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Produced Water Treatment Using The Residue Catalytic Cracking (RCC) Spent Catalyst As Ceramic Filter Material Integrated With Reverse Osmosis (RO) System

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A cylindrical tube shape ceramic filter made from a mixture of the residue catalytic cracking (RCC) unit spent catalyst, natural clay, and gadung (Dioscorea hispida Dennst) starch was employed for produced water (PW) treatment. The parameter of produced water characterized were total dissolved solids (TDS), phenol, and barium concentration. Two types of ceramic filters were made using activated, and non-activated RCC spent catalysts in various compositions. Produced water transferred to the ceramic filter at operating times of 30, 60, 90 min, respectively, and applied pressures into the ceramic filter were set as 2.3, 2.5, and 2.7 kg/cm2 followed by a reverse osmosis (RO) membrane. The results showed that at contact time 90 min and applied pressure 2.7 kg/cm2 ceramic filter D (25% activated RCC, 70% natural clay, 5% gadung starch) could reduce TDS, phenol, and barium by 43.21%, 68.69%, 26.21%, respectively and followed by 86.68% TDS, 82.40% phenol, and 93.33% barium in RO permeate. Different results were obtained using ceramic filter B (70% activated RCC spent catalyst, 25% natural clay, and 5% gadung starch) could reduce TDS, phenol, and barium as 34.11%, 71.84%, and 38.30%, respectively and in RO permeates were 67.93%, 91.77%, and 82.92%, respectively. From these results, activation of the RCC spent catalyst will increase the total removal of TDS, phenol, and barium from PW. The ceramic filter made from the activated RCC spent catalyst could be a pretreatment before RO processes.

Keywords: Ceramic filter, Clay, Produced water, Reverse osmosis, RCC Spent catalyst

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1. Introduction

Most of the wastewater generated during oil and gas production is produced water (PW). It combines formation water and water injected into the reservoir to enhance oil and gas recovery. PW from oil and gas reservoirs accommodates various contaminants, such as oil and grease, dissolved minerals, chemical compounds, and dissolved solids [1]. In addition, the produced water contains high concentrations of metal ions and hydrocarbon compounds. These contents can be harmful and hazardous to the environment and human health. Therefore, produced water must comply with predetermined quality standards before being discharged into the water bodies. PW usually has high total dissolved solids (TDS), phenol, and barium concentration. TDS in PW is a problem for most oil and gas reservoirs. Barium is the most common and abundant ion found in PW from oil and gas reservoirs [2]. It is suggested that the concentration of barium ions in produced water primarily in the form of barium sulfate is a strong indication of radium isotopes [3].

Table 1. Ceramic Filter Composition.

Ceramic Filter	RCC Spent Catalyst (%wt)		Natural Clay (%wt)	Gadung Starch (%wt)
	Without Activation	With Activation		
A	70	-	25	5
B	-	70	25	5
C	25	-	70	5
D	-	25	70	5
E	47.5	-	47.5	5
F	-	47.5	47.5	5

The application of membranes in water and wastewater separation has increased in the last decade. Separation using membranes has advantages such as no need for additional chemicals, low energy requirements, lower sludge production, and producing high-quality permeate. There are mainly two membrane types applied in water and wastewater: ceramic and polymeric membranes. Ceramic membranes have an advantage because of their superior thermal and chemical stability and can be fabricated using a wide range of metal oxide [4]. The residue catalytic cracking unit in the oil refinery uses metal oxide-based catalysts in crude oil cracking.

The amount of RCC unit spent catalyst in one of oil and gas industry in South Sumatra has been estimated as 15.98 tons per year, and 10.30 tons is only stored in a toxic and hazardous waste and warehouse [5]. RCC spent catalysts are mainly composed of silica and alumina, which are the most common ceramic materials used to make ceramic filter [6]. Therefore, this RCC spent catalyst potential for reuse as an adsorbent in PW treatment.

In the ceramic filter preparation, adding substances that function as pore formers and binding agents is necessary. The pore-forming material and binding agent used in this research is *Dioscorea hispida Dennst* starch. Dioscorea hispida is locally known as gadung in Indonesia. Starch is widely used to produce porosity in ceramics because it will form pores during combustion at around 500 °C [7].

