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Fabrication of porous ceramic pot filters for adsorptive removal of pollutants in tannery wastewater

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ABSTRACT

The use of porous ceramic pot filters for wastewater treatment is considered a cost-effective technology however presence of appropriate pore size to trap pollutants and microorganisms from industrial wastewater remain a challenge. In this study, naturally available kaolin was first characterized and porous ceramic pot filters from different ratios of the kaolin to sawdust which served as combustible material and fired at 910 °C were produced. Ten different filters were fabricated with different mixing ratios of kaolin and sawdust. The clay materials were characterized for their chemical compositions, morphology and thermal stability using different analytical instruments. The performance of the regular and scrubbed ceramic water filters from the different proportions at different the flow rate, their porosities and removal efficiencies of some pollutants in tannery wastewater were evaluated The chemical compositions of the raw material (kaolin) contained oxides of silicon (51.03%) and aluminium (33.75%), respectively. Physicochemical and microbial analysis of the tannery wastewater were carried out using standard methods and the results of the analysis showed significant differences in the removal efficiencies of the pollutants by the various ceramic pot filters. The least porous filter pots coded (T8) containing 67% kaolin and 33% sawdust with flexural strength (55.53 MPa) was found most effective for the removal of COD (78.37%), BOD (91.66%), chloride (88.77%), nitrate (49.07%), sulphate (82.97%), TDS (86.84%), TSS (78.70%), TH (70.00 %) and TA (86.26%) compared to other fabricated pot filters (T1-T7, T9 and T10). All the ceramic filters exhibited excellent antimicrobial removal efficiency towards *Fusarium chlamydosporium*, *Bacillus subtilis* and *Bacillus megaterium* in tannery wastewater. From the results, it was concluded that regular ceramic pot filters performed better than the scrubbed filters due to the clogging effect of the former than the latter. Finally, the degree of porosity affects the performance and removal efficiency of the fabricated kaolin based filters.

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Introduction

Inadequate sanitation facilities and lack of access to safe and sustainable drinking water by the citizens have been recognized as one of the challenges of the 21st century especially in developing countries. In addition, the demand for safe drinking water already exceeds supply in most cities in developing countries. The mortality rate of children below the age of five years caused by diarrhoea, cholera, typhoid and giardiasis emanated from consumption of unhygienic water continued to rise on daily basis. The untreated industrial wastewater from agricultural, mining, pharmaceutical, dyeing, tanning and electroplating activities among others discharged into water bodies also caused death of fresh water organisms. Tannery wastewater obtained via conversion of animal hides into stable products contain high biological oxygen demand (BOD), high chemical oxygen demand (COD), nitrates, sulphates, chlorides, ammonia and microbial organisms such as *Bacillus stibulus* and *Fusarium chlamydosporium* [1]. Tannery wastewater is rich in chromium complex, surfactants, organic dyes, colourants amongst others and their presence in the environment distort the ecosystem. Exposure to tannery wastewater often lead to peeling of skin, skin cancer, vomiting, disruption of endocrine and respiratory tract organs to mention but a few. Therefore, there is an exigent need to protect and improve water supply for people suffering from water-borne infectious disease via development of efficient water treatment method.

Water treatment techniques used to eliminate microbes and other pollutants include boiling, coagulation, flocculation, sedimentation, membrane bioreactor, activated carbon adsorption, filtration and chlorination technology [2]. The oldest and most recognized technologies for killing microbes and removing particles are boiling, chlorination, sedimentation and filtration. Filtration method is known to be simple and economical for the treatment of water. The efficacy of a filter during this process strongly depends on its filtration medium/porosity, composition and quality. Fouling of membrane limits industrial application of nanofiltration and nanomembrane. Generation of toxic slugde and occupation of space remain a serious setback for coagulation and flocculation technology. High cost of importation of activated carbon affect adsorption process for wastewater treatment. In view of the highlighted demerits of conventional wastewater treatment method, there is need to develop cheap and effective technique of purifying industrial wastewater.

Porous Ceramic technology is known to be an efficient method of wastewater purification which consists of a membrane layer and porous ceramic support. Ceramic membranes, however, are expensive [3], although the high cost mainly depend on the raw material, sintering temperature and method of fabrication. On the other hand, ceramic membranes fabricated using naturally occurring material like kaolin are cost-effective and environmentally friendly and can serve as substitutes to the commercially available membranes. Natural clay acts as a good absorbent and photo-oxidant during the removal of contaminants/pollutants from water but the setbacks associated with it are permeability and permanent porosity [4]. To overcome these challenges, the use of composites of clay/porous materials could help not only in creating porosity but also in stabilizing/strengthening the cohesion/adhesion between the compounding materials thereby increasing the stability and avoiding cracking of the filter thus produced.

Ceramic pot filters produced from locally available natural resources like clay and agricultural wastes have shown high ability in effective reduction of water-borne diseases in water [5–8]. Successful trials using natural industrial materials for the production of ceramic pot filters have led to an increase in access to safe water. Therefore, safe and robust ceramic filtration possesses the advantage of cost-effectiveness, use of local materials and the possibility for local investment and entrepreneurship. The quality of ceramic filters produced in factories depends on flow rate and it has been reported that the flow rate of a ceramic filter decreases with time due to clogging via suspended particles [9–11]. Although, scrubbing is the best alternative method to a temporary increase in the flow rate [1,12,13]. Scrubbing has a negative effect on the original flow rate over time due to clogging, thus, resulting in a flow rate reduction. In addition, the volume of filtrate has been one of the major physical constraints using ceramic pot filters. A critical production parameter namely porosity which is essential for improving the quality of ceramic pot produced locally has been understated. This parameter needs to be thoroughly examined during water treatment. In this study, ten (10) trials of ceramic pot filters were done with different mixing ratios of kaolin and sawdust. The fabricated pot filters were characterized using different analytical facilities and subsequently investigated for their removal efficiency towards some physicochemical parameters and microorganisms such as *Fusarium chlamydosporium, Bacillus subtilis* and *Bacillus megaterium* in tannery wastewater.

Materials and methods

The precursors used in this study for the production of ceramic filter production were kaolin, sawdust which acts as combustible material and de-ionized water. The natural kaolin was collected from Gbako Local Government Area, Niger State, Nigeria and first pretreated using the method as described by Mustapha et al. [14]. This was followed by characterization by different analytical techniques to evaluate its elemental compositions, functional groups, phase identification and morphology. The standard Atterberg limit test was used to assess its moisture content, the plasticity and loss on ignition (LOI).

Hydrogen tetraoxosulphate (VI) (H_2SO_4, 98%, Sigma Aldrich) and sodium hydroxide (NaOH, 97%, Merck) used in this study were of analytical grade.

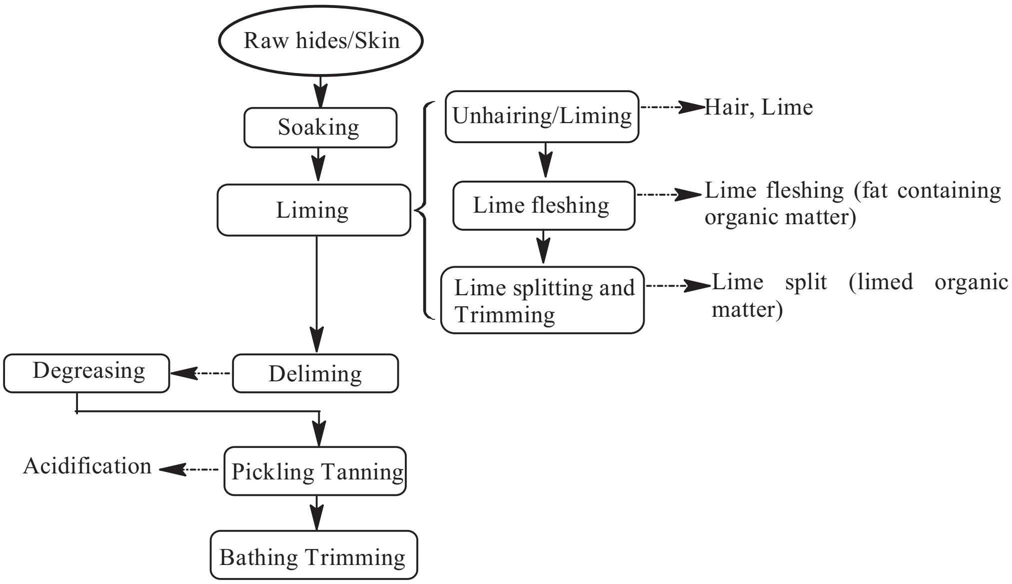


Fig. 1. A schematic diagram of the tanning process.

