Evaluation of the Efficiency of Ceramic Membrane Pots in Treatment of Food Beverages.

F.T. Owoeye, B.Eng[1]*; O.S. Olokode, Ph.D.[1]; Prof. P.O. Aiyedun[1]; S.I. Kuye, Ph.D.[1]; B.U. Anyanwu, B.Eng.[1]; H.O. Balogun, B.Eng.[1];and J.N. Nwonah, M.Sc.[2]

[1]Mechanical Engineering Department, P.M.B. 2240, Federal University of Agriculture, Abeokuta, Ogun State, Nigeria.
[2]Moshood Abiola Polytechnic Abeokuta, Ogun State, Nigeria.

E-mail: lordphems2008@yahoo.com*

ABSTRACT

Ceramic membranes provide superior thermal and chemical stability when compared to commercially available organic membranes and may find widespread use in fossil energy systems to improve the efficiency and performance of a wide range of processes. The goal of this project was to develop low-cost membranes from indigenous materials and to evaluate the exact proportion of types of ceramic membrane that can be used for food and beverages treatment with the mechanical properties of the ceramic microfiltration membrane. The four samples membranes were prepared separately, from different compositions of raw materials such as clay, kaolin, sawdust, wood charcoal as well as sodium carbonate. The tapper bucket type membranes (246 mm height, 300 mm top, 205 mm bottom and 8mm thickness) were prepared by paste casting and were shaped by wet pressing at 2.5Pa, then fired at 1200^{O}C. The chemical analysis was determined by Atomic Absorption. Spectrophotometer (AAS), X-Ray Diffractometer was determined by Radicon MD-10, version 2.00, CuKα radiation at exposure time of 1200/1200 seconds and compressibility test was determined by Instron Universal Testing Machine with Model Number of 3369. The results from the chemical analysis showed that the kaolin is composed mainly of SiO_2 and Al_2O_3, with the other oxides being present in trace amounts. The filtration rates for sample A, sample B, sample C and sample D were 52.173%, 26.522%, 17.652% and 3.653% respectively.

Our research showed that with increase in the amount of kaolin and decrease in the amount of clay, the weight of samples increased which took longer drying period. It was found that with increasing the amount of kaolin and decreasing the amount of clay, the pore diameter was decreasing. The membrane pore size and pore density were predicted directly from the particle size distribution of the clay and kaolin. Conclusively, the best two membranes among the four were sample B and that of C; and this was due to their stability in all properties observed during the course of research project and they could be utilized.

(Keywords: ceramic membrane, porosity, XRD, microfiltration)

INTRODUCTION

In the inorganic membrane markets, ceramic membrane materials are dominant, especially alumina membranes which are widely used. Ceramic membranes are especially suitable for processes with high temperatures and harsh chemical environments or for processes where sterilizability of the membrane is important. Because of this, the ceramic membranes have found many applications in the food, beverage, biotechnological and pharmaceutical industries as well as in the petrochemical industry, environmental control, electronic industry, gas separation and other process industries. In 1986, the market of membrane industry worldwide was about $1 billion. In 1989, the market of inorganic membranes was about $32 million and of ceramic membranes $19 million. Nowadays, the worldwide market of the membrane industry is about 10 billion US$ per year. In food, beverage, and biotechnology applications inorganic membranes constitute 12% of the market. The main usage (80%) of inorganic membranes is in the dairy industry [1 and 2].

Porous ceramic membranes are asymmetric with a support thickness of about 1-3 mm. The microfiltration layer is usually 10-30μm thick and the most common oxides used for the membranes are zirconia (ZrO_2) and alumina (Al_2O_3). Ultrafiltration membranes are membranes used in filtration systems which can be broadly classified into organic and inorganic filters. Organic filters, such as those made up of cellulose acetate, polyamides, and polysulfones, dominate the market today despite poorer performance in comparison to inorganic membranes in several aspects. In general, inorganic membranes, particularly those made of ceramics, offer superior chemical resistance, wider operational temperature limits, greater resistance to extreme pH conditions, higher pressure limits, longer operating lifetimes, and improved back flushing capabilities.

