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Preparation and Characterization of Ceramic Nanofiltration Membrane Prepared from Hazardous Industrial Waste

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ORIGINAL ARTICLE

Preparation and characterization of ceramic nanofiltration membrane prepared from hazardous industrial waste

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Abstract

Kiln rollers, which are widely used in ceramic tiles production, are usually subjected to surface grinding to remove the contaminations. The resulted fine powder is considered useless waste and a hazardous source of environmental pollution particularly as it contains health-threatening fine free silica. In the present paper, the grind waste from kiln rollers was reused as raw material in the fabrication of nanofiltration ceramic membrane. The samples of produced ceramic membranes were formed into disks by adding 15% (by weight) organic binder solution with 2% concentration, then pressed at 35 MPa, dried and fired at temperatures range from 1100°C to 1300°C for 1 hour soaking time. It was found that the best firing temperature to produce nanofiltration ceramic membrane is 1250°C, where the ceramic membrane provides high removal of turbidity and high monovalent, divalent, and trivalent salts separation percentage.

KEYWORDS

ceramic membranes, ceramic waste recycling, kiln rollers, water filtration, water standards

1 | INTRODUCTION

Ceramic membranes are separation devices made from inorganic ceramic materials (zirconia, alumina, titania, etc). They usually consist of several layers of one or more expensive ceramic material¹ with direct bearing on the commercial ceramic membrane price.²

Preparing low-cost ceramic membranes was investigated using many types of wastes such as: fly ash,^{3,4} Coal fly ash,^{5–7} rice husk,⁸ Sawdust,^{9,10} eggshell,¹¹ and recently, kiln rollers fine waste.^{12,13}

Ceramic membranes are becoming one of the important separation techniques due to their high chemical, mechanical, and thermal stability beside their high separation efficiency. Any specific application will depend on the pore size and its distribution along the ceramic membrane. Microfiltration and ultrafiltration ceramic membranes are used in many industrial applications due to their high flux and low operating cost.¹⁴

Low-cost ceramic membranes can be used in the separation field such as oily wastewater resulting from refineries and metallurgical plants,^{15–17} clarification of juice,^{18,19} corn

syrup,²⁰ wine from raw rice,²¹ and treatment of waste water from processing sardine fish.²² On the other hand, roller kilns are mainly used in the fast-firing processes of flat ceramic ware such as ceramic tiles, flat dinnerware, and small insulating refractories. The operations of such kilns give way to several environmental problems. One of them is the need to make periodic surface grinding of the kiln rollers. These rollers are usually fabricated from high alumina clays which continuously contain glaze from tile edges or depositions of alkali salts by condensation from vapor phase. These latter salts, in turn, arise from some additions made on the original ceramic body recipe to impart some specific properties. This fine waste is usually stored in piles outdoors and constitutes a health hazard as silicon can result from its continuous inhalation.^{5,23} Some previous researches have been made concerning the utilization of this hazardous waste in the production of alumino-silicates refractories or wall and floor tiles depending on the original composition of the rollers.^{8,24,25} In that respect, Ahmed²³ found that the use of this powder depends on the firing temperature as it was used as shaped refractories on firing at 1300°C for 6 hours and as porcelain tiles on firing at 1350°C for 2 hours. Recently, Amin et al¹² and Abdallah et al¹³ have succeeded in using this waste to produce nano-sized ceramic membrane for water desalination.

The aim of the present work is to utilize this hazardous fine industrial waste in the production of stable, high active ceramic nanofiltration membrane for water separation and treatment.

In this work, the powder waste was analyzed then ceramic membrane samples were shaped in disks and fired at temperatures ranging from 1100°C to 1300°C. The prepared membranes were characterized by fully characterized by SEM, and its pore size distribution determined. Finally, the performance of the recommended ceramic membrane was tested using various salt concentrations. The values of filtrate water quality and salt separation were determined according to the decree of the Minister of Health and Population No. (458/ 2007) concerning

Egyptian standards and specifications to be met by drinking and domestic use water (In accordance with Egyptian standards²⁶).

2 | EXPERIMENTAL WORK

2.1 | Raw materials

The support ceramic membrane used throughout this study was mainly that prepared from kiln rollers hazardous, non-plastic fine waste,^{12,13,27} which was kindly supplied by Ceramica Prima Company, (Sadat Industrial City, 70 km North of Cairo). It was characterized using X-ray fluorescence (XRF) by an Axios, P analytical 2005, and wavelength dispersive (WD-XRF) sequential spectrometer for chemical composition. The raw materials mineralogical composition was defined by X-ray diffraction (XRD) Bruker D8 advanced computerized X-ray diffractometer apparatus with monochromatized CuK_α radiation, operated at 40 kV and 40 mA. Also, the thermal behavior was followed up by differential thermal analysis-thermal gravimetric analysis (DTA-TGA) by a Netzsch STA 409 C/CD apparatus with heating rate of 10°C min⁻¹. Runs were carried out in air.

Polyvinyl alcohol (PVA; Molecular weight 115 000) was used as binder kindly supplied by Oxford Company, India. Used PVA has typical TGA analysis (5°C/min, N₂), shows two regions, 300°C-325°C and 400°C-425°C, according to Figure 1. Final decomposition to gaseous species takes place at 425°C.²⁸

2.2 | Support ceramic membrane preparation

Specimens of cylindrical disk shape of diameter 50 mm and about 20 mm thick were carried out by mixing of fine waste powder with 15% (by weight) PVA binder solution with 2% concentration to increase their workability before molding. The specimens were molded by dry pressing under uniaxial

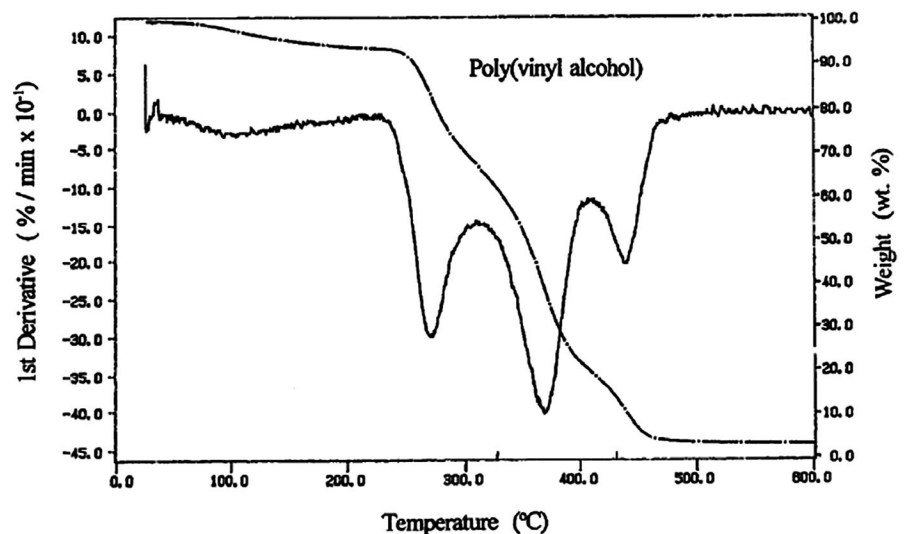


FIGURE 1 TGA of Poly(vinyl alcohol)²⁸

pressure of 35 MPa then dried overnight at $(110 \pm 5)^\circ\text{C}$. The specimens were subsequently fired using a laboratory muffle furnace, Protherm–electrical furnace model PLF 14015, under five different temperatures (1100°C , 1150°C , 1200°C , 1250°C , and 1300°C), for 1 hour soaking time at each temperature. Heating rates were operated at $10^\circ\text{C min}^{-1}$. The firing technique used is the single fast firing.