Characterization of kaolin and kaolin/sawdust mixture

The chemical compositions of the kaolin were analysed using X-ray fluorescence (XRF). The prepared powdered sample was mixed with a binder and the mixture was pelletized at a pressure of 15 Kbars for 60 s. A Phillips PANalytical PW1480 X-ray fluorescence spectrometer model using a Rhodium Tube as the X-ray source was used to analyse the major oxides (%) determined. The morphologies of kaolin and kaolin/sawdust sintered at 910 °C were obtained with high-resolution electron microscopy (HRSEM) (Zeiss Auriga, USA). The powdered sample (0.05 mg) was sprinkled onto carbon adhesive tape and sputter-coated with Au-Pd using a Quorum T15OT for 15 min. The microscope was operated with electron high tension at 5 kV for imaging. The thermal gravimetric analysis (TGA) and thermal differential analysis (DTA) Perkin Elmer thermogravimetric analyser (TGA-4000) were used to investigate the thermal stability profiles of the two materials. The thermal analyses of Gbako kaolin were performed in the temperature range at 30 to 900 °C with a heating rate of 10 °C/min under nitrogen atmosphere. The Raman analysis of the kaolin sample was done in a Renishaw Invia Raman Microscope.

The pore size distribution of the sample was calculated using Image J software on the HRSEM image. The average pore size (d) was evaluated according to the following formula:

$$d = \sqrt{\frac{\sum_{i=1}^{n} n_i d_i^2}{\sum_{i=1}^{n} n_i}} \tag{1}$$

where n_i and d_1 are the pore diameter and the number of pores, respectively.

The mechanical properties of the pot filters were measured using the bending test (Ametek, Lloyd Instruments, United Kingdom). Flexural (F_s) and tensile strength in MPa were calculated using Eqs. 2 and 3, respectively.

$$F_s\ (\text{MPa}) = \frac{3Fd}{2el^2} \tag{2}$$

where F is the strength (N), d is the distance between the supports, e and l are the thickness and width of the rectangular specimen (mm), respectively.

$$T_s = \frac{2F_T}{\pi Dh} \tag{3}$$

where F_T is the strength (N), D and h are the diameter and height of the specimen (mm), respectively.

Physicochemical parameters of tannery wastewater

The tannery wastewater sample was collected into clean gallons from the outlet point of tanning stage in Majema tannery industry, Manuri Road, Tudun Wada Area, Sokoto State. The wastewater from the tannery industry used in this study consists of different tanning stages namely; soaking, liming, deliming, pickling/acidification and bathing. The flow chart (Fig. 1) shows the input of waste that is either liquid or solid released during the tanning process.

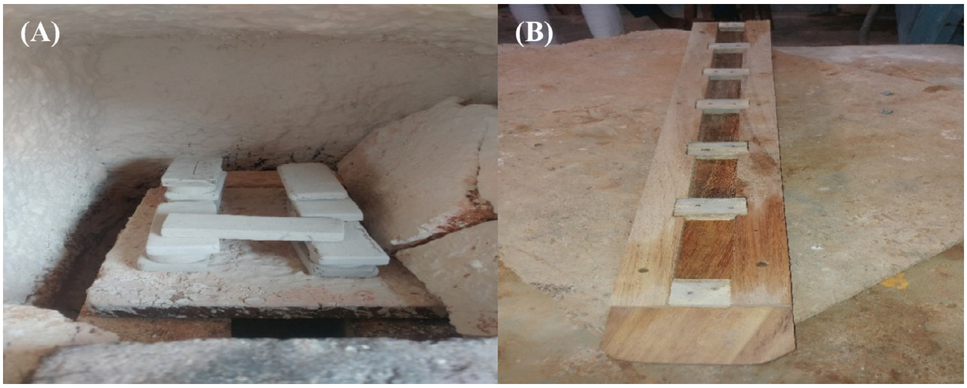


Plate 1. (A) Trial bars prepared from beneficiated kaolin and (B) Mould.

The wastewater was transferred to the laboratory for analysis. The following parameters of the tannery wastewater such as COD, BOD, total suspended solids (TSS), total dissolved solids (TDS), sulphate, nitrate, phosphate, chloride, turbidity, total hardness and total alkalinity (TA) were measured according to the standard methods reported by American Public Health Association [15].

Bacteriological analysis of tannery wastewater

The microbial colony counter was used for counting the number of microbial cells in the tannery wastewater and the result was recorded as cfu (colony forming unit) which is the S.I unit of microbial counts. After counting the microbial cells, the pure culture was made by sub-culturing a single colony into a fresh nutrient Agar plate for bacteria and suborned dextrose Agar for microbes to obtain a pure isolate. After sub-culturing, the plates were incubated accordingly. These were used for characterization (identification) of the isolates and finally naming of the organisms. A smear was made on a clean grease-free glass slide. The smear was flooded with crystal violet and allowed to stay for 30 s and then diluted with Grains of iodine after 30 s, this acted as a mordant on the smear and left for another 30 s. This was then drained and decolourized with 95% ethyl alcohol for 1 min. After decolonization, the smear was counterstained with safranine and left for 1 min. This was finally washed with distilled water and moped with Whatman filter paper 42 and finally viewed at x 100 (objective lens) using a binocular microscope. This gave the evolutionary trend of bacteria. The Gram positive retained the colour of wastewater dye of crystal violet. The Gram-negative retained the colour and the cell shape was either cylindrical (rod) or spherical (cocci), the argent of the cells only 0.2 connected (pain), scattered (clusters), or connected in long rod (chain).

Ceramic filter production

A laboratory-scale test was used for the fabrication of ceramic pot filter. The production of the ceramic filters were carried out at the Industrial Design Department of the Federal University of Technology, Akure, Nigeria. Different ratios of raw kaolin and sawdust were sieved using 250 μm mesh sieve to obtain uniform size for prevention issues with control of the compaction step and the final product features. The kaolin was moulded into shapes via wooden moulds and lubricant was applied to the surface of the moulds to prevent the kaolin sample from sticking to their surfaces. The initial shape was spherical with a width of 3.5 cm and the second shape was a rectangular bar with length 6 cm as shown in Plate 1.

The blends of kaolin/sawdust were hydrated by the addition of de-ionized water and homogenously mixed and kneaded with friable material. The resultant mixture was allowed to age for 72 h. The ageing of the mixture will be caused plasticity, fermentation and some physical changes in the material. Plate 1 describes the used kaolin bars calcined at 910 °C for the determination of porosity, water absorption and shrinkage. The filter bars produced were designated as T1 to T10.

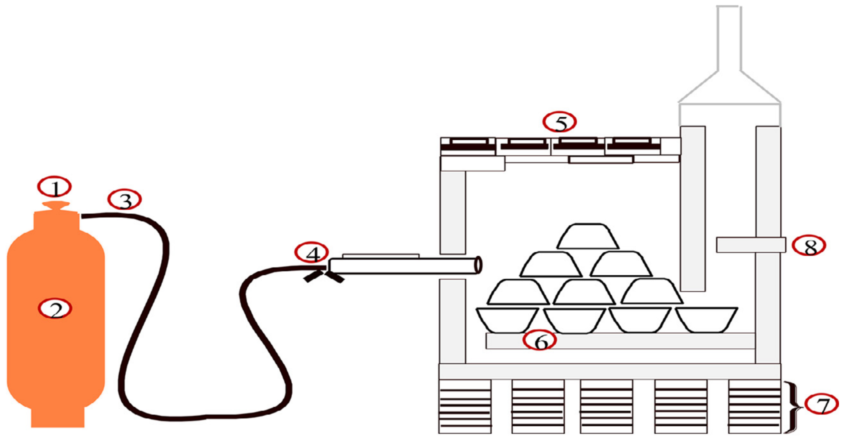
The mixtures were moulded using giggle jolly machine as shown in Plate 2 (A). The moulded filter materials were dried at ambient temperature for 7 days to prevent the manufactured pot filters from cracking when sintering in a kiln. Afterwards, the dried ceramic filters were placed inside a kiln as seen in Plate 2 (B) and then fired in a kiln at 910 °C. The firing consisted of different stages namely; dehydration (100–300 °C), oxidation (300–500 °C) and vitrification (500–900 °C). Ten (10) ceramic pot filters at different kaolin to sawdust ratios (see Table 1) produced using a gas-fired temperature-controlled kiln as presented in Fig. 2.