RO membranes can remove most compounds in water and wastewater. It has an advantage such as no chemicals in operation, relatively low energy consumption, easy to handle, and maintenance. RO technology can also be applied in the PW treatment to reduce the high TDS concentration [8, 9]. Several researchers have carried out studies to evaluate the effectiveness of RO membrane for PW treatment. For instance, the removal efficiency of the RO membrane in the acidity range of 4-9 by RO in the PW treatment shows the overall phenol, TDS, and barium removal efficiency as 99.7%, 71.4%, and 90.67%, respectively [10, 11]. In their simulation, Piemonte et al. (2015) showed that the RO process decreased TDS to the limits for reuse and suggested the viability of the RO to treat PW for high-quality water [12]. In the current work, a ceramic filter from the RCC spent catalyst is used in combination with RO to treat produced water. The used of a stand-alone RO membrane in produced water treatment will easily foul the membrane. Therefore the used of ceramic filter in pretreatment is an alternative to prevent the RO membrane from fouling (as the pretreatment).

2. Materials and methods

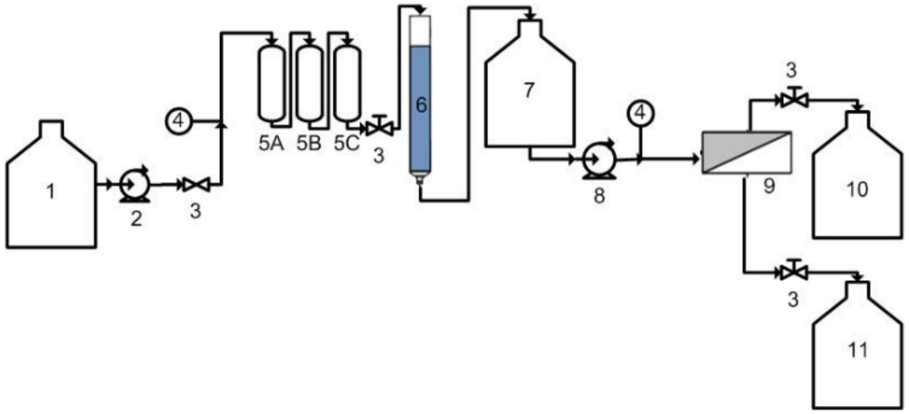
2.1. Materials

The PW is provided from an oil and gas industry in South Sumatra, Indonesia. PW characterization showed value of phenol (9.88 mg/L), acidity (7.80), TDS (12,500 mg/L), barium (5.07 mg/L), chemical oxygen demand (28 mg/L), oil and grease (0.4 mg/L), total ammonia (3.98 mg/L) and sulfide (0.236 mg/L).

The RCC spent catalyst with a particle size of 100 mesh was activated using 1 M hydrogen chloride (HCl), washed with demineralized water until neutral, and then dried overnight in the oven at 110 °C. The ceramic materials used in this research were the RCC unit spent catalyst, natural clay, and gadung (Dioscorea hispida) starch. Ceramic filter was designed as the cylindrical tube filter with an outer diameter of 6 cm, an inner diameter of 3 cm, and a length of 25 cm. The RCC spent catalysts was supplied by oil and gas in South Sumatra. The natural clay comes from Talang Jambe, South Sumatra, Indonesia, and gadung starch purchased from a local supplier in Bantul, Yogyakarta, Indonesia. The materials for the experiment were used without any further purification. The ceramic filter is placed in the propylene cartridge filter housing. The commercial CSM-RO membrane-type RE 4040 BE with an effective area was 7.9 m^2 was employed in the experiment. A scanning electron microscope with Energy Dispersive X-ray (SEM-EDX) type Vega 3 was applied to examine the ceramic filters' surface and morphology.

2.2. Ceramic filter composition

Ceramic filter composition can show in Table 1.



1. Feedwater tank
2.Pump
3. Valve
4.Pressure Gauge
5A. Filter spoon (0.5μm)
5B. Filter spoon (0.3µm)
5C. Filter spoon (0.1µm)
6. Ceramic Filter Housing
7. Filtrate Storage Tank
8. RO Pump
9. Reverse Osmosis (RO) Membrane
10. RO Concentrate Tank
11. RO Permeate Tank

Fig. 1. Produced water treatment using Ceramic Filter–RO.