2.3 | Measurements and investigations

2.3.1 | Filtration experimental work

Filtration was performed on the laboratory filtration unit as shown in Figure 2.^{12,13} Filtration setup consists of a stainless steel plate module of 50 mm in diameter; it has three openings for feeding, concentrate, and permeates. A closed feeding tank (5 L) was used for feeding using pressure pump. In all experiments, two actual water samples, one from Mansuriyah Sluiceway, and the other from Regwa Farm Water Well has a depth of 150 m located at 76 km from Cairo on Cairo–Alexandria Road, Egypt. Table 1 illustrates the complete analysis of both Mansuriyah Sluiceway Water and Regwa Farm Water Well samples. Feeding water is continuously fed to the ceramic membrane module, at pressure 8 bars and temperature of 25°C . The both of turbidity and salt separation of the prepared ceramic membrane were studied.

2.3.2 | Fouling test

The fouling test was carried out on prepared membrane which provides highest rejection (membrane prepared under firing temperature 1250°C) to explain the flux and fouling properties. And we use humic acid in the fouling test to indicate the

efficiency of membrane separation. The fouling test was carried out for 5 hours to indicate the effect of organic materials like humic acid on the membrane performance. The distilled water was flow through the membrane for 1 hour then humic acid solution of concentration 1 mg/L flow through membrane for 1 hour finally the distilled water flow through membrane again for 1 hour. The experiments were repeated for 5 hour.

The flux recovery ratio (FRR) was calculated as follow²⁹:

$$\text{FRR}\% = \frac{J_{w2}}{J_{w1}} \times 100 \quad (1)$$

Reversible fouling ratio (R_r) and irreversible fouling ratio (R_{ir}). The resistances can be calculated by following equations, respectively²⁹:

$$R_r\% = \left(\frac{J_{w2} - J_p}{J_{w1}} \right) \times 100 \quad (2)$$

$$R_{ir}\% = \left(\frac{J_{w1} - J_{w2}}{J_{w1}} \right) \times 100 \quad (3)$$

2.3.3 | Characterization of recommended ceramic membrane

The produced ceramic membrane sample with 20 mm height and 50 mm diameter were examined and subjected to the following analyses:

1. Physical parameters like water absorption, bulk density, apparent porosity, and apparent specific gravity were studied according to ASTM C 373.³⁰ Three specimens were prepared and the average value was taken each time.
2. A pore size distribution was determined by Hg-Porosimetry method.
3. The compressive strength test was done by using UTM apparatus at a shear rate of 3 mm min^{-1} .
4. The microstructure of the produced ceramic filter surface was assessed using scanning electron microscopy (SEM) equipment that is called Quanta 250 FEG (Field Emission Gun) and attached with EDX Unit (Energy Dispersive X-ray Analyses).

3 | RESULTS AND DISCUSSIONS

3.1 | As-received Roller Kiln fine waste characterization

3.1.1 | Chemical analysis (XRF)

Table 2 depicts the results of the chemical composition of the received roller kiln fine waste. The analyzed hazardous fine

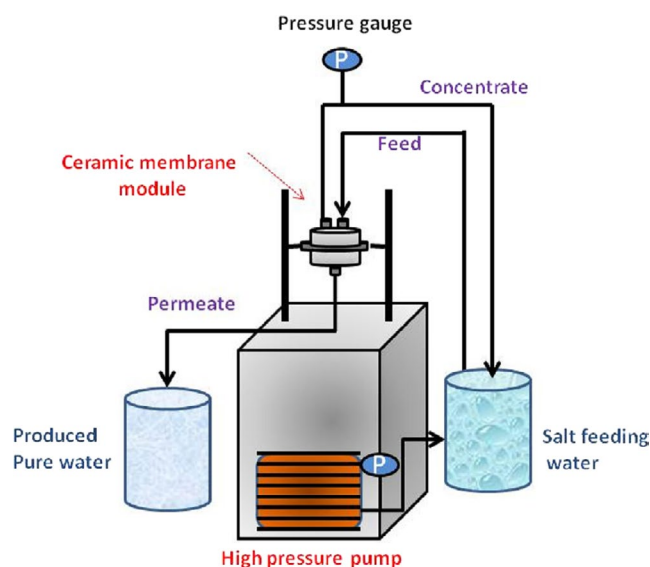


FIGURE 2 Ceramic filter laboratory testing unit [Color figure can be viewed at wileyonlinelibrary.com]

TABLE 1 Complete analysis of two types of real water samples²⁶

Element to be analyzed	Mansuriyah Sluiceway Water	Regwa farm water well	Units	Accepted limits (acc. to ES 190–1/2007)
Physical parameters				
Color	30	Colorless	mg/l Pt Co	Colorless
Turbidity	80	3	N.T.U	<1
Odor	Odorless	Odorless	Odorless	Odorless
Physicochemical parameters				
pH	7.85	7.55		6.5–8.5
Conductivity	2400	6670	μS/cm	<1200
Total hardness as (CaCO ₃)	460	1700	mg/L	<500
Calcium (Ca ⁺⁺)	110	420	mg/L	<140
Magnesium (Mg ⁺⁺)	53	190	mg/L	<36
Bicarbonate (HCO ₃ [−])	322	225	mg/L	—
Total alkalinity as (Ca CO ₃)	264	184	mg/L	<120
Bicarbonate alkalinity as (Ca CO ₃)	264	184	mg/L	—
Carbonate alkalinity as (Ca CO ₃)	0	0	mg/L	—
Hydroxide alkalinity as (Ca CO ₃)	0	0	mg/L	—
Sodium [Na ⁺]	340	2190	mg/L	<200
Potassium [K ⁺]	28	72	mg/L	<10
Chloride [Cl [−]]	480	1852	mg/L	<250
Sulfate [SO ₄ ^{−−}]	350	1200	mg/L	<250
Total dissolved solids [TDS]	1500	4308	mg/L	<1000
Undesirable substances				
Ammonium [NH ₄ ⁺]	0.21	0.17	mg/L	<0.50
Nitrates [NO ₃ [−]]	32.55	12.53	mg/L	<45
Nitrite [N]	0.065	0.050	mg/L	<0.060
Phosphate [PO ₄]	0.43	0.36	mg/L	<0.40
Silica [SiO ₂]	15.2	16.28	mg/L	—
Iron [Fe ⁺⁺]	0.67	0.11	mg/L	<0.30
Manganese [Mn ⁺⁺]	0.13	0.08	mg/L	<0.40
Copper [Cu ⁺⁺]	0.03	0.05	mg/L	<2.0
Zinc [Zn ⁺⁺]	0.50	0.34	mg/L	<3.0
Total chlorine	<0.02	<0.02	mg/L	—
Free chlorine	<0.02	<0.02	mg/L	—
Combined chlorine	<0.02	<0.02	mg/L	—
Cyanide [CN [−]]	<0.001	<0.001	mg/L	<0.05