The filters fired up to 500 °C at the rate of 100 °C/h were stopped after heating for 1 h and continued until the maximum temperature of 910 °C was attained. The gas cylinder was closed and the filter pots were naturally allowed to cool completely in the kiln. The fired ceramic pot filter samples labelled (T1-T10) were tested for water absorption, flow rate, porosity, microbial and some physicochemical parameters removal efficiency.

Plate 2. (A) A Pictorial representation of giggle jolly machine and (B) Kiln.

Table 1
Various trials of pot filters at different proportions of kaolin/sawdust.

Sample	Percentage (%)		Total
	Clay	Sawdust	
T1	40	60	100
T2	44	56	100
T3	33	67	100
T4	46	54	100
T5	50	50	100
T6	60	40	100
T7	54	46	100
T8	67	33	100
T9	65	35	100
T10	56	44	100



1. Tap 2. Gas cylinder 3. Nozzle 4. Burner 5. Roof parts 6. Internal floor 7. Pillars 8. Chimney

Fig. 2. A gas fixed temperature-controlled kiln for the production of pot filters.

Characterization of moulded ceramic filter pots

Shrinkage test

The original lengths of the moulded spherical and rectangular bars were recorded immediately after moulding as L_1 (cm). The pieces of the bar were then air-dried for 3 days and subsequently, oven-dried for at 105 °C for 72 h to attain constant weights. The shrinkages from the marks on the bars were determined and recorded as L_2 (cm). The sample bars were further calcined at a temperature of 910 °C. The shrinkages of the bars from the marks after firing at 910 °C were recorded as L_3 (cm) and linear and total shrinkage were determined using Eqs. 4 and 5.

$$\text{Linear shrinkage}(\%) = \frac{L_1 - L_3}{L_2} \times 100 \tag{4}$$

$$\text{Total shrinkage}(\%) = \frac{L_1 - L_3}{L_1} \times 100 \tag{5}$$

Determination of water absorption

The calcined test bars and filter pots were weighed and recorded as M_1 (g). Afterwards, the sample bars were soaked in de-ionized water for 24 h, then removed, cleaned and weighed immediately as M_2 (g). The percentage of water absorption was calculated using Eq. 6.

$$\text{Water absorption}(\%) = \frac{M_2 - M_1}{M_1} \times 100 \tag{6}$$

Determination of porosity

The fired bars and filter pots at 910°C were weighed and soaked in water for 24 h. The apparent porosity of samples was calculated using Eq. 7.

$$\text{Apparent porosity}(\%) = \frac{M_w - M_f}{\rho_e V_f} \tag{7}$$

where M_f is the weight of the fired sample (g), M_w is the weight of soaked sample in water for 24 h (g), V_f and ρ_e is the volume of fired sample (cm^3) and volumic mass of water (g/cm^3), respectively.

Chemical resistance analysis

The chemical resistance was performed on the optimum filter fired at 910 °C. The flat bar and ceramic filter were immersed in solution and pH of the solution was varied from 1–12 and adjusted to the desired value using either 0.5 M H_2SO_4 and 0.5 M NaOH and after the adjustment the flat bars were left in the solution for 48 h. Thereafter, the bars and ceramic pot filter were removed from the solution weighed and the chemical resistance was computed as follows:

$$\%\ R = \frac{M_1 - M_2}{M_1} \times 100 \tag{8}$$

where M_1 and M_2 are the mass of the sample before pH attach and mass after removing from acidic/basic solution (g).

Pollutants removal

The fabricated ceramic pot filters were assessed for the treatment of tannery wastewater based on the filtration rates and the collected filtrates were tested for the final concentrations of the selected analytes using standard analytical methods. The initial and final concentrations of some physicochemical parameters of the wastewater were performed to evaluate the treatment efficacy of the pot filters using Eq. 9.

$$\text{Removal efficiency} = \frac{C_i - C_f}{C_i} \times 100 \tag{9}$$

where C_i and C_f are the initial and final concentrations of the pollutants, respectively.

Results and discussion

Characterization of kaolin

The XRF analysis showed that the main constituents of the beneficiated kaolin were silica, alumina and hematite as shown in Table 2. From the results, the chemical compositions of the raw material contained oxides of silicon (51.03%) and aluminium (33.75%), respectively. The presence of CaO and K_2O indicate that the kaolin sample lack swelling properties which make it suitable for the refractory application. Furthermore, the result suggests that the presence of CaO in the kaolin could prevent cracking of the sample when fired at a certain higher temperature. Also, the presence of TiO_2 in the kaolin could impact corrosion resistance property on the final products (ceramic filter) because it serves an opacifier. The small amounts of TiO_2 and P_2O_5 could support the antimicrobial properties of the kaolin in the presence of light [6]. More so, the result of Raman analysis of kaolin presented in Fig. 3 revealed absorption bands of kaolinite at 396, 497 and 637 cm^{-1} and these corresponds to the bending motion and stretching region.

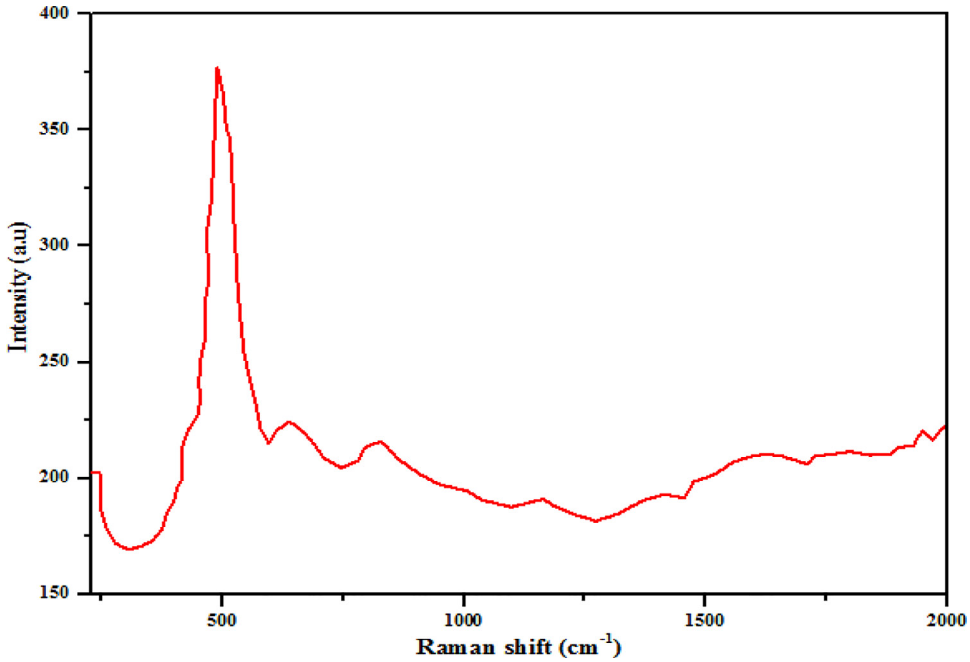


Fig. 3. Analysis of kaolin using Raman spectroscopy.

Table 2
XRF analysis showing the composition of kaolin sample from Gbako Local Government Area, Niger State, Nigeria.

Constituent	Wt %
SiO_2	51.03
Al_2O_3	33.75
Fe_2O_3	3.27
MgO	2.26
CaO	1.63
TiO_2	3.48
K_2O	3.08
Na_2O	1.40
P_2O_5	0.07
SO_3	0.03

TGA/DTA analysis

The thermal stability profile of compacted pellet of kaolin/sawdust blend at 67/33 mixed proportion (optimal filter) was investigated using TGA and corresponding result is represented in Fig. 4. It can be observed that complete dehydration of water and burn off combustible material (sawdust) of the sample occurred at 338.57 °C accompanied with a mass loss of 1.82%. At the temperature range of 320.85 °C to 527.79 °C, remarkable loss total volatile compounds and saw dust were observed. This allowed dehydroxylation process, ashing of sawdust material and the formation of metakaolin to occur. At this region, a DTA sharp endothermic peak at 407.38 °C was observed. The peak could be attributed to kaolinite dehydroxylation process which depends on the material, particle size, rate of gas flows and heating rate. This observation is supported by researcher [16].

