Å) at 34 kV and 25 mA, the X-ray diffraction patterns of the clay sample was examined using a "X-PertPro PANalytical, LR 39487C XRD diffractometer" in a 2θ angular range between 10 and 90 degrees, with measurements made every 0.04^o for 6 s.

2.3 Flow Rate Measurement

The filters were fitted into plastic containers as can be seen in Figure 4a. The flow rate was tested by pouring distilled water into a media that the filter has been worked into and leaving the water to flow through the filter uninterrupted (Figure 4b). The effluent water was then measured in liters using a measuring cylinder. Flow rate calculation is given in liters per hour, which means that it can be calculated using equation 1 [33];

$$\text{Flow Rate (FR)} = \frac{quantity\ of\ effluent\ water\ (Q)}{time\ taken(T)} \quad (1)$$

(a) (b)

Figure 4: (a) Filter samples fixed in plastic containers (b) typical flowrate testing process

2.4 Water Tests

Escherichia Coliform and Total Coliform tests were carried out using Eosin Methylene Blue (EMB) agar and McConkey agar respectively. The pour plate method [35] was used for the measurement of both E. coli and Total Coliforms. Samples were incubated at 370C for 48 hours after which readings were taken in cfu/mL.

The turbidity of the samples was obtained using the Hach dr600. 10mL samples were dispensed into cuvettes and placed in the equipment, and turbidity values were read off the LCD display of the equipment.

The equation used in calculating percentage removal of the contaminants from water is given in equation 2 [36]

$$\% \text{ Removal Efficiency} = \frac{Composition\ before - \ Composition\ after}{Composition\ before} \times 100 \quad (2)$$

3. RESULTS AND DISCUSSION

3.1 Clay Type Characterizations

The XRF of the clay sample showing its elemental composition is given in Table 2. This result indicates an alumino-silicate, clay type which falls under the illite clay mineral group. This result was further confirmed by the XRD results and is comparable to the results of Gaudette [37]. This clay type is preferred due to its weak shrinking and swelling ability causing it to maintain the product dimensions with temperature change.

Table 2: XRF elemental analysis of clay sample

Elements	**Si**	**Al**	**Ca**	**Mg**	**Mn**	**Fe**	**K**	**Cl**
Composition (%)	65.30	15.70	1.50	0.94	0.02	7.67	0.29	0.07

The result shown in Figure 4 revealed that the clay is characterized by two major peaks which are the Quartz (SiO_2) and the Aluminium Silicate (Al_2SiO_3) at diffraction angles (2Ө) 26.9643° and 24.38° and interplanar distance of 3.31 and 3.65 respectively which confirmed the assertions made by the XRF result of a possible illite type clay [37]. This describes the crystalline nature of the clay. Table 4.2 shows the distinctive list of identified phases which is comparable to the results obtained from the XRF of the clay.