waste powder shows a chemical composition that is in good agreement with its XRD analysis.^{12,13,27} Table 2 indicates that the Alumina and silica is the main components in the powder waste. Also, an almost zero loss on ignition was observed.

3.1.2 | Mineralogical analysis (XRD)

Figure 3 illustrates the mineralogical analysis of the roller kiln fine waste powder, which shows that: the fine waste

TABLE 2 Chemical analysis of received roller kiln fine waste

Constituents (wt%)	Kiln rollers waste powder
SiO ₂	23.55
Al ₂ O ₃	63.86
Fe ₂ O ₃	1.16
TiO ₂	0.33
ZrO ₂	3.45
CuO	0.22
ZnO	0.22
MgO	1.96
CaO	3.51
Na ₂ O	0.8
K ₂ O	0.48
HfO ₂	0.18
Co ₃ O ₄	0.12
P ₂ O ₅	0.17
L.O.I.	0.00
Total	100.01

powder consists of four different phases mullite ($\text{Al}(\text{Al}_{1.272}\text{Si}_{0.728}\text{O}_{4.864})$), corundum (Al_2O_3), gehlenite ($\text{Ca}_2\text{Al}_2\text{SiO}_7$), and anorthite ($\text{CaAl}_2\text{SiO}_8$). Trace elements did not, however, appear on XRD because of their low percents.^{12,13,27}

3.1.3 | Thermal analysis (DTA and TGA)

Received kiln roller fine waste was subjected to a DTA and TGA, in order to study the decomposition effects. The thermal analysis obtained on heating at 1000°C of the waste powder in air at a rate of 10°C min⁻¹.

Thermal analysis (DTA and TGA) for hazardous fine waste powder is illustrated in Figure 4. The results indicate that there is a slight exothermic activity due to oxidation of minor organic impurities which explains losses in the range of 110°C–170°C. The weight increases from 4.1 mg at 300°C to 4.35 mg at 1000°C that is probably because of the sloping base line. The results of TGA expect in view of the inert nature of its constituents (quartz + mullite).^{12,13,27}

3.2 | Application to water filtration

3.2.1 | Effect of firing temperature of the prepared ceramic membranes on water turbidity

Turbidity is the amount of cloudiness in the water. It is an indication of water quality such as organisms which can cause disease. High turbidity means high organism level such as viruses, parasites, and some bacteria.

Turbidity separation was studied according to the firing temperature of the membrane using 2% PVA with soaking time of 1 hour. Here, water turbidity was studied using two different samples of water, well water and sluiceway water.

Figure 5 indicates that the maximum separation can be obtained at a firing temperature of 1250°C. Using such membranes, the turbidity of sluiceway water decreased from 80 N.T.U. down to 0.85 N.T.U. Similarly, the turbidity of well water decreased from 3 N.T.U. to reach 0.03 N.T.U when using a ceramic membrane fired at 1250°C.

Regular gradient in the pore sizes in the different membranes led to the reduction of water turbidity to about 1% of its original value. This is mainly due to the nano-metric size of membranes fired at 1250°C.

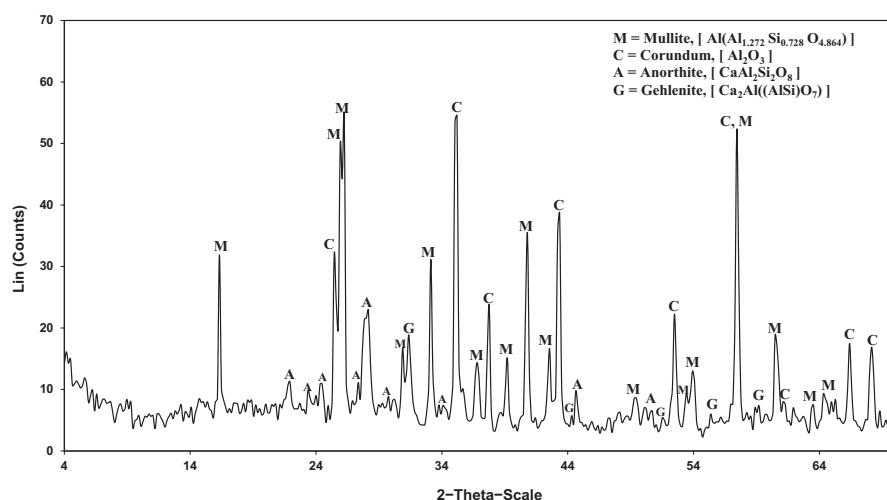
**FIGURE 3** XRD pattern of received roller kiln fine waste