Pore size distribution

The HRSEM micrograph of kaolin and compacted pellet of kaolin/sawdust is shown in Plate 3. Compared to Plate 3 (a), which revealed closely packed network structure of kaolinite particles, a significant increase in the porosity and absence of cracks can be noticed in Plate 3 (b). On the other hand, cracks may likely occur in kaolin/sawdust mixture if fired above the calcination temperature of 910 °C. The absence of porosity and highly packed crystals arrangement in Plate 3 (a) could be due to vitrification. The pore size distribution of the sintered samples (kaolin and kaolin/sawdust) as presented in Fig. 5 showed pore diameters of 2.17 μm and 2.79 μm, respectively. The high resulting value of pore size distribution was

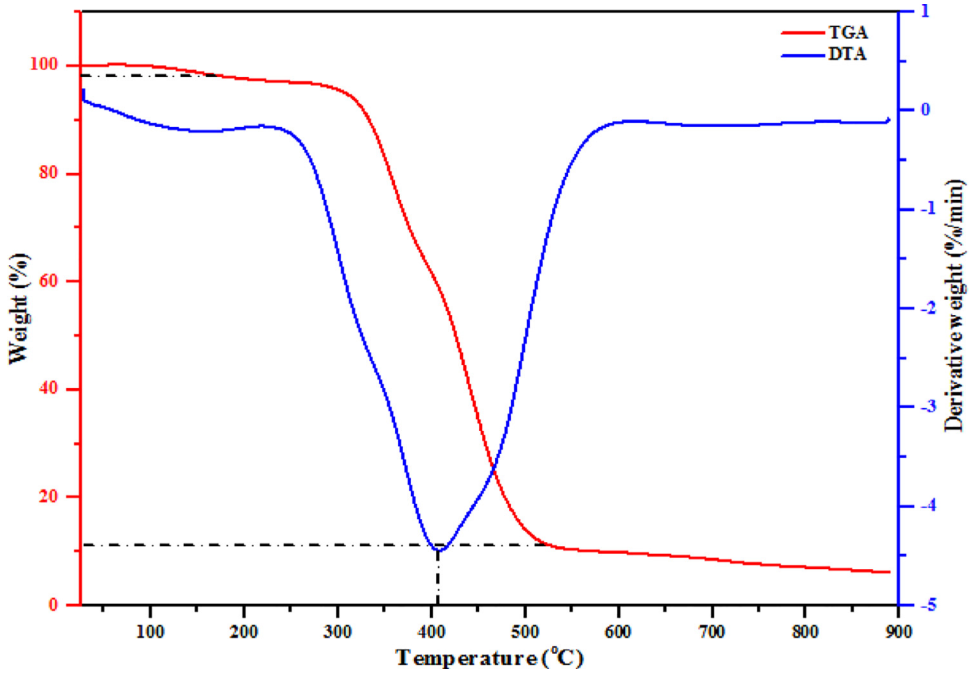


Fig. 4. TGA/DTA of kaolin/sawdust calcined at 910°C.

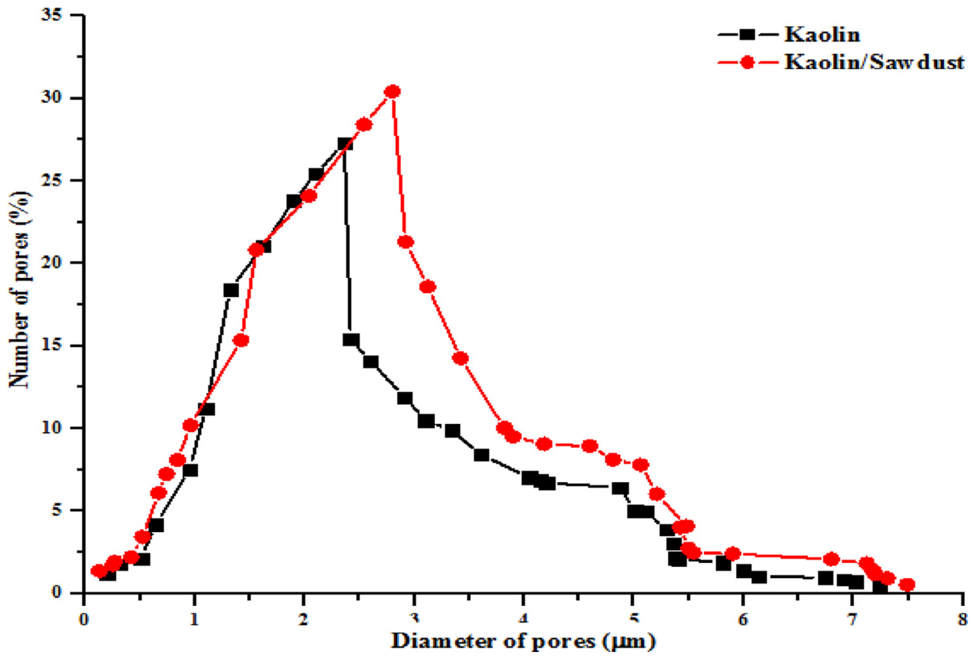


Fig. 5. Pore size distribution of kaolin and kaolin/sawdust sinter at 910 °C.

noticed on addition of sawdust to kaolin. Similar study was observed by Mouiya et al. [17] who concluded that the presence of banana peel powder reduced the contact between the clay grains, thus leading to formation of high pores. The presence of sawdust in the filter was responsible for the enhanced porosity in Plate 3 (b).

Fabrication of pot filters

The results from the percentage porosity and correlation coefficients of kaolin pot filters are presented in Table 3. It could be seen from the results that higher amounts of kaolin in the mixture may be responsible for reduction in the %

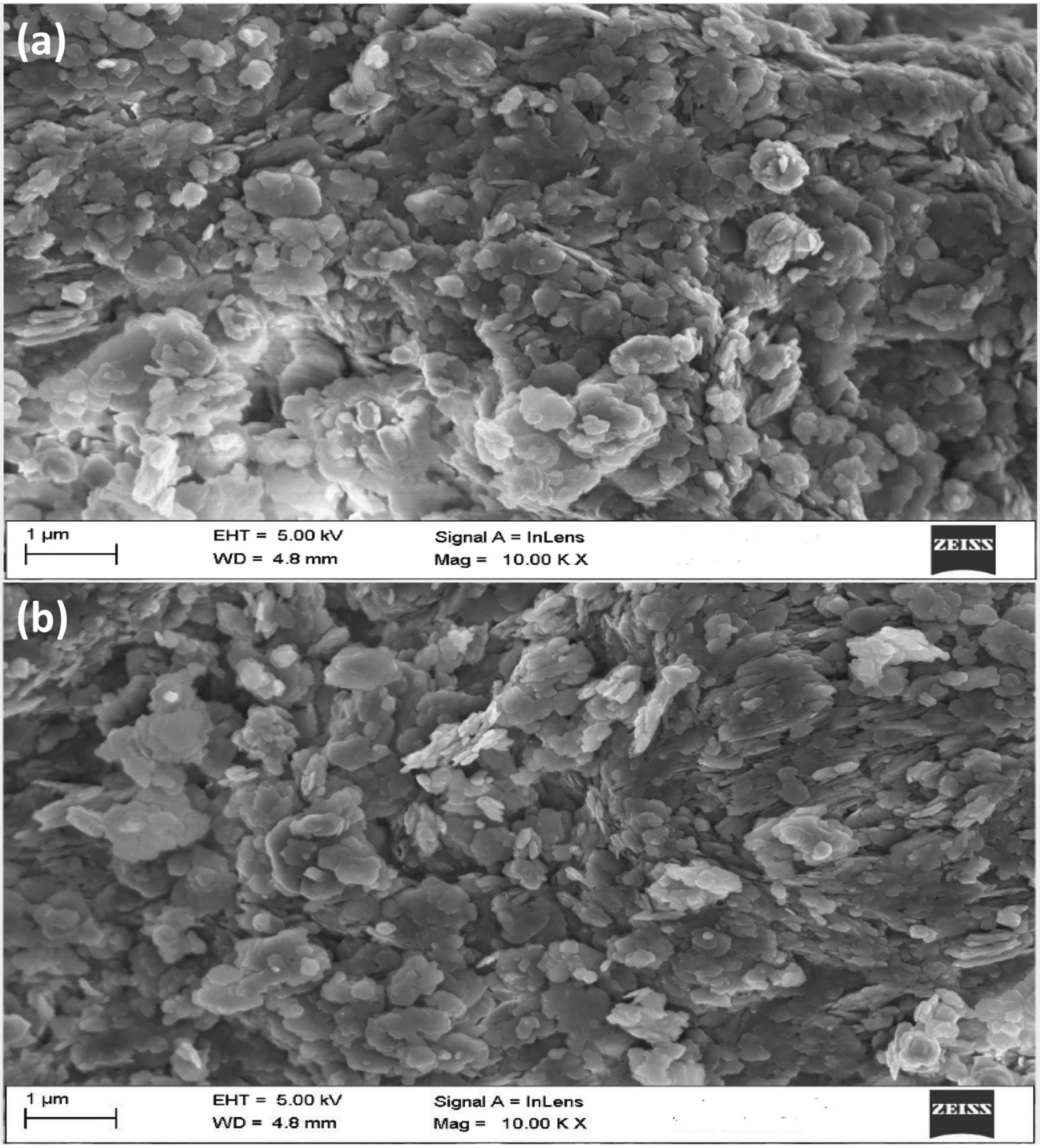


Plate 3. HRSEM microimages of kaolin (a) and compacted pellet of kaolin/sawdust (b) sintered at 910 °C.