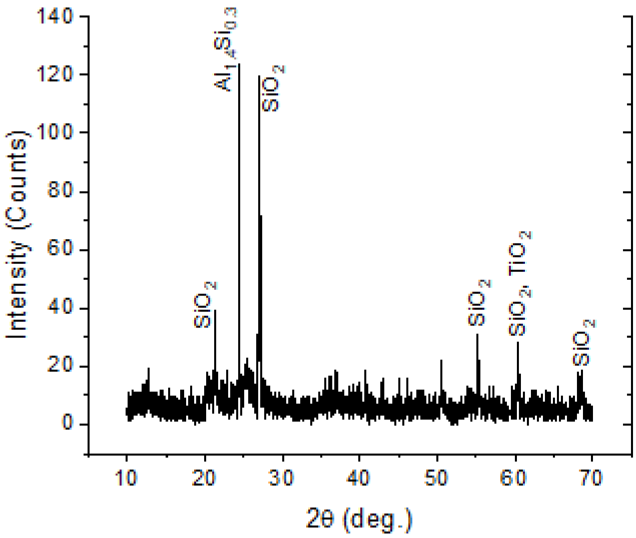

Figure 4: XRD profile of Clay sample

3.2 Flow Rate of Clay Composite Filters

The flow rate of the filter samples is shown in Figure 5. There is a significant increase in the flow rate with increasing temperature and an increase in the wt% of Nano particulate coconut shells (NPCS). The ratio of coconut shell in the mix has a significant effect on the flow rate of the material which is consistent with other studies [28,36,38,39,40] where the increase in composition of carbonaceous material led to an increase in the flow rate. The main justification for adding burnout materials in ceramic water filters is to increase the filtration rate, while achieving an elevated level of treatment. Sample 1A has a flowrate of 2.12 L/hr while sample 7A has a flow rate of 0.17 l/hr. The sample A with blend ratio of 60 wt% clay, 30 wt% Activated carbon and 10 wt% NPCS supports the reduction in flow rate with reducing firing temperature from 1000 °C to 700 °C. For the filter samples A and D, peak flow rate was achieved at firing temperature of 900 °C and samples B and C peaked at 850 °C. This indicates that filter blends of clay, coconut shell and activated carbon does not necessarily need to be fired to 1000 °C before optimal flowrate can be achieved which explains why most literatures have a maximum firing temperature of 900 °C [2,41]. The whooping increase in the flow rate of filter samples fired at 800 °C upwards compared to those fired at 700 °C and 750 °C shows that the NPCS is still present in large quantities at these temperatures. It is therefore logical to indicate that more pores are available in the filter as the NPCS contents in the filter composition increase and as the firing temperature increase up to 900 °C. The reduction in flow rate for filters fired after 900 °C was explained by Zereffa and Belako [41] to be caused by reduction in grain size with further increase in temperature.

The size of carbonaceous material used in this study is in Nano particle size range which indicates smaller pore sizes are distributed in the samples. The smaller the grain size, the lesser the flow rate according to Soppe *et al.* [33] but this is not the case in this study as the flowrate obtained by the filter samples at various temperatures are comparably higher than those in other studies [36,41,42]. The relatively higher flow rate obtained in this study can be attributed to the presence of activated carbon in the blend. This was corroborated by Erhuanga *et al.* [43] where charcoal was added to the blend and this led to an increase in the porosity and ultimately increase in flow rate.

Figure 6 shows a clear comparison of the increase in flowrate obtainable with increasing temperature of sample A. This shows the distinct effect of increasing temperature on flowrate of a given filter blend.

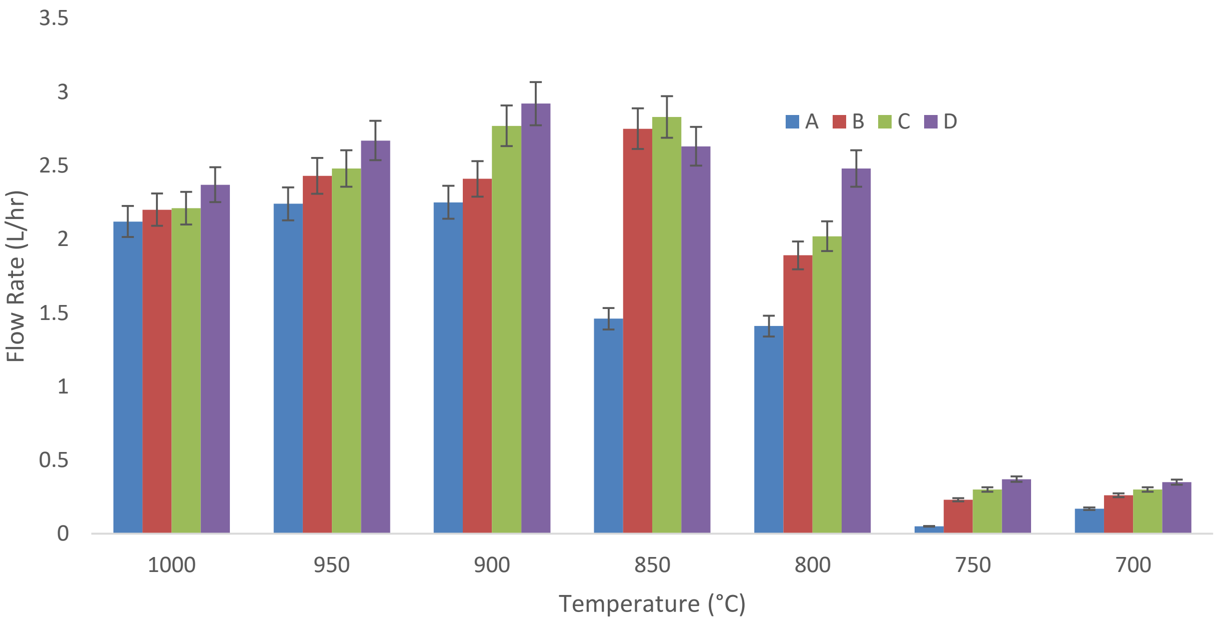

Figure 5: Result of flow rate test carried out on the sample clay composite filters

3.2 Water Tests

A significant obstacle to bacteria is the surface of the ceramic disks; it has the highest portion of E. coli stored. The result obtained from the test of E. coli, total coliform, and Turbidity of the control influent water (Lagoon water) is 11 cfu/ml, 16 cfu/ml, and 23 NTU respectively. The results (Table 3) showed that most of the sample filters produced water within the Nigerian Standards for Drinking Water Quality, (NSDWQ) requirements [44]. About 75 % of the filter samples can remove more than 90 % E. coli from the water sample. The same goes for the removal of Turbidity. In accordance with literature [33,36,37] there is a reduction in the filter efficiency with an increase in the composition of carbonaceous material.