FIGURE 4 DTA-TGA pattern of received roller kiln fine waste

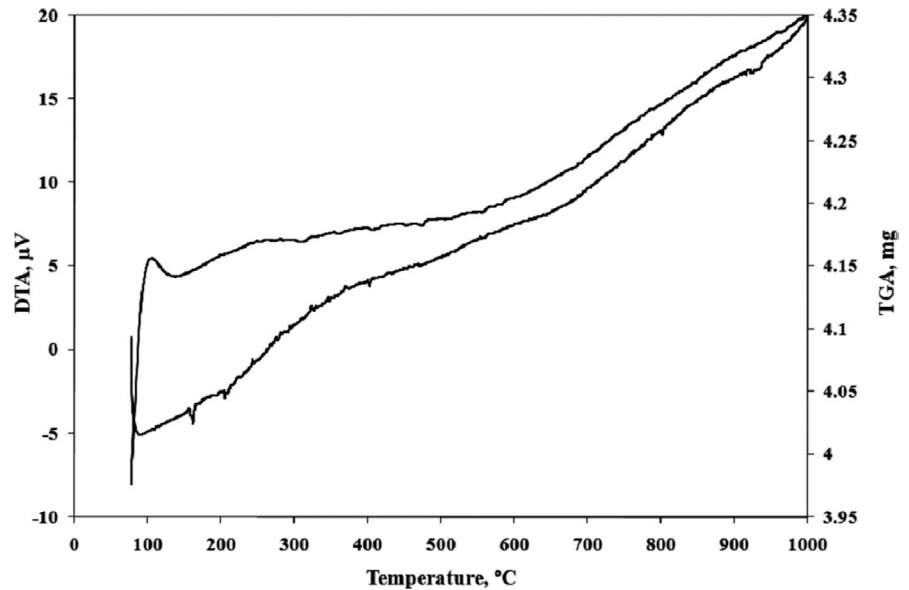
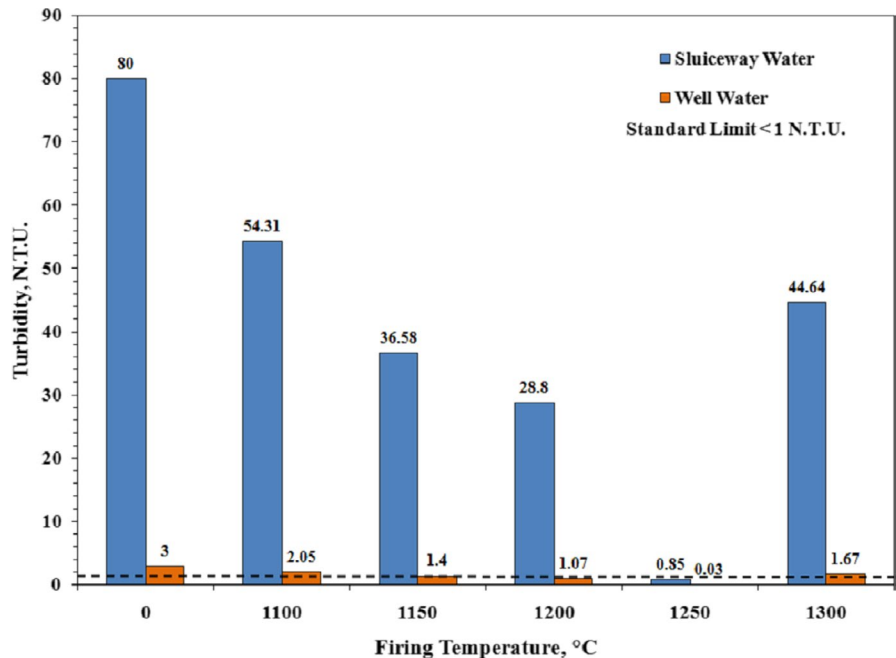


FIGURE 5 Effect of firing temperatures of prepared ceramic membranes vs turbidity



3.2.2 | Effect of firing temperature of the prepared ceramic membranes on salt separation

Salt separation from actual water samples; well water and sluiceway water was also studied using membranes prepared with 2% concentration of PVA as additive binder and soaked for 1 hour at different firing temperatures.

The conductivity of these water samples is illustrated in Figure 6. The results indicate that the conductivity of sluiceway water decreased from 2400 $\mu\text{S}/\text{cm}$ to 361 $\mu\text{S}/\text{cm}$ as the firing temperatures of the membranes used increased from

1100 to 1250°C. Owing to decreased porosity, due to excessive glass formation, the separation performance decreased on using a membrane fired at 1300°C. The highest level of salt removal was thus obtained on using a ceramic membrane fired for 1 hour at 1250°C. Well water was more saline than sluiceway water and here also, the best removal of salt was achieved on using a ceramic membrane prepared by firing at 1250°C, whereby the conductivity decreased from 6670 $\mu\text{S}/\text{cm}$ to 1000 $\mu\text{S}/\text{cm}$ on rising temperature to increase once more when a membrane fired at 1300°C was used.

Next, the removal of various ions from water (mono, di, and trivalent ions) was investigated. The two water samples

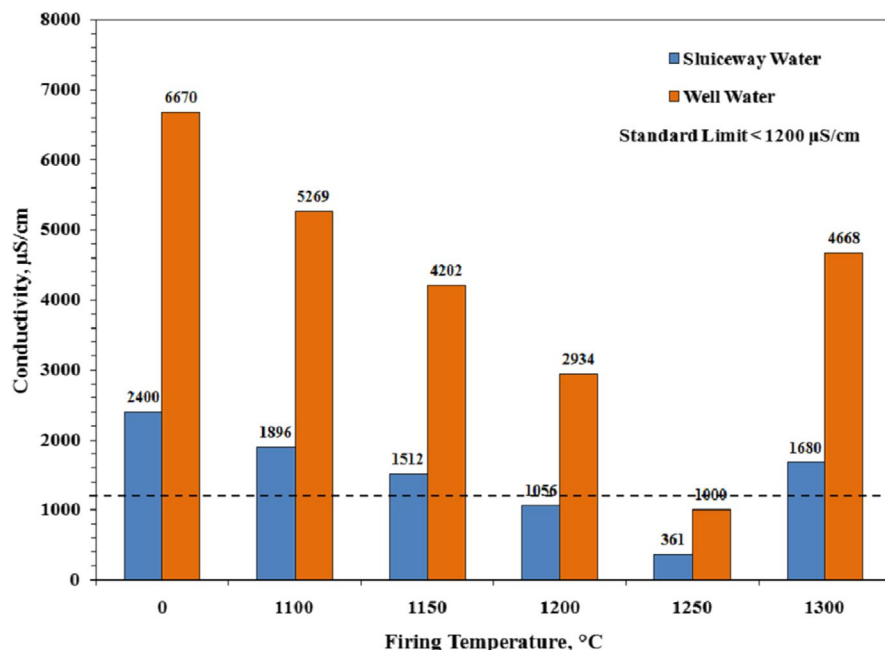


FIGURE 6 Effect of firing temperatures of prepared ceramic membranes vs conductivity of water

from well and sluceway were used in the experiments. Figure 7 indicates the effect of firing temperatures of prepared ceramic membranes on monovalent sodium ions removal, where the concentration of sodium ions decreased from 2190 ppm in well sample to 322 ppm when a membrane fired at 1300°C was used, corresponding to 85.3% Na^+ removal. Also, when using the same ceramic membrane to treat sluceway water the removal reached 85% Na^+ .

Figures 8 and 9 indicate the effect of firing temperatures of prepared ceramic membranes on monovalent potassium ions and chlorine ions removal, where the removal of potassium

ions amounted to 86.5% K^+ and that of Cl^- to 85.5% using the same ceramic membrane referred to above.