porosity of the composites (kaolin/sawdust). The low percentage of porosity will lead to a low flow rate of liquid through the porous pot filters (permeability). On the other hand, higher amount of saw dust in the composite contributed to the increased percentage porosity.

Table 3 shows the effect of temperature on different trials of pot filters. On the application of heat, the combustible material began to burn at 250 °C and smoke was observed coming out of the chimney of the furnace. At 600 °C, there was no smoke any more, indicating that complete burning of the impregnated sawdust. The oxides in the kaolin could have act as fluxes, thereby combining with silica and alumina, thus reducing the refractoriness of the filter samples. As presented in Table 3, the loss in the ignition (LOI) was 5.03% which is attributed to the dehydroxylation reaction in the kaolin mineral. This low LOI is an indication of low porosity in the manufacture of the product during kilning. The result obtained shows that the kaolin filter samples had fair refractory properties and did not rupture at 910 °C as displayed in Plate 5. The shrinkage, plasticity and porosity are important physical factors to be considered after kilning at a temperature of 910 °C as shown in Table 3.

Table 3
Various trials done on pot filters

Sample			After firing					%
	Kaolin/Sawdust	Wt. before firing (g)	Line (cm)	Weight	Line	Weight	Weight after soaking	Porosity
T1	40/60	36	54.00	26	10.00	58.07	41	54.07
T2	44/56	44	55.50	34	7.50	45.60	48	42.20
T3	33/67	34	53.00	24	11.67	61.29	40	64.72
T4	46/54	48	55.00	36	8.33	41.94	49	38.01
T5	50/50	46	56.00	37	6.67	42.85	50	36.49
T6	60/40	54	56.00	44	6.67	29.03	55.6	20.14
T7	54/46	50	58.00	40	3.33	35.48	52	26.19
T8	67/33	52	58.50	47	2.50	24.18	53	15.40
T9	65/35	54	56.00	46	6.67	32.25	54	17.82
T10	56/44	52	56.00	47	6.67	25.81	56	23.60

Initial line = 60 mm; LOI of the beneficiated clay = 5.03 %; Refractoriness = 910±5 °C; Colour after firing = light ash.

Plate 4. Fabricated kaolin pot filters at different proportions of sawdust and kaolin

Plate 5. Fusarium chlamydosporium.

The % porosity and shrinkage after firing decreased and also increased with respect to the blend ratio of kaolin/dust. It was seen that at higher ratios of kaolin to sawdust, there were low % porosity and shrinkage. This implies that on the application of temperature, the product tended to compress while the low shrinkage indicates high levels of non-fluxing impurities. The decrease in the shrinkage for the refractory material is attributed to the coming together and closure of the pores of the clay body [18]. In this study, porosity was found to be directly related to shrinkage.

There were interstitial spaces between the kaolin after kilning the kaolin/dust. These small spaces are as a result of the loss of the carbonaceous material at high temperatures. Therefore, temperature, the ratio of kaolin and dust formulation are the principal factors governing the flow rate and percentage of porosity of the pot filters. A greater amount of sawdust over kaolin increased water absorption. While there was a decrease in water absorption on the addition of more kaolin and this was due to the reduction in porosity and making of the kaolin/sawdust composite more plastic. The high plasticity tends to make the moulding of clay pot workable and easily mouldable. The fabricated pot filters are presented in Plate 4.

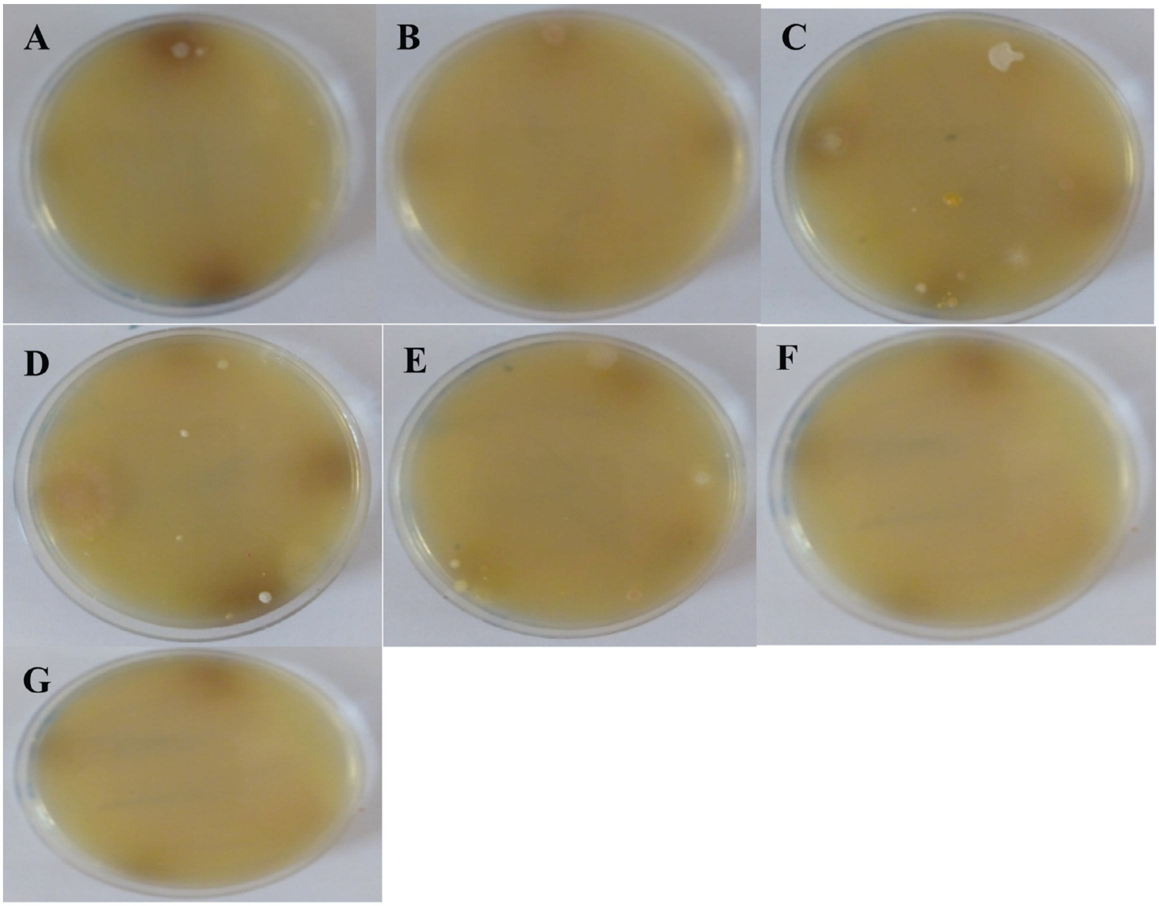


Plate 6. Inhibition growth of bacteria using T4-T10 pot filters.