Despite these benefits, inorganic membranes suffer from high fabrication costs primarily due to expensive powder processing and sintering at high temperatures. Porous ceramics supports are, generally, needed for membranes manufacturing. For the development of high-quality supports, the following properties are of major importance: pore size distribution, total porosity ratio, surface quality with the absence of large defects or large pores, good mechanical properties and chemical stability [3]. In fact, the top layer is closely related to its support. In addition, the quality of the support is of crucial importance to the integrity of the membrane layers that are applied in the subsequent preparation steps. The required thickness of the membrane is further limited by the smoothness of the support because the membrane material must cover all irregularities of the support to form a continuous, defects free layer [4].

The conventional method of preparing ceramic tubes is extrusion. Nevertheless, a problem of extruded ceramic tubes may be encountered such as low surface smoothness and larger average pore sizes [5]. Consequently, an alternative method for such a support preparation has been proposed (Ceramic filtration technology and is often called "dead-end filtration" and "depth filtration"). Filtration: The most common filters are dual-media filters, in which water flows by gravity through a porous bed of two layers of granular media [6]. There are several mechanisms by which the ceramic element filters out particles as a dead-end filtration. Bridging smaller than 0.5 μm particles may be too small to be intercepted; however two particles hitting the obstruction at the same time will form a bridge across the pore adhering to each other. Bridged particles may not plug the pore creating even smaller pore gradually forming a "filter cake". This "cake" creates a finer filtration for subsequent interception at the cost of decreased flow rate and eventually no flow rate.

Ceramic depth filtration will filter out considerably smaller particles than equivalent pore size membrane for the following reasons:

Particles intercepted within the ceramic depth are much smaller than the pores measured by porometry. This is because particle laden water has to navigate through intricate maze of labyrinths. The path through the filter twists and turns trough sharp angles due to complicated ceramic structure and so the particles that may have penetrated the topmost layer become trapped within the structure [7].

MATERIALS AND METHODS

Materials

The raw materials (Clay, kaolin, wood charcoal, sawdust and the binding agent) used were collected from Abeokuta in Ogun State, South West of Nigeria. The chemical analysis and other characterizations of all raw materials and prepared samples were determined by Scanning Electron Microscopy/Optical, Atomic Absorption Spectrophotometer (AAS) and X-Ray Diffractometer were examined.

Experimental Procedures

All the samples were sun-dried for some days. The dried clay and kaolin samples were cleaned up thoroughly by removing foreign materials such as stones, dead roots and dried leaves. Four (4) the tapper bucket type membranes (246 mm height, 300 mm top, 205 mm bottom and 8mm thickness type microfiltration membranes of diameter 200 mm and thickness of 8 mm each were fabricated by paste and casting method (wet pressing also utilized) from different percentage compositions of clay, kaolin, sawdust, wood charcoal and binding agent of sodium carbonate as shown in Table 1.

Table 1: Percentages of Raw Materials Measured for Fabrication of Membranes Samples.

SP	% of Clay	% of Kaolin	% of Sawdust	% of Charcoal	Na_2CO_3
A	90	0	9.0	0	1.0
B	80	10	9.0	0	1.0
C	40	40	10.0	9	1.0
D	20	60	10.0	9	1.0

Compressibility Test

All the compressibility tests were also obtained from Engineering Materials and Development Institute (EMDI) Akure and the tests were determined by Instron Universal Testing Machine with Model Number of 3369.

Physical Properties

Percentage Apparent Porosity: In calculation for the percentage apparent porosity, all the specimens were been measured to get the initial weight (Weight in air).

$$Percentage\ Apparent\ Porosity = \frac{Soaked\ weight - Mass\ in\ air\ (g)}{Soaked\ weight - Suspended\ weight\ (g)}\ x\ 100$$

Percentage of Water Absorption: Water absorption of all samples was been determined by weight differences.

$$Water\ absorption = \frac{Wet\ weight - Weight\ in\ air\ (g)}{Weight\ in\ air\ (g)}\ x\ 100$$

Bulk Density: The bulk density of the fired membrane samples was determined by displacement of water from beaker using Archimedes principle.

$$Bulk\ density\ \left(\frac{g}{ml}\right) = \frac{Mass\ in\ air\ (g)}{Volume\ of\ water\ displaced\ (ml)}$$

Figure 1: Demonstration of Filtration Rate.