On the other hand, Figures 10–12 indicate the effect of firing temperatures of prepared ceramic membranes on divalent ions such as calcium, magnesium, and sulfate, where the concentration of calcium ions decreased from 420 ppm in well sample and 110 ppm in sluceway water to 69 and 18 ppm respectively a ceramic membrane prepared by firing at 1250°C as shown in Figure 10. This corresponds to 84% Ca^{++} removal approximately. Likewise, the Mg^{++} concentration decreased on using the same membrane by 86.2%

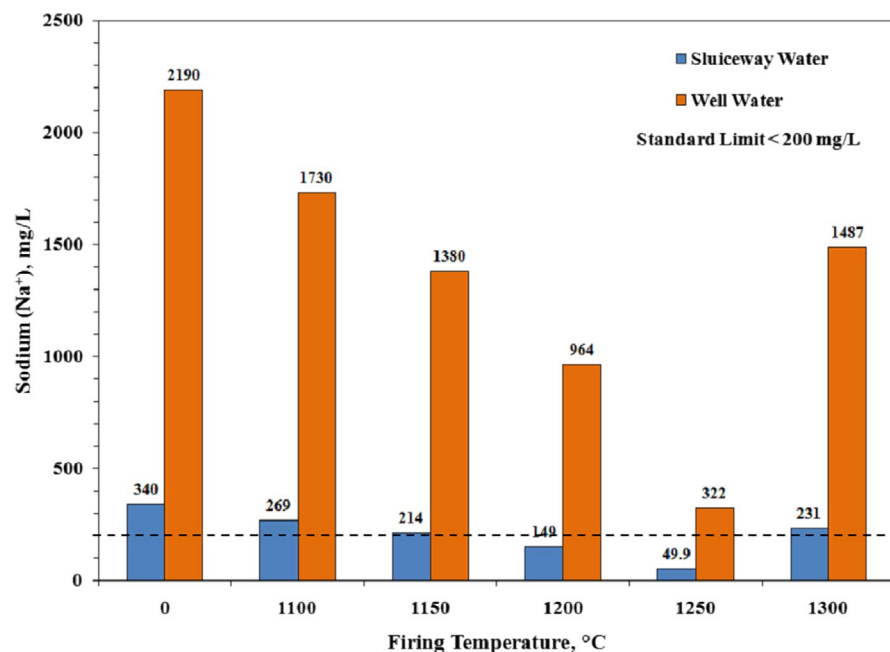


FIGURE 7 Effect of firing temperatures of prepared ceramic membranes on monovalent sodium ions removal]

FIGURE 8 Effect of firing temperatures of prepared ceramic membranes on monovalent Potassium ions removal

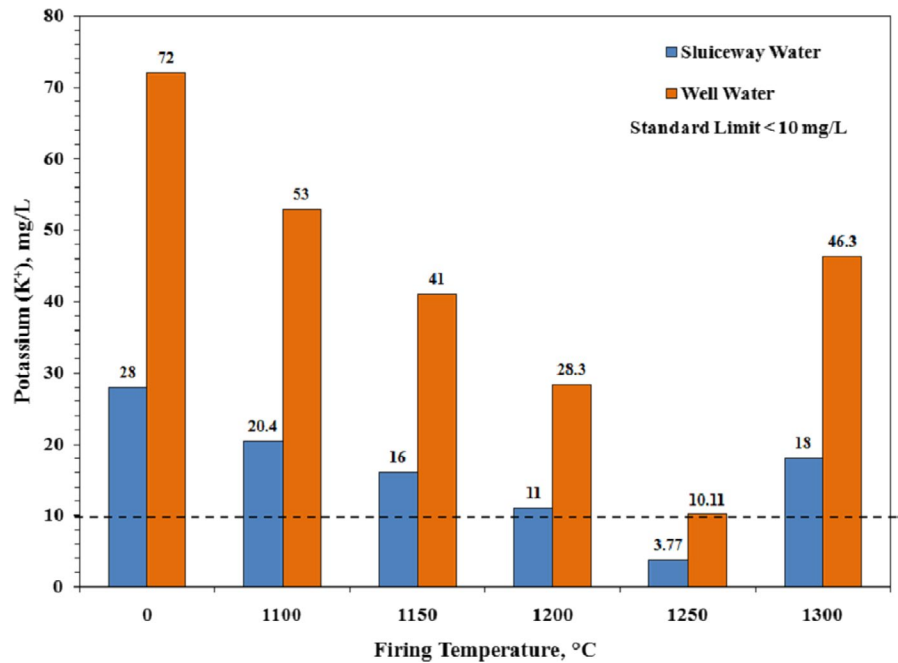
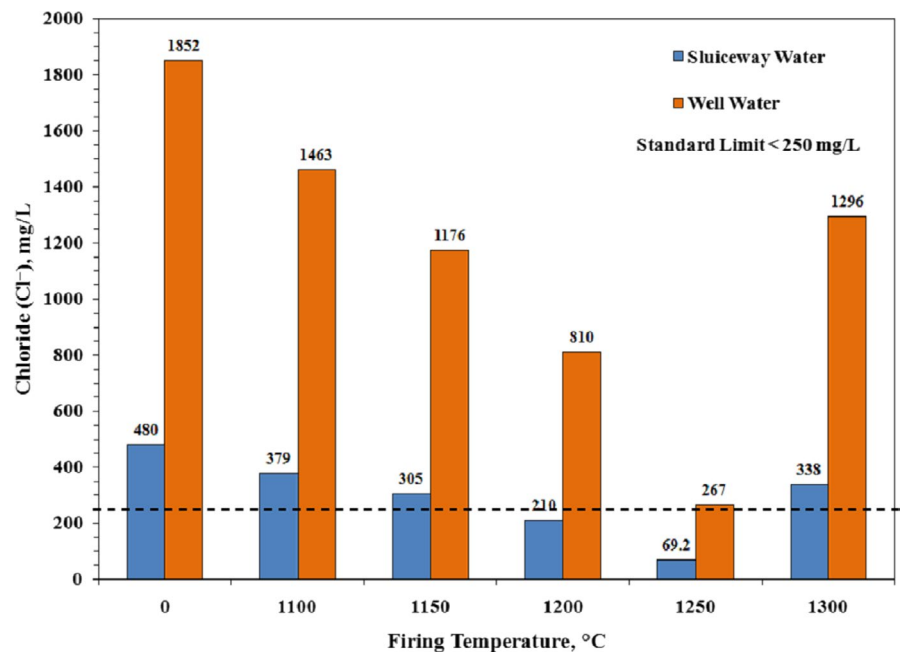


FIGURE 9 Effect of firing temperatures of prepared ceramic membranes on monovalent chloride ions removal [Color figure can be viewed at wileyonlinelibrary.com]



for both water samples as shown in Figure 11. Also, Figure 12 indicates that the concentration of sulfate ions decreased using the aforementioned ceramic membranes by about 87% for both water samples.