Table 4
Mechanical properties of pot filters

Sample	Flexural strength (MPa)	Tensile strength (MPa)
T1	30.12±0.51	15.62±0.20
T2	38.30±1.20	19.70±0.64
T3	26.52±0.80	9.42±1.31
T4	40.15±0.13	25.40±0.81
T5	41.24±0.48	26.15±0.25
T6	47.60±0.52	28.30±0.58
T7	49.17±0.19	31.52±0.45
T8	55.53±0.82	43.10±0.30
T9	52.40±1.30	36.23±0.75
T10	54.58±0.70	41.65±0.54

Mechanical properties

The flexural and tensile strength values of the pot filters at different compositions of kaolin/sawdust are presented in Table 4. The results imply that improving the amount of sawdust in the mixture caused a decrease in the flexural strength. This could be as a result of the increase in porosity of higher ratio of the filters with the increase in the ratio of sawdust as seen in Table 4 which indicates the porosity increased with flexural strength and tensile strength decreased. A similar result was demonstrated by Mouiya et al. [17]. In this study, the mechanical properties of T8 exhibited the optimum having the lowest porosity and highest tensile and flexural strengths. This highlights the efficiency of the amount of kaolin in the porous pot filter.

Table 5
Summary of the flow rate of the filters.

Sample	Kaolin/Sawdust (mixed ratio)	% Porosity	Flow rate (cm^3/hr)		
			Regular	First scrubbed	Second scrubbed
T1	40/60	54.07	736	680	670
T2	44/56	42.20	692	620	585
T3	33/67	64.72	850	790	740
T4	46/54	38.01	620	575	540
T5	50/50	36.49	612	570	530
T6	60/40	20.14	530	490	470
T7	54/46	26.19	510	440	420
T8	67/33	15.40	260	210	205
T9	65/35	17.82	305	280	260
T10	56/44	23.60	480	420	410

Performance evaluation of pot filters

Table 5 shows that the flow rate could be effectively used to check the amount of filtered wastewater. The flow rate was optimized by changing the proportion of kaolin and sawdust as shown in the table. The amount of sawdust loaded on the pot filter formulation served as the controller of flow rate and percentage of porosity of the pot filter. The flow rate was found to vary depending on the ratios of the kaolin and sawdust. The porous pot filters studied at different percentage ratios of kaolin to sawdust in descending order were: 33/67 (**T3**), 40/60 (**T1**), 44/56 (**T2**), 46/54 (**T4**), 50/50 (**T5**). 60/40 (**T6**), 54/46 (**T7**), 56/44 (**T10**), 65/35 (**T9**) and 67/33 (**T8**). The least and highest flow rate recorded were 12.77 and 66.67%, respectively. The slowest flow rates were associated with a high amount of loaded kaolin. This might be the consequence of strong interlinking and bond network of kaolin-based ceramic filter which resulted in less filtration of the water molecule [4]. The respective flow rates at minimum (260 cm^3/hr) and maximum (850 cm^3/hr) in this work were below the observed values reported by Bulta and Michael [19] and Ajayi and Lamidi [20]. This might be the consequence of mixing ratio of the raw materials. It is worthwhile to note that the flow rate of wastewater discharged by the pot filters increased with porosity and this finding corroborates with the study of Nnaji et al. [21].

It was observed in this study that the direct use of the tannery wastewater on the pot filter affected the flow rate which invariably resulted in a low flow rate. It was thus opined that factor such as suspended solid of the wastewater could have influenced the flow rate. Therefore, a rapid estimation of the flow rate was performed firstly by removing particulates from wastewater using Whatman filter paper to prevent the pot filter fouling by suspended matter. Finally, the discharged filtrate was filtered through the fabricated pot filter and reasonable amounts of filtrates were produced. Some of the physicochemical parameters of tannery wastewater filtrate collected from the fabricated pot filters were determined using APHA [15] method. Table 5 shows the results of the physicochemical parameters of the treated tannery wastewater using the filtration system. The removal efficiency of the filters was essentially attributed to the percentage of porosity of the filters and the functional groups of the precursor (kaolin). The results obtained show that the level of porosity also affected the degree of pollutants removal by the ceramic filters. It should be noted that the focus of this study was on the degree of porosity and pollutants removal efficiency of kaolin-sawdust ceramic pot filters, as this is an uncommon practice in most fabricated ceramic pot filters. Therefore, as a reference, the pollutants removal efficiency was linked to the percentage of porosity in this study. The order of removal of pollutants from wastewater in this study by the fabricated pot filters are arranged thus: T8 > T9 > T10 > T7 > T6 > T5 > T4.

The long-term performance of the pot filters was also assessed. During the experiment, the pot filters were scrubbed for two successive times and the physicochemical properties of the filtrates were determined and the results are presented in Tables 7 and 8. In the first scrubbing of the filtration system, flow rates dropped, likewise for the subsequent run. The differences in the filtratation rates were attributed primarily to clogging in all filters caused by suspended particles/foulants from wastewater. The system performance of the pollutants removal efficiency of the scrubbed pot filters was highly variable, influenced by the cleaning activity and wastewater concentration as presented in Tables 7 and 8. This can be linked to the successful accummulation of fouling pollutants on the surface of the filters. Thus, this could be improved through the use of sponge on the filter, rinsing with de-ionized water and firing in a kiln.

The removal efficiencies of the filter pots were observed for regular and scrubbed pot filter (T4-T10) as shown in Figs. 6 and 7. The removal rates decreased noticeably in scrubbed filter pots.

The actual measurement of filtration rates was not recorded but the observation was on the adsorption of the pollutants from the wastewater. There were significant changes in the performance of the filter pots due to the effect of scrubbing. Previous studies show that regular filter pots perform better but differ in flow rates as a result of different ratios of burnt material in clay mixtures [5,22]. Furthermore, Van Halem et al. [5] reported that foulant accumulated in/on the regular filters after scrubbing yielded large amounts of flow rates and microorganisms. In contrast to the study of Lawal et al. [23], the produced ceramic filters were evaluated on the reduction of some physicochemical parameters in wastewater who failed to check the quality performance of ceramic filters after cleaning. It was interesting to note that the highest percentage

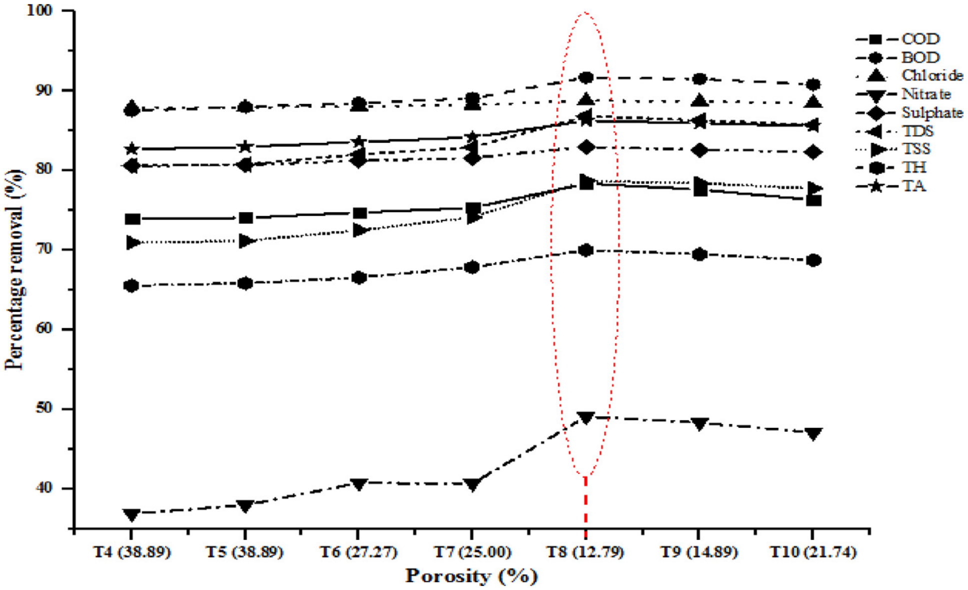


Fig. 6. The total removal efficiencies of the regular pot filters.