The result from the study of Ajibade *et al.* [40] for total coliform and *E. coli* removal ranges from 12 % and 4 % for 30:70 clay to sawdust ratio to 100 % for 90:10 clay to sawdust ratio, respectively. This was also supported by Bulta and Micheal [36] who reported that the microbial removal efficiency of CCWF increases with increase in the percentage composition of clay and firing temperature which is contrary to the results obtained in this present study. Where the temperature of firing has little to no effect on the removal efficiency of the filter samples. The very little to no significant effect noticed with increase in quantity of carbonaceous material could be attributed to the use of carbonaceous material in the Nano particle size range which will invariably cause the pores in the filter samples to be small and maintain the efficiency of the filters in microbial and contaminants removal. The smaller the particle size of carbonaceous material, the smaller the pore size of the produced filter [33]. Bacteria microorganisms have a size range between 0.3 to 60 microns [45] which is still much bigger than the nanoparticle pore sizes that will be left in the filters after firing.

The E. coli removal efficiency of the filters range from 86.81 % to 99% and total coliform values are between 81.81% to 93.31%. Turbidity removal of the filters was between 87.04% and 95.07%. The removal efficiency of E. coli is higher than that obtained for the total coliform for the filters.

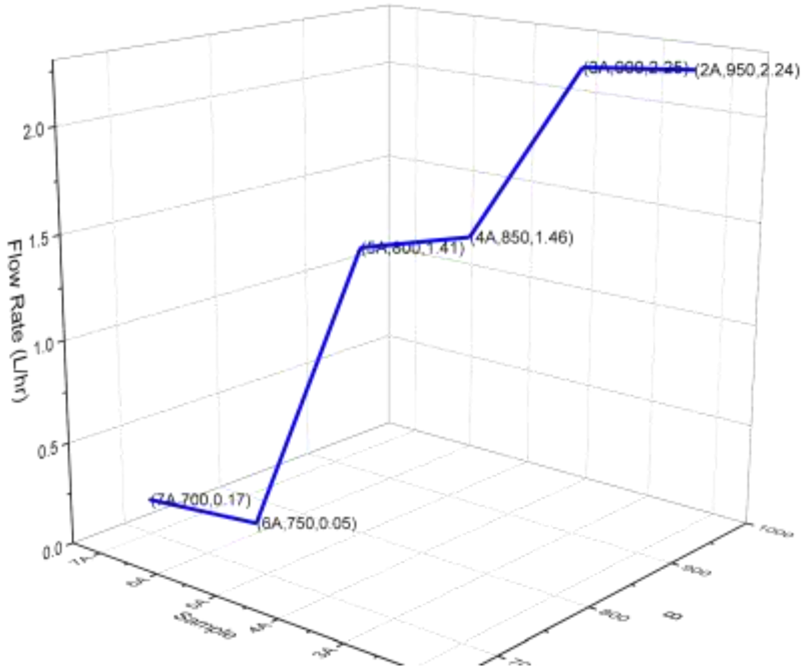

Figure 6: Comparing trend in flow rate of sample A fired at different temperature

Table 3: Results of water purification by filter samples

	Clay	Activated Carbon	NPCS	Temperature (°C)	*E. coli*	Total Coliform	Turbidity
1A	60	30	10	1000	97.18	87.43	89.39
2A	60	30	10	950	96.27	87.00	89.47
3A	60	30	10	900	98.00	90.56	90.56
4A	60	30	10	850	98.63	90.50	91.65
5A	60	30	10	800	97.54	89.63	92.26
6A	60	30	10	750	96.00	89.19	93.83
7A	60	30	10	700	95.64	88.18	95.26
1B	60	20	20	1000	91.91	87.50	89.57
2B	60	20	20	950	93.36	87.75	89.96
3B	60	20	20	900	95.27	93.81	94.83
4B	60	20	20	850	97.18	93.50	95.87
5B	60	20	20	800	96.18	92.69	95.39
6B	60	20	20	750	95.36	89.56	94.87
7B	60	20	20	700	95.18	88.19	94.39
1C	60	10	30	1000	91.91	87.94	90.62
2C	60	10	30	950	93.91	88.31	90.83
3C	60	10	30	900	99.00	90.56	94.26
4C	60	10	30	850	98.18	88.50	95.43
5C	60	10	30	800	95.55	88.13	94.52
6C	60	10	30	750	96.09	88.81	93.22
7C	60	10	30	700	96.18	90.25	94.30
1D	60	0	40	1000	88.00	81.31	87.04
2D	60	0	40	950	89.91	84.19	87.96
3D	60	0	40	900	86.73	83.31	88.35
4D	60	0	40	850	89.55	83.18	89.09
5D	60	0	40	800	89.00	88.50	89.22
6D	60	0	40	750	86.81	89.18	89.74
7D	60	0	40	700	91.36	89.75	89.91