The removal of total dissolved solids also was investigated as illustrated in Figure 13, which reveals that the TDS decreased using the prepared ceramic membranes reaching a minimum value on using a membrane fired at 1250°C. The removal percentage reached 84.3% approximately for the two water samples.

The results indicate that the effectiveness of separation of ceramic membranes prepared at different firing temperatures

depend on the pore size distribution. Each membrane can be used in turbidity removal and salt filtration or removal at different kind of salt ions as shown in previous results. The prepared ceramic membranes at firing temperature from 1100 to 1150°C can be considered as microfiltration ceramic membranes while the prepared ceramic membranes at firing temperature from 1200°C to 1250°C can be viewed as nanofiltration membranes. The prepared ceramic membrane at 1250°C provides highest turbidity and salt removal.

In all cases, raising the firing temperature to 1300°C had for effect to start vitrification that has for effect to close the available pores, thereby reducing the effectiveness of the

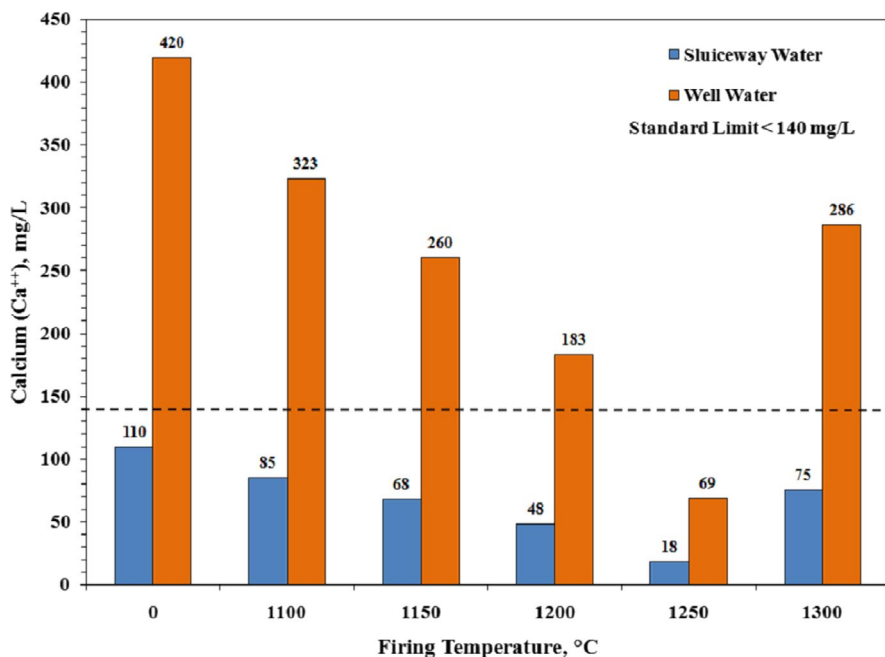


FIGURE 10 Effect of firing temperatures of prepared ceramic membranes on divalent ions Ca^{++} removal [Color figure can be viewed at wileyonlinelibrary.com]

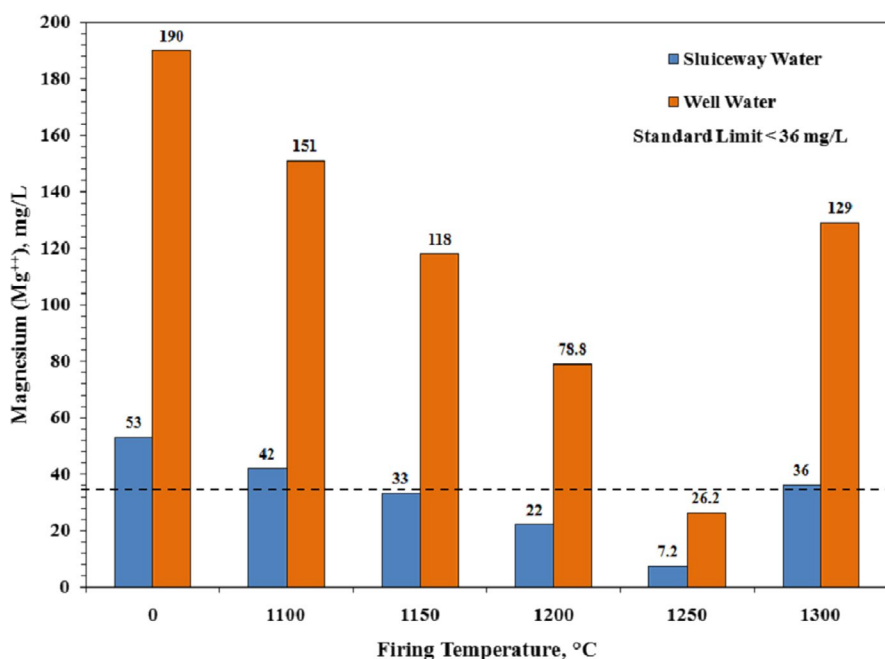


FIGURE 11 Effect of firing temperatures of prepared ceramic membranes on divalent ions Mg^{++} removal [Color figure can be viewed at wileyonlinelibrary.com]

filtration process as evidenced by the lowering in the removal values for all investigated ions.

3.3 | Fouling test

The fouling test was carried out on prepared membrane which provides highest rejection (membrane prepared under firing temperature 1250°C) to explain the flux and fouling properties. Using 1 mg/L humic acid concentration, the distilled water was flow through the membrane for 1 hour to calculate J_{w1} , then humic acid solution of concentration 1 mg/L flow

through membrane for 1 hour to calculate J_p and finally the distilled water flow through membrane again for 1 hour to calculate J_{w2} . The experiments were repeated for 5 hour. The results indicate that the permeate flux of distilled water was 109 L/m² h after 1 hour. While, the flux reduced to 77.9 L/m² h after 1 h from using humic acid solution. The fouling test indicates the reversible fouling (R_r) was 16%, irreversible fouling (R_{ir}) was 10% and flux recovery was 89.5%, that indicate the prepared ceramic membrane is antifouling membrane, where the fouling can be easily separated by washing. Figure 14 indicates the results of fouling test for 5 hour.