RESULTS AND DISCUSSIONS

Chemical Analysis

Table 2 showed the percentage chemical composition of the raw material used for this work. The presence of silica and alumina with percentage compositions of 46.4% and 34.0% respectively showed the high purity of the clay used.

Table 2: Chemical Composition of Clay.

Clay	% Composition
Fe_2O_3	2.49
TiO_2	1.69
Al_2O_3	34.0
P_2O_5	0.04
SiO_2	46.6
MgO	0.04
CaO	0.02
Na_2O	0.03
K_2O	0.08
MnO	ND
LOI	17.7

ND: Not Detectable

Drying Weight Samples

The heaviest sample of membrane produced was sample D due to highest percentage of Kaolin content while the least weight was that of sample A which had more of clay without any Kaolin material. The weights of all membranes produced decreased with increased in the number of days on the drying shelf. The rate of drying of the membranes decreased from sample A to sample B.

Figure1 showed the graphs of compressive stress versus compressive strain for all the bucket membranes produced. In sample A, sample B and sample C the line of graph started from -2MPa & 0mm/mm and moved to cover certain distance before rising up. For sample D it started from 0MPa and rose to 5MPa at 0.2mm of compressive strain before declined to 1MPa and maintained 0MPa throughout despite increase in compressive strain while other samples were increasing at high compressive stress.

In Table 4, sample D had the highest value of bulk density (11.056g/ml) and this was as a result of large percentage of kaolin content in the mixture as also stated by [9], while the sample A with highest content of clay had the lowest bulk density of 10.120g/ml.

Percentage of Water Absorption: The percentage of water absorption for all the membranes produced was also displayed in Figure 2, sample A that had the least of bulk density (10.120g/ml) and got the highest percentage value of water absorption (27.189%).

Percentage of Apparent Porosity: The highest value of 117.250% was also found in sample A, and this was due to high percentage of clay content in the sample preparation. Sample D of much kaolin content had the least value of apparent porosity of 71.481%

Table 3: Samples Compressibility Tests.

Samples	Compressive stress (Mpa)							
SP A		-2	2	0	5	30	120	142
SP B		-2	8	2	10	40	50	112
SP C		-2	2	1	5	20	100	154
SP D		0	5	1	0	0	0	0
Compressive strain (mm/mm)		0	0.2	0.4	2	2.2	2.4	2.6

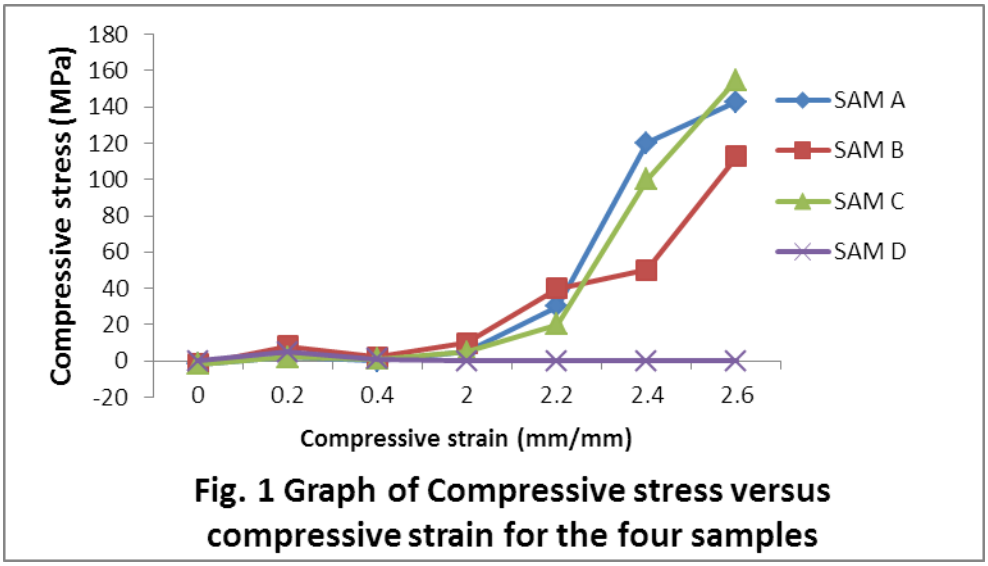


Fig. 1 Graph of Compressive stress versus compressive strain for the four samples

Table 4: Physical Properties of the Prepared Four Membranes.