The filter sample 3C displayed very good flow rate coupled with the obtainable high removal of microbial and physical contaminants from water. Other water tests like iron, nitrate and total dissolved solids were tested on the water filtered by sample 3C and the result is compared with the NSDWQ and control data as in Figure 5. There is a visibly lower value of physicochemical and microbial contaminants in the water filtered by sample 3C.

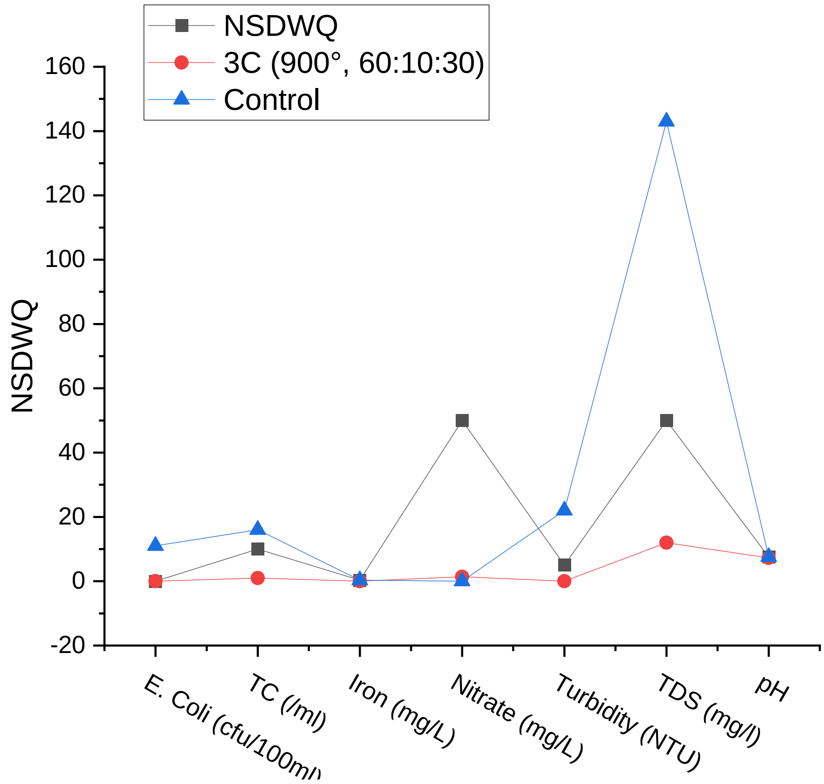

Figure 5: Water test of filter sample 3C in comparison with the NSDWQ and control (Lagoon water)

4. CONCLUSION

This study has produced a clay composite filter with different blends of clay, Nano particulate coconut shell and activated carbon and different firing temperatures. The microstructural, and physical characteristics of developed filters were carried out and the efficiency of the filters was tested by carrying out flowrate and microbial test on the water samples. The following conclusions are drawn from the results:

1. The study revealed that there is an increase in flow rate with increase in the wt% addition of Nano particle coconut shell due to the increase in the number of pore spaces as the carbonaceous material burns out of the blend.
2. The study revealed an increase in the flow rate with increasing temperature up to 900°C followed by a decrease in flow rate with further temperature increase which indicates that the maximum required firing temperature for a CCWF is 900°C.
3. The addition of activated carbon to the blend of clay and coconut shell for the production of filters for water filtrations showed effectiveness with 10% addition, while further additions did not significantly reduce the contaminant removal efficiency of the filter.
4. The use of Nano particle coconut shell instead of the conventional micro particle size carbonaceous material caused the high *E, coli,* total coliform and turbidity removal of between 87 % to approximately 99 % even at increased wt% addition. This means there is no trade-off between the flowrate and efficiency of the filter samples as is the case with existing ceramic filters.

It can therefore be concluded that the use of a Nano particle carbonaceous material for producing ceramic filter erases the need for balance between flowrate and contaminant removal efficiency of the filters. The addition of activated carbon to the blend is believed to increase the efficiency of the filter in a way because AC are good chemical adsorbents. It is therefore recommended that future research focus on testing the efficiency of the filter to removing chemical contaminants in water.

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