FIGURE 12 Effect of firing temperatures of prepared ceramic membranes on divalent ions SO_4^{--} removal [Color figure can be viewed at wileyonlinelibrary.com]

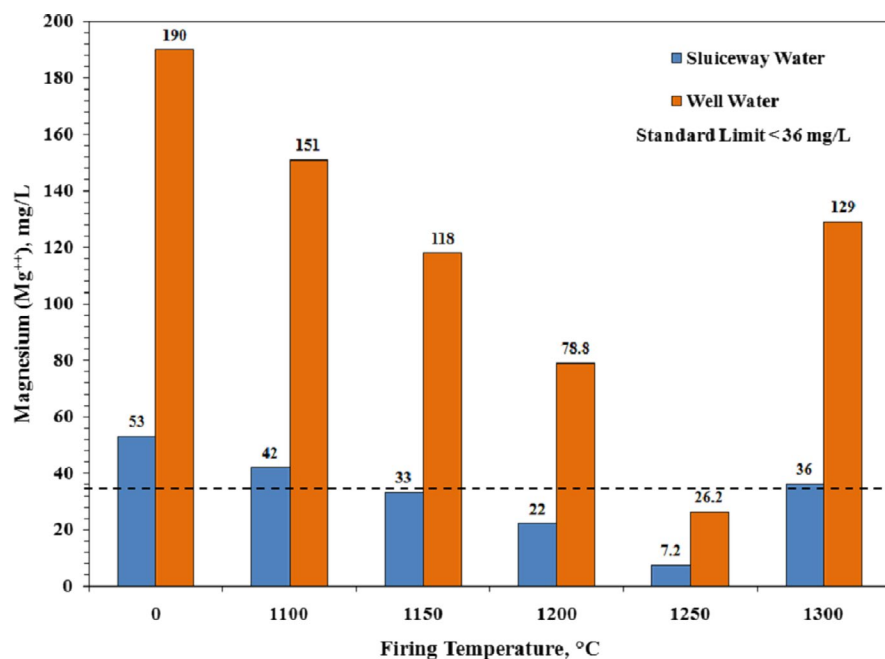


FIGURE 13 Effect of firing temperatures of prepared ceramic membranes on total dissolved solids removal [Color figure can be viewed at wileyonlinelibrary.com]

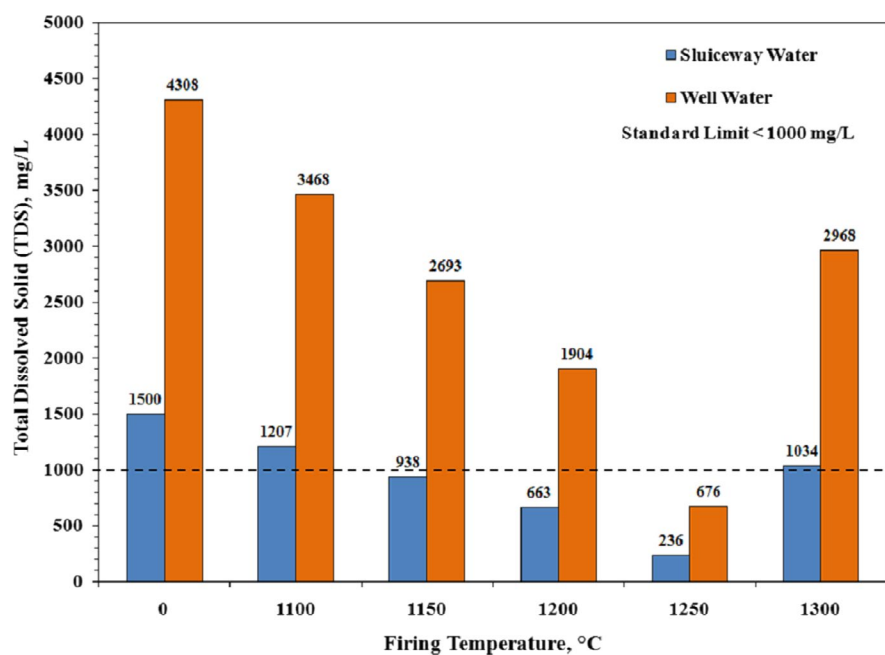
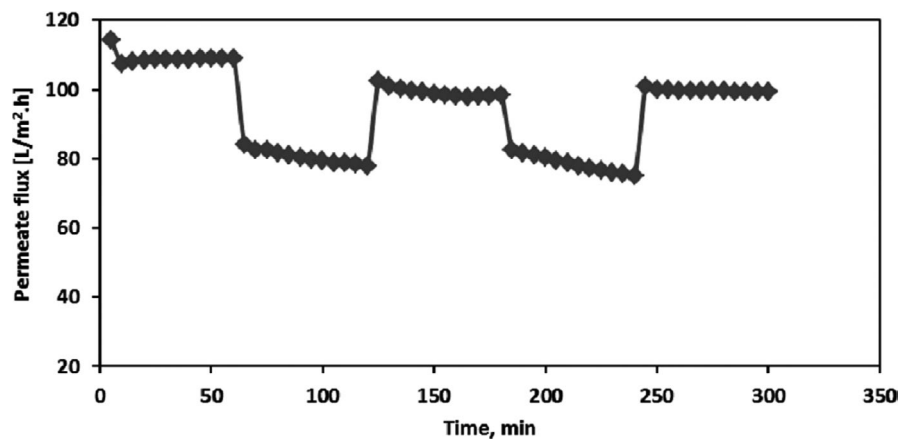


FIGURE 14 Fouling test of ceramic membrane



3.4 | Properties of the recommended ceramic membrane

The vitrification parameters of recommended ceramic membrane sample which was fired at 1250°C for 1 hour soaking time was measured according to ASTM C 373.³⁰ Alternatively, the mercury porosimeter was used to determine the pore size distribution, average pore diameter, total porosity, and total pore area. Their results indicated that the values of pore size for the prepared membrane range from (0.1-0.005) μm suggesting its potential use in nanofiltration (0.005-0.01) μm and ultrafiltration applications (0.01-0.1) μm . Table 3 summarized the properties of recommended ceramic membrane.¹³

The pore size distribution was done as shown in Figure 15 there are pores less than 0.01 μm (10 nm) which in the range of the nanofiltration membranes, where the range of nanofiltration from 10 to 1 nm. In our membrane the average

TABLE 3 Properties of recommended ceramic membrane

Properties	Unit	Value
Water absorption	%	28.61
Bulk density	G cm ⁻³	1.67
Apparent porosity	%	47.73
Closed porosity	%	1.27
Total porosity	%	49
Apparent specific gravity	—	3.19
Total pore area	m ² g ⁻¹	8.318
Average pore diameter	μm (nm)	0.0905 (90.5)

pore size was 90 nm (in a total area) which in the range of ultrafiltration but already the membrane has pores smaller than 90 nm and others may be larger than 90 nm depending on the ceramic membrane fabrication method. However, the prepared membranes separate salts that due to the electrostatic charges between membrane surface, which is mostly positive according to the highest concentration percentage of alumina, where the charge density and charge distribution control the ionic transport in the membrane.