Samples	Bulk Density (g/ml)	Water absorption (%)	% Apparent Porosity
SP A	10.120	27.189	117.250
SP B	10.512	26.835	73.541
SP C	10.635	25.378	84.612
SP D	11.056	23.144	71.481

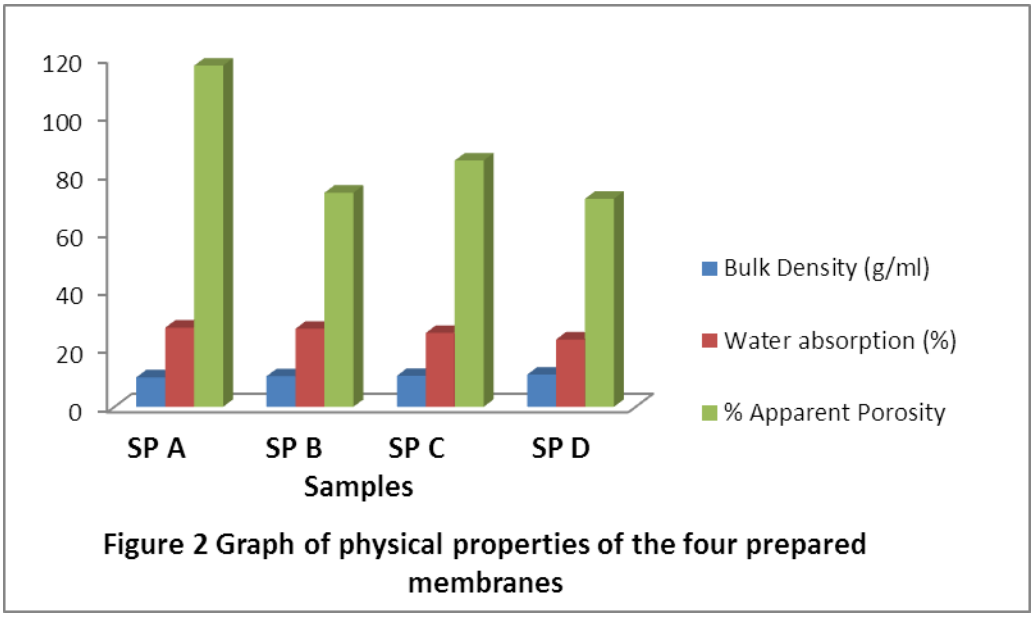


Figure 2 Graph of physical properties of the four prepared membranes

Table 5: Filtration Rate of the Four Membranes.

Samples/ Time (Hrs)	0Hr	2Hrs	4Hrs	6Hrs	8Hrs	10Hrs
SP 1	0	2500	4600	6750	9500	12000
SP 2	0	1200	2350	3500	4870	6050
SP 3	0	700	1300	2500	3150	4310
SP 4	0	400	490	520	525	540