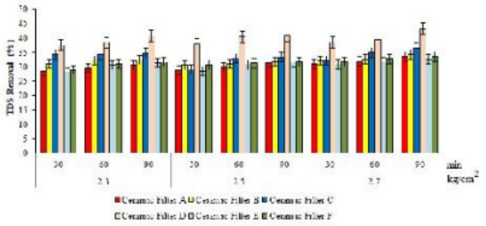


Fig. 2. Removal of TDS using Ceramic Filter.

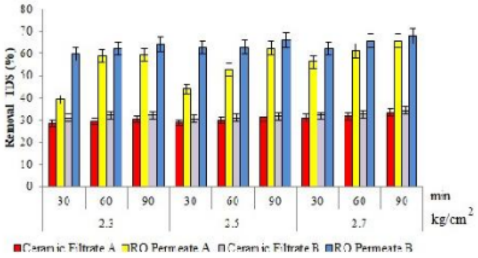


Fig. 3. TDS removal of integrated ceramic A-RO and ceramic filter B-RO.

2.3. Methods

The PW samples were analyzed for TDS, acidity, barium, and phenol concentration. The sample is pumped into a ceramic filter. The filtrate from the ceramic filter is placed in the filtrate tank with a capacity of 250 L, which also feeds to the RO membrane. From the RO feed tank, the PW is transferred using a high-pressure pump to the RO. The permeate and concentrate RO were placed in permeate and concentrate tanks. The filtrate and permeate were collected into bottle samples for TDS, acidity, barium, and phenol analysis.

The laboratory analyzes for TDS used a total dissolved solid meter (Thermo Scientific, EUTECH TN-100). Barium ions and phenol concentration were measured using APHA 3120:2017 and Amino Antipyrine methods. The TDS, barium, and phenol removal can be calculated using Eq. (1).

$$\text{Removal percentage} = \frac{(c_1 - c_2)}{c_1} \times 100\% \qquad (1)$$

where C_1 is the initial concentration (mg/L) and C_2 is the final concentration (mg/L).

The SEM-EDX type Vega 3 was employed to determine the structure and morphology of ceramic filter surface and chemical analysis of the elements. The PW treatment using ceramic filter and RO can be seen in Fig. 1.

Table 2. The acidity of filtrate of various Ceramic Filter.

Pressure (kg/cm^2)	Time (min.)	Ceramic filter					
		A	B	C	D	E	F
		pH					
2.3	30	8.50	8.29	8.28	8.12	8.46	8.47
	60	8.50	8.44	8.25	8.06	8.45	8.53
	90	8.55	8.41	8.25	7.98	8.40	8.54
2.5	30	8.41	8.49	8.43	8.28	8.52	8.45
	60	8.52	8.53	8.42	8.19	8.46	8.45
	90	8.51	8.45	8.33	8.17	8.52	8.45
2.7	30	8.39	8.30	8.49	8.23	8.51	8.52
	60	8.43	8.38	8.44	8.25	8.52	8.53
	90	8.44	8.54	8.41	8.18	8.51	8.54

3. Results and discussion

3.1. TDS removal

The process in the ceramic filter is a pretreatment stage before the PW enters the RO membrane. The TDS concentration of PW in this study was still too high, namely 12,500 mg/L. The TDS removal of PW by ceramic filter with various compositions of RCC spent catalyst, natural clay, and gadung starch can be seen in Fig. 2.

Fig. 2 shows that the composition of the ceramic filter plays an important role in decreasing the TDS of PW. The TDS removal by ceramic filter B was higher than those of ceramic filter A. The spent catalyst which is activated by the addition of 1 M hydrochloric acid (HCl) could modify the properties of the spent catalyst and improve its adsorbent capacity. In addition, hydrochloric acid can dissolve impurities that cover the pores. Therefore, the effective surface area of the ceramic filter will increase.