Also, the charge groups positive or negative on the surface of membrane leads to an increase in the electrostatic interaction phenomenon between the dissolved ions and membrane surface, which called counter ion site binding.³¹⁻³³ The rejection of salts was occurred according to the electrostatic repulsion of cations due to the positive charge of membrane, which means the cations are thrown toward the feeding solution while the anions retain on the upstream solution of membrane to keep the electroneutrality, which agreement with the mechanism of the Donnan exclusion retention.^{34,35}

Furthermore, the recommended membrane was subjected to compression loading. It withstood a maximum stress of 25 MPa without failure. This reveals that such membrane can be safely used under common water pressures. Finally, the SEM micrograph for recommended ceramic membrane is displayed in Figure 16. It shows a lot of open pores, which are smaller in size, typical of appreciable sintering setting in when firing at 1250°C. The reduction in porosity (Figure 17) is mainly affected by the firing temperature the increase of which promotes sintering and vitrification.¹³

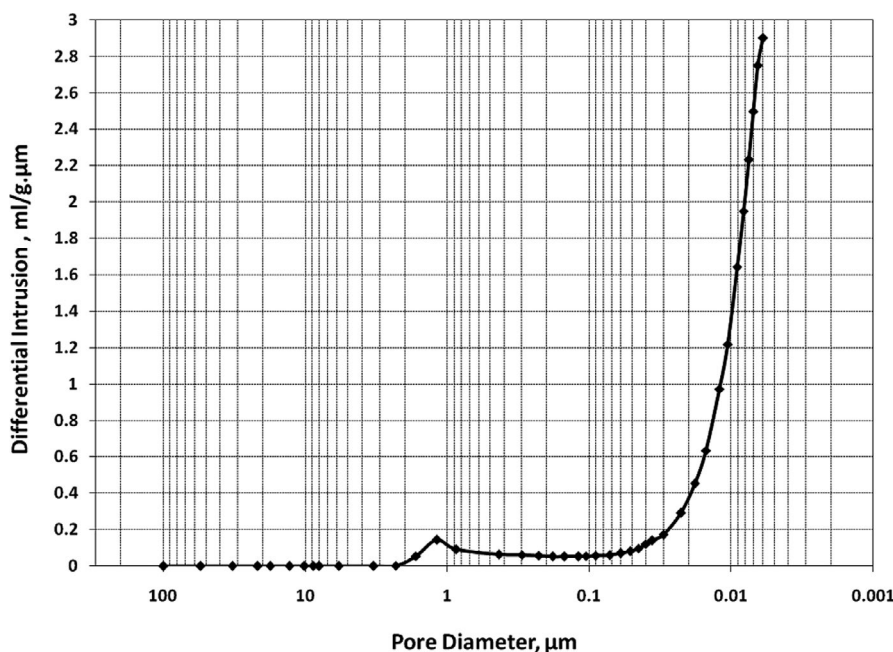


FIGURE 15 The pore size distribution for ceramic membrane fired at 1250°C

FIGURE 16 SEM micrograph for recommended ceramic membrane fired at 1250°C

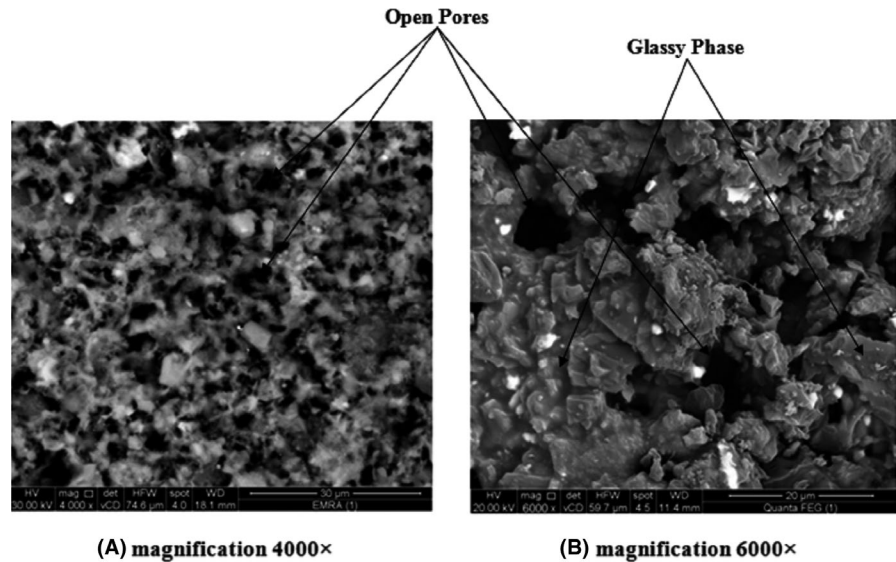
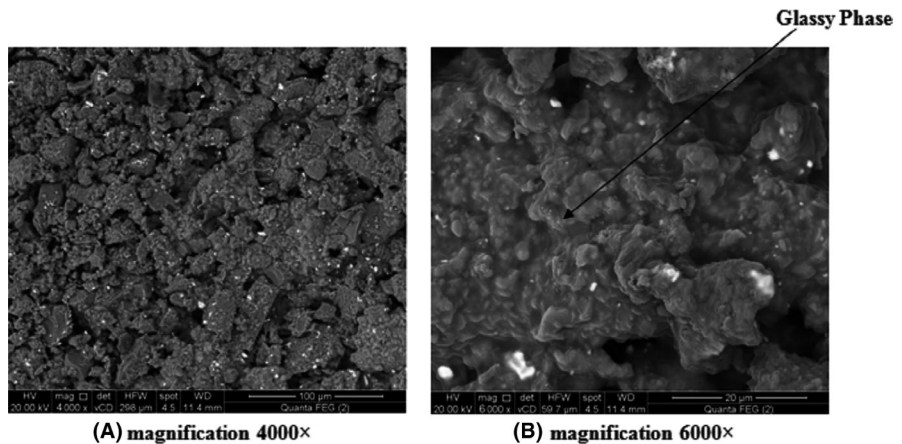


FIGURE 17 SEM micrograph for ceramic membrane fired at 1300°C



4 | CONCLUSION

Hazardous waste from industrial roller kiln was used in the preparation of ceramic nanofiltration membranes for water filtration. The characterization of wastes indicates that it have high amount of alumina and silica. The results indicate that, the prepared ceramic membranes at firing temperature from 1100°C to 1150°C can be considered for use as microfiltration ceramic membranes while the ceramic membranes fired at 1200°C to 1250°C can be considered for use as nanofiltration membranes. The highest removal of both turbidity and salt is exhibited by using ceramic membrane fired at 1250°C.

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