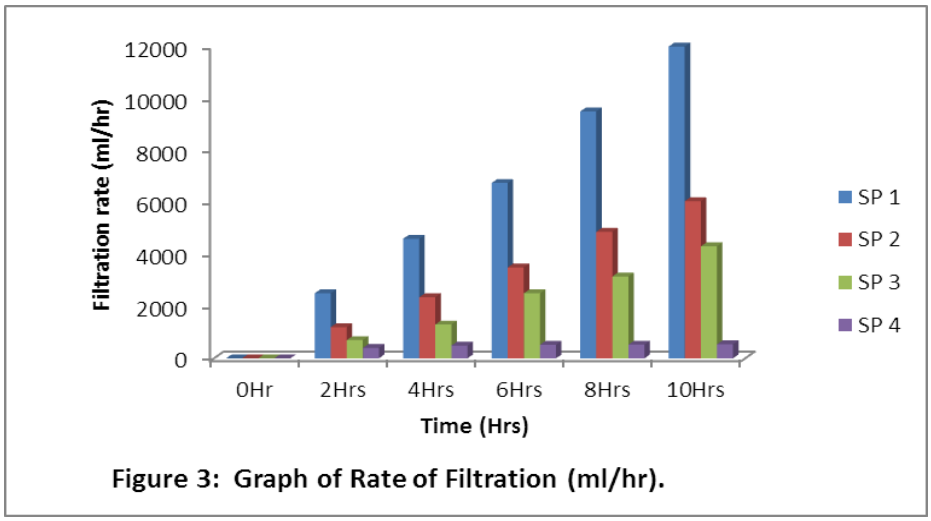
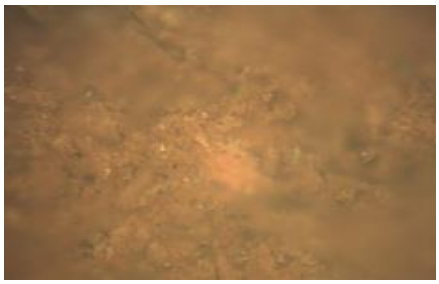


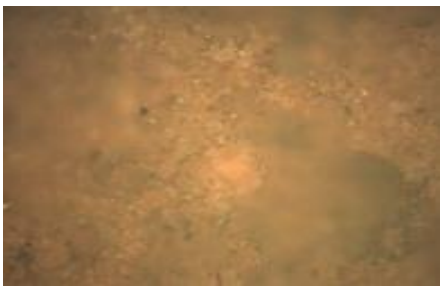
Figure 3: Graph of Rate of Filtration (ml/hr).

Scanning Electron Microscopy of Membrane Samples (SEM)

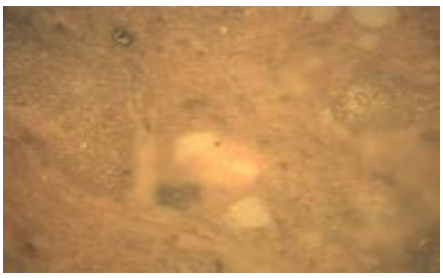
Plates 1, 2, 3, and 4 are scanning electron microscopy (SEM) for Samples A, B, C, and D, respectively. Magnifications of 200 were taken for all the sample membranes.



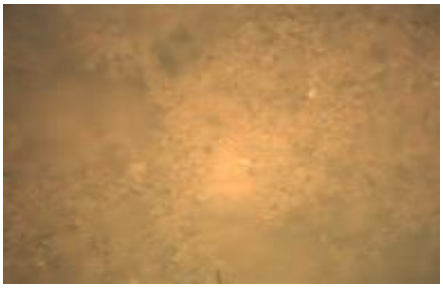
M: X200
Plate 1 Scanning Electron Microscopy of Sample A



M: X200
Plate 2 Scanning Electron Microscopy of Sample B



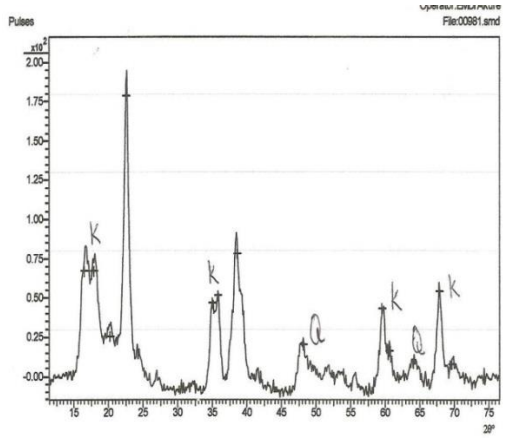
M: X200
Plate 3 Scanning Electron Microscopy of Sample C