Ceramic filters with dominantly natural clay components are better than those with a high RCC spent catalyst composition. It is shown that ceramic filter D could lower TDS than other filters. It is due to the clay being fired at 620°C and organic matter in the clay acting as a chelating agent. Silicon dioxide (SiO_2) and Aluminium oxide (Al_2O_3) as adsorbents for most pollutants and their presence in natural clay as the main components can increase the adsorption rate [13]. Removal of dissolved solids also resulted in the consequent reduction of organic and inorganic materials, such as metals, minerals, salts, and ions [14]. Ceramic filter D, the content of the natural clay, which is composed mainly of silica and alumina, and RCC spent catalyst as a carbon activated for treated water. Natural clay with a pore volume was 0.1374 cm^3/g consists of negatively charged aluminosilicate layers kept together by cations, and they have the ability to adsorb water between the layers [15–17]. Silica particles are vital metal oxides that have the ability to remove heavy metal ions from aqueous solutions due to their large surface area, excellent adsorption capacity, and could increase the strength of the filter [18, 19].The difference in operating pressure and operating time affects the performance of ceramic membranes. The greater the pressure difference and the longer the operating time, the higher the decrease in TDS, so the better the resulting filtrate. Pressure increases cause a driving force for the dissolved substances in the water to diffuse through the membrane. The removal of TDS PW becomes higher because the dissolved substances are retained on the membrane surface.

Although the average TDS value of the filtrate by the ceramic filter is 6,570 mg/L, it is still above the regulation. TDS removal reached an average of 33.45%. The result considers that the ceramic filter's filtration can be used as a pretreatment for PW.

Furthermore, the filtrate from the ceramic filter will be fed to the RO membrane. From Fig. 3, it can be seen that the increasing percentage of RO permeate after processing on ceramic filter A and ceramic filter B was 65.84% and 67.93%, respectively. The higher the decrease in TDS obtained, the better RO permeate quality. At an applied pressure of 2.7 kg/cm^2 and an operating time of 90 min, the TDS of RO permeate was obtained in ranges of 1,650 - 2,290 mg/L. It shows that the TDS permeate value (maksimum value of TDS 4,000 mg/L) obtained has met the South Sumatra Governor Regulation No. 8/ 2012 concerning Quality Standards for Liquid Waste for Industrial, Hotel, Hospital, Domestic and Coal Mining Activities.

3.2. Acidity

Measurement of acidity shows the nature of the solution, namely acid or base. The acidity parameter can also determine the characteristics of a liquid. In the analysis of the initial sample of PW, the initial acidity value was 7.80. The acidity value of the filtrate from ceramic filters with various compositions of RCC spent catalyst, natural clay, and gadung starch can be seen in Table 2.

Table 3. The acidity of RO permeate at the different compositions of ceramic filter.

Pressure (kg/cm^2)	Time (min.)	Ceramic Filter Variation					
		A	B	C	D	E	F
		pH					
2.3	30	8.50	8.32	8.22	8.04	8.38	8.52
	60	8.41	8.27	8.15	7.88	8.36	8.51
	90	8.51	8.38	8.17	7.83	8.38	8.51
2.5	30	8.45	8.50	8.41	8.13	8.51	8.52
	60	8.45	8.51	8.41	7.93	8.43	8.45
	90	8.40	8.46	8.38	7.90	8.35	8.50
2.7	30	8.34	8.33	7.94	8.28	8.44	8.53
	60	8.41	8.36	8.33	8.12	8.35	8.53
	90	8.39	8.53	8.35	8.29	8.49	8.51

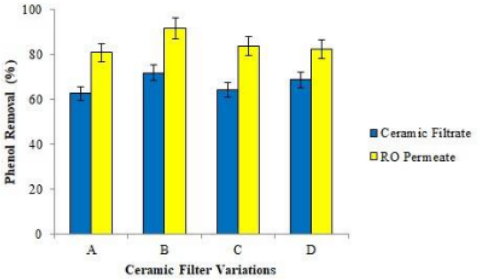


Fig. 4. Phenol removal by ceramic filter and RO.

Table 2 shows the acidity of ceramic filter filtrate at 30-90 min intervals at applied pressures of 2.3, 2.5, and 2.7 kg/cm2, respectively, ranging from 