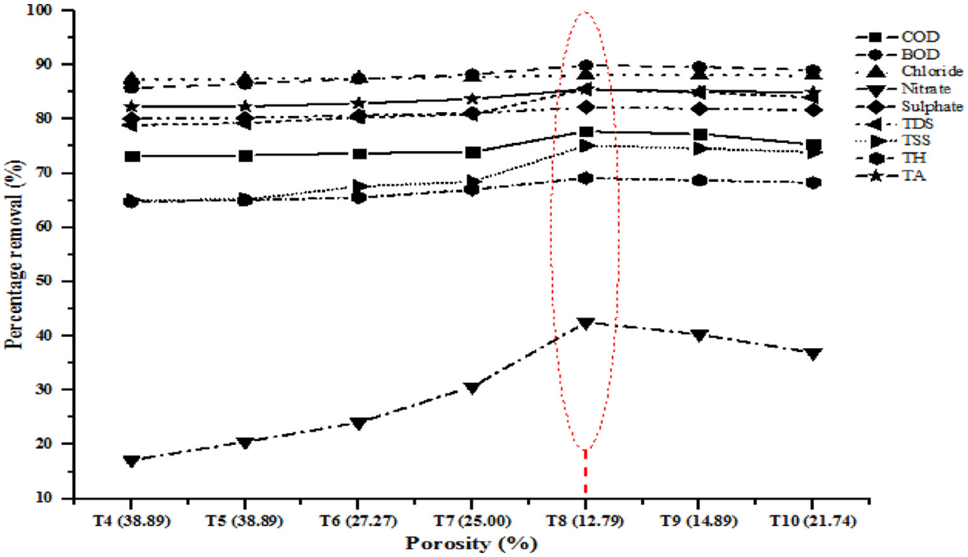


Fig. 7. The total removal efficiencies of the first scrubbed pot filters.

removal of the pollutants was found in T8. This may be due to the use of kaolin material with the highest proportion (65%) to sawdust (35 (%). The results indicate that porosity and flow rate had a significant effect on pollutants removal using the filter pots. In addition to removal efficiency and flow rate, the porosity of the pot filters also affected their mechanical properties thus, porosity decreased with increasing flexural and tensile strengths [24].

Bacterial removal efficiency

The antibacterial activities of kaolin/sawdust composites were observed for 30 min using the bacterial suspensions of the wastewater. It was observed that bacterial inactivation was exhibited by the pot filters from T4 to T10 as presented in Plate 6. No bacteria were detected in the filtrates collected using filter T8. The antimicrobial efficiency removal was found at 100 %. Thus, indicating that the efficiency of bacteria removal depends on the porosity of the pot filters. It has been documented that the porosity of ceramic filters is the determining factor for the removal of particles and pathogen from water. Therefore, the observation in this study corroborates with the finding of Bulta and Michael [18] and Zereffa and Bekalo [25] who reported that low porous ceramic filters had high-efficiency removal of microorganisms. Thus, the clear pictures of an inhibition zone show the antibacterial effect of the filters and can be attributed to physicochemical

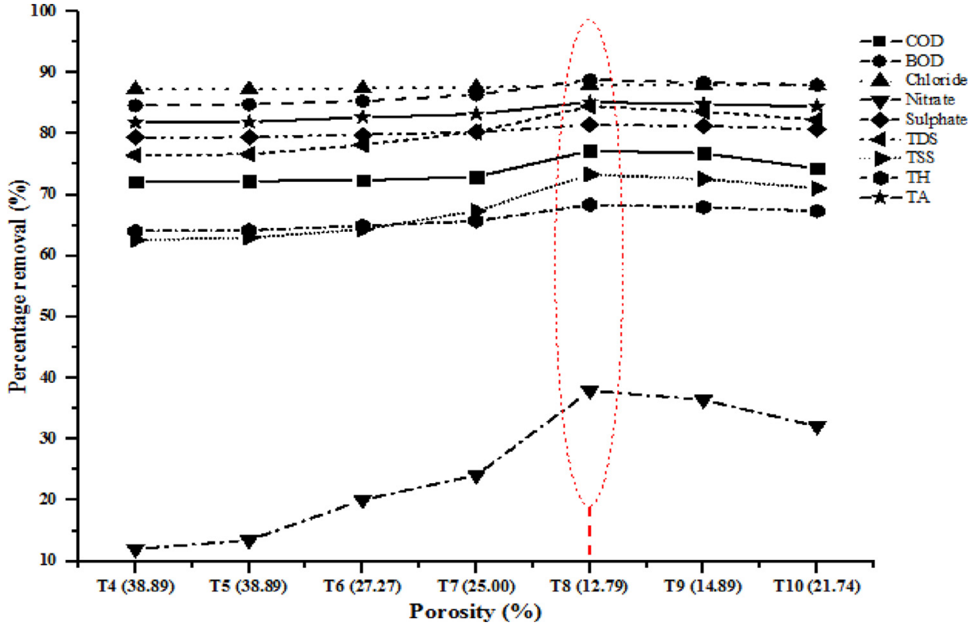


Fig. 8. The total removal efficiencies of the second scrubbed pot filters.

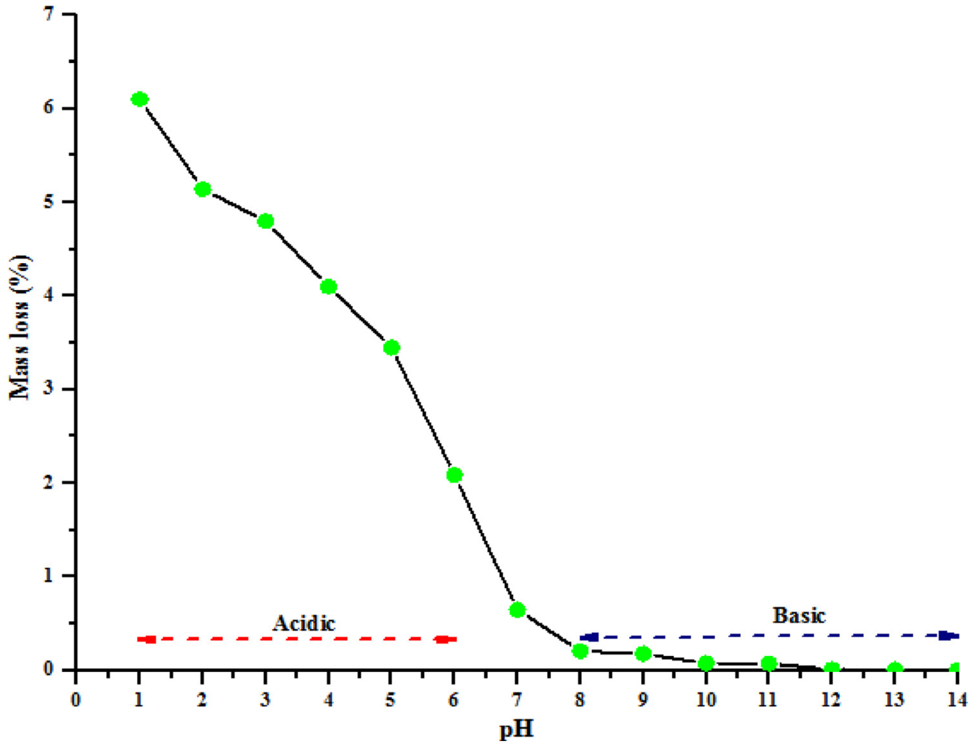


Fig. 9. Weight loss (%) of optimal sintered kaolin/sawdust filter inacidic and basic medium at ambient temperature for 48 h.

properties as well as the surface area of the kaolin. Therefore, the raw material used in this study was efficient in which no viable bacteria was detected in the wastewater, and implies that the material exhibited good antibacterial properties on the pathogens viz *Bacillus subtilis* and *Bacillus megaterium*. The findings of this study are generally analogous with a previously published report by Karim et al. [26] who used commercially produced mineral pot filter for the removal of bacteria from water The microbial activity of the tannery wastewater presented in Table 9 and Plate 5 was performed to determine the type of bacteria that exists in the wastewater.

Table 6
Removal of some physicochemical in wastewater properties using regular pot filters.

Parameter	Control	T4	T5	T6	T7	T8	T9	T10
COD (mg/dm^3)	1988.60±0.23	518.12±0.16	516.04±0.40	503.10±0.18	491.02±0.40	430.20±0.10	445.23±0.13	471.30±0.72
BOD (mg/dm^3)	625.30±0.10	78.20±0.05	75.20±0.28	72.10±0.09	68.50±0.12	52.15±0.02	53.10±0.22	57.60±0.07
Chloride (mg/dm^3)	7580.50±0.42	916.20±0.04	915.38±0.01	908.30±0.16	892.45±0.15	851.20±0.24	860.14±0.18	874.10±0.89
Nitrate (mg/dm^3)	118.30±0.16	74.65±0.09	73.40±0.06	70.10±0.53	70.20±0.05	60.25±0.01	61.13±0.10	62.61±0.18
Sulphate (mg/dm^3)	2944.00±0.22	572.40±0.03	568.90±0.01	552.20±0.45	542.01±0.04	501.25±0.08	512.60±0.41	519.40±0.90
TDS (mg/dm^3)	724.00±0.10	140.40±0.07	139.20±0.19	130.19±0.56	123.68±0.19	95.30±0.35	99.18±0.60	103.20±0.32
TSS (mg/dm^3)	438.00±0.30	127.32±0.16	126.35±0.30	120.45±0.04	113.40±0.15	93.28±0.12	94.60±0.19	97.50±0.05
TH (mg/dm^3)	1500.00±0.61	517.30±0.06	512.48±0.03	501.80±0.23	482.30±0.07	450.02±0.01	458.17±0.32	469.20±0.80
TA (mg/dm^3)	2200.00±0.91	381.01±0.02	374.30±0.03	361.02±0.08	348.20±0.01	302.24±0.10	309.30±0.26	316.50±0.19

Table 7
Removal of some physicochemical properties in wastewater using first scrubbed pot filters.