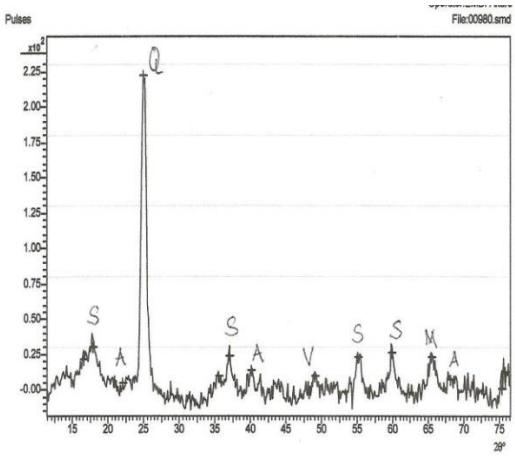
M: X200
Plate 4 Scanning Electron Microscopy of Sample D

X-RAY DIFFRACTOMETER OF SAMPLE MEMBRANES (X-RD)

Figures 3 to 6 showed the X-Ray Diffractometer (X-RD) of all the raw materials used in fabrication of membranes at different percentage of mixture while Figures 7 to 10 represented the XRD of sample A to sample D, respectively. The main minerals identified by X-Ray Diffractometer (XRD) in the kaolin deposits are kaolinite $Al_2Si_2O_5(OH)_4$, Kaolinite is the dominant and the chemical composition of the kaolin is essentially SiO_2 and Al_2O_3 [8 and 9].

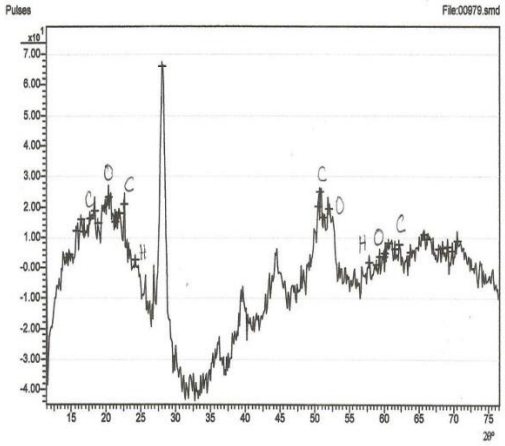


K- Kaolinite Q-Quartz
Figure 3: X-RD Kaolin

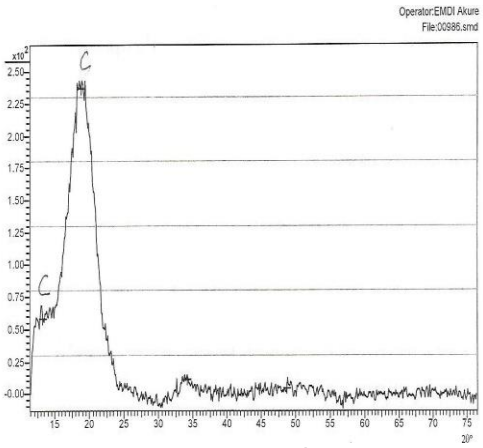


S- Silicate, A-Alumina, Q-Quartz, V-Vermicullite, M-Montmorillonite

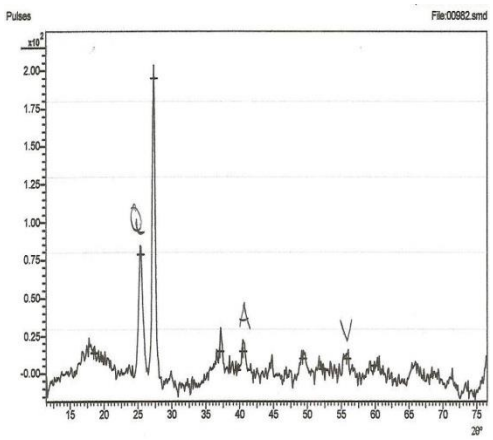
Figure 4: X-RD of Clay Powder.



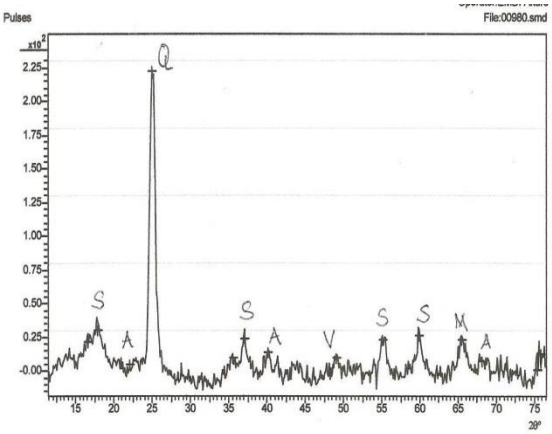
C-Carbon, H-Hydrogen, O-Oxygen
Figure 5: X-RD of Charcoal Powder.



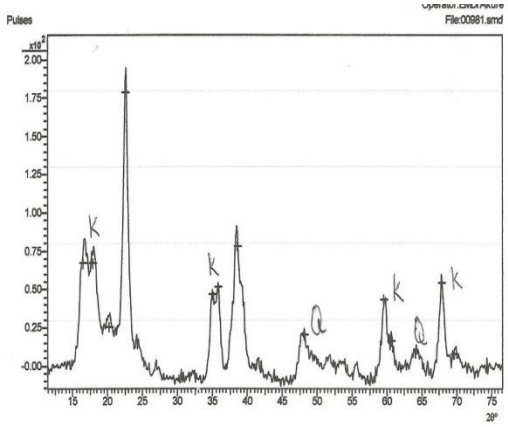
C-Carbon
Figure 6: X-Ray Diffractometer of Sawdust.



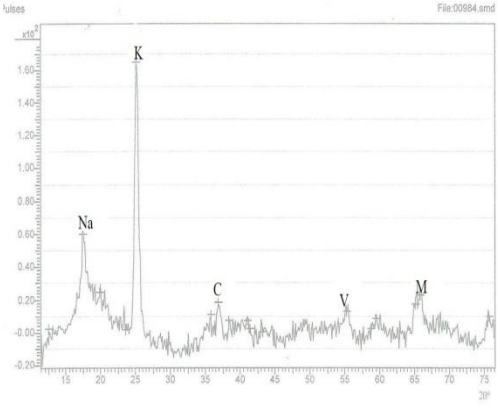
Q-Quartz, A-Alumina, V-Vermicullite,
Figure 7: X-RD of Sample A.



A-Alumina, M-Montmorillonite, Q-Quartz, V-Vermicullite, S- Silica
Figure: 8 X-RD of Sample B.



Q-Quartz, K- Kaolin
Figure 9: X-RD of Sample C.



C- Carbon V-Vermicullite, M-Montmorillonite K-Kaolin, Na- Sodium
Figure 10: X-RD of Sample D.

CONCLUSIONS

There was significant effect of kaolin content where added in the sample membranes produced; the sample D that has the highest percentage of kaolin had the highest bulk density more than other membrane samples and this was what made the sample to have high mechanical stability as a result of its densification. The scanning electron of microscopy of all the samples displayed the good quality of clay and kaolin materials use in the production of the membranes since there was no crack on any of the samples despite high firing temperature of 1200^{o}C.

It is hereby concluded that the best two membranes among the four were sample B and that of C this was due to their stability in all properties observed during the course of research project and they could be utilized.

REFERENCES

1. Hsieh, H.P. 1996. *Inorganic Membranes for Separation and Reaction*. Elsevier Science: Amsterdam, Netherlands.

2. Guizard, C. 1996. “Sol-Gel Chemistry and its Application to Porous Membrane Processing”. In: Burggraaf, A.J. and Cot, L., (eds.). *Fundamentals of Inorganic Membrane Science and Technology*.

3. Gestel, V.T., C. Dotrement, J. Luyten, R. Leysen, J. Schaep, G. Maes, and C. Vadecasteele. 1991. “Corrosion Aspects of Al_2O_3-TiO_2 Mesoporous Membranes”. *Euromembrane 99*. September 19-22, Book of Abstracts, Vol.2 Posters. Leuven, Belgium.

4. Gillot, J., D. Garcéra, R. Soria, and G. Brinkman. 1990. “New Developments in MEMBRALOX Micro- and Ultrafiltration Membranes”. *Proceeding of 5th World Filtration Congress*. June 5-8, Vol. 1, Nice, France, 155-158.