Parameter	Control	T4	T5	T6	T7	T8	T9	T10
COD (mg/dm^3)	1988.60±0.23	532.05±0.20	530.64±0.61	522.30±0.45	517.92±0.50	442.16±0.54	451.87±0.80	490.62±0.40
BOD (mg/dm^3)	625.30±0.10	89.01±0.59	84.40±0.46	78.28±0.80	73.35±0.38	63.04±0.20	64.50±0.32	68.37±0.95
Chloride (mg/dm^3)	7580.50±0.42	956.10±0.28	954.43±0.13	941.09±0.35	932.30±0.30	895.20±0.90	899.24±0.60	903.15±0.30
Nitrate (mg/dm^3)	118.30±0.16	98.02±0.14	94.02±0.25	89.84±0.80	82.01±0.07	68.00±0.75	70.58±0.28	74.58±0.74
Sulphate (mg/dm^3)	2944.00±0.22	585.14±0.86	581.02±0.10	568.35±0.47	557.01±0.60	524.02±0.65	530.75±0.41	539.01±0.01
TDS (mg/dm^3)	724.00±0.10	152.50±0.40	150.09±0.40	142.35±0.20	138.27±0.29	104.52±0.42	108.94±0.52	115.48±0.75
TSS (mg/dm^3)	438.00±0.30	153.06±0.80	152.36±0.27	141.96±0.60	138.04±0.47	109.01±0.85	111.28±0.40	114.27±031
TH (mg/dm^3)	1500.00±0.61	528.20±0.38	524.01±0.50	516.46±0.04	495.02±0.50	462.64±0.60	469.30±0.10	475.36±0.47
TA (mg/dm^3)	2200.00±0.91	390.14±0.11	388.05±0.48	375.27±0.09	358.19±0.38	318.05±0.47	327.53±0.28	330.03±0.82

Table 8
Removal of some physicochemical properties in wastewater using second scrubbed pot filters.

Parameter	Control	T4	T5	T6	T7	T8	T9	T10
COD (mg/dm^3)	1988.60±0.23	553.70±0.28	551.90±0.58	549.25±0.36	538.10±0.09	453.02±0.50	461.33±0.14	510.40±0.51
BOD (mg/dm^3)	625.30±0.10	96.15±0.43	95.03±0.59	91.60±0.48	85.01±0.15	70.10±0.25	72.50±0.37	75.35±0.35
Chloride (mg/dm^3)	7580.50±0.42	967.20±0.70	965.05±0.46	953.17±0.62	940.30±0.24	908.16±0.30	910.40±0.41	918.40±0.40
Nitrate (mg/dm^3)	118.30±0.16	104.10±0.50	102.40±0.75	94.57±0.16	89.80±0.60	73.41±0.19	75.20±0.70	80.30±0.97
Sulphate (mg/dm^3)	2944.00±0.22	608.25±0.80	605.18±0.80	593.64±0.02	582.07±0.40	545.09±0.56	551.96±0.10	565.20±0.18
TDS (mg/dm^3)	724.00±0.10	170.23±0.12	169.15±0.93	158.15±0.05	142.70±0.35	112.40±0.02	118.62±0.32	128.20±0.45
TSS (mg/dm^3)	438.00±0.30	163.86±0.39	162.17±0.81	156.30±0.81	142.95±0.28	116.80±0.43	120.10±0.40	126.80±0.03
TH (mg/dm^3)	1500.00±0.61	539.40±0.48	537.41±0.76	526.34±0.06	514.20±0.42	474.17±0.05	480.35±0.61	489.35±0.01
TA (mg/dm^3)	2200.00±0.91	400.20±0.72	397.38±0.14	381.25±0.35	369.20±0.17	326.27±0.50	333.43±0.24	341.18±0.15

Table 9
Microbial analysis of wastewater before treatment.

Sample	TAC (cfu/mL)	TANC (cfu/mL)	TCC (cfu/mL)	TSC (cfu/mL)	TSTC (cfu/mL)	TSPC (cfu/mL)	Macro-culture	S	GS	COUG	CT	SH	CIT	MR	IND	L	S	G	Name of the organism
Tannery wastewater	2.80×10^6	NIL	NIL	NIL	NIL	NIL	Mucoid, spherical, dull-white raised or concave colonies	R	+	-	+	+	+	-	-	-	+	+	*Bacillus subtilis*
							Non-mucoid, spherical beads like in shape and aragant, whitish colonies	R	-	-	+	+	-	-	-	-	+	+	*Bacillus megaterium*

Key: TAC = Total aerobic mesophilic count; TCC = Total coliform count; TSC = Total salmonella count; TSTC = Total streptococcus species count; TSPC = Total staphylococcus species count; TANC = Total anaerobic bacteria count; S = Shape of bacteria cells; GS = Grain's stain; COUG = Cougulase test; CT = Citrate utilization test; SH = Starch hydrolysis test; MR = Methyl res test; IND = Indole utilization test; L = Lactose sugar; S = Sucrose sugar; G = Glucose test; + = Positive (Has reaction); − = (No reaction); cfu = Colony forming unit (S.I unit of microbial count); mL = per one millimeter of sample.

Chemical resistance behaviour of the filter

The resistance to acid/base corrosion performed on the optimal filter produced showed that significant weight loss was observed on soaking in acidic solution (pH 1–6). The produced filter exhibited slight weight loss in a weak acidic solution compared to the concentrated regions. Fig. 9 shows the mass loss of optimal filter produced in acidic and basic solution at constant time (48 h). This poor acid corrosion resistance of the filter may be as a result of the dissolution of some elements from kaolin that made the porous sample become fragile, suggesting that it is liable to a decrease in the fraction strength. The porous filter showed little or no significant weight loss in aqueous NaOH (8-14). This is due to strong interaction between hydroxide ions and the porous sample which provide sufficient mechanical strength. For porous kaolin starch/potassium phosphate supports with porosity, the mass loss during corrosion test showed a good chemical corrosion resistance for both acid and base [27]. This finding is not commensurable compared with kaolin/sawdust support in this study. The disparity in the studies could be attributed to different materials used as supports. However, the present study supported the findings reported in the literature [17,24]. This shows that the porous sample (kaolin/sawdust) is alkali resistant and could not withstand acidic medium in term of chemical cleaning.

Conclusion

In this work, the fabricated composite pot filters from locally available materials (sawdust and kaolin) were investigated for their pollutants removal in tannery wastewater. Studies were performed on the pot filters fired at 910 °C to determine the level of porosity of porous ceramic pot filters, mechanical properties and their removal efficiencies of pollutants in tannery wastewater. The optimal ceramic membrane had the highest flexural strength (55.53 MPa) and strong basic corrosion resistance. It was established that the percentage ratio of kaolin to sawdust formulation controlled to flow rate and porosity of the pot filters. The removal of pollutants in wastewater depends on the porosity of the pot filters. It was found that formulation T8 (67% of kaolin to 33% of sawdust) showed the highest mechanical properties with the least porosity, 15.40%. The least porous filter was able to remove microorganisms efficiently and it also confirmed the highest COD, BOD, chloride, nitrate, sulphate, TDS, TSS, TH and TA removal efficiencies. The efficacy of the pot filters was slightly lowered in scrubbed pot filters than the regular filters and this could be as a result of clogging and availability of pollutants on the surfaces of filters. It will be required to investigate the water flux evolution with time at different pressure of filters and colour removal in tannery wastewater using filters. Further research is thus needed on the pilot scale for the implementation of the pot filters in the industries in order to reduce the contents of pollutants in discharged effluents Table 6.

Declaration of Competing Interest

Authors have declared that no competing interests exist.

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