5. Gupta, B.B., B. Zaboubi, and M.Y. Jaffrin. 1993. “Scaling up Pulsatile Filtration Flow Methods to a Pilot Apparatus Equipped with Mineral Membranes”. *Journal of Membrane Science*. (80):1320.

6. Anonymous. 1994. “Filter Technology 1994”. Laboratory Unit. Mikrosuodatus, Ultrasuodatus ja Nanosuodatus.Ser.Nr. 01001/1994. Käyttöohje., Oy Metalcity Ab Ltd.: Turku, Finland.

7. Afonso, M.D. and de M.N. Pinho. 1991. "Membrane Separation Processes in the Pulp and Paper Industry". *Desalination*. (85):53-58.

8. Nyakairu. G.W.A., C. Koeberl, and H. Kurzweil. 2001. "The Buwambo Kaolin Deposit in Central Uganda: Mineralogical and Chemical Composition". *Geochemical Journal*. 35:245-256.

9. Owoeye, F.T., O.S. Olokode, P.O. Aiyedun, H.O. Balogun, and B.U. Anyanwu. 2012. "Preparation and Characterization of Ceramic Microfiltration Membrane for Water Treatment". *Pacific Journal of Science and Technology*. 13(2):28-38.

ABOUT THE AUTHORS

Mr. F.T. Owoeye, a graduate of Mechanical Engineering in Federal University of Agriculture, Abeokuta, Nigeria in 2008 with second class honors degree (B. Eng.). He is almost finished his Master's degree program. His research interests include materials (membranes) and corrosion engineering.

Dr. O.S. Olokode, is a Senior Lecturer and Acting Head of Mechanical Engineering Department, Federal University of Agriculture, Abeokuta. He holds Ph.D. degrees from Imperial College, London and Federal University of Agriculture, Abeokuta.

Prof. P.O. Aiyedun, is a Professor of Mechanical Engineering, Federal University of Agriculture, Abeokuta. He is a registered Engineer and a Fellow of the Nigerian Society of Engineers. He holds a Ph.D. in Metallurgy and Material Engineering. He was a former Dean, College of Engineering, Federal University of Agriculture, Abeokuta-Nigeria. His research interest is in the area of steel production and material engineering.

Dr. Kuye S.I, is a Lecturer in the Mechanical Engineering Department, Federal University of Agriculture, Abeokuta. She holds two Masters Degrees in both Mechanical and Metallurgical Engineering and a Ph.D. in Systems Engineering from University of Lagos.

Mr. H.O. Balogun, is a staff member of the Ministry of Transportation, Lagos State Government. He earned his Higher National Diploma from Ibadan, Post graduate Diploma from Federal University of Technology, Akure and is winding up his Master degree in the Mechanical Engineering Department of the Federal University of Agriculture, Abeokuta, Nigeria. His research interests include materials engineering.

Engr. B.U. Anyanwu, is a Senior staff in the Federal University of Agriculture, Abeokuta. He is a registered Member of the Nigerian Society of Engineers and a Registered Engineer with the Council for Regulation of Engineering in Nigeria (COREN).

Engr. J.N. Nwonah, is a lecturer in the Department of Mechanical Engineering, Moshood Abiola Polytechnic, Abeokuta. She holds a Bachelor of Engineering Degree in Metallurgical and Materials Engineering from Nnamdi Azikiwe University, Awka and a Masters' Degree in Metallurgical and Materials Engineering from University of Lagos. She is a registered Engineer with the Council for the Regulation of Engineering in Nigeria (COREN) and a member of the Nigerian Society of Engineers. Her research interests is in the area of nanotechnology and materials engineering

SUGGESTED CITATION

Owoeye, F.T., O.S. Olokode, P.O. Aiyedun, S.I. Kuye, B.U. Anyanwu, H.O. Balogun, and J.N. Nwonah. 2013. "Evaluation of the Efficiency of Ceramic Membrane Pots in Treatment of Food Beverages". *Pacific Journal of Science and Technology*. 14(1):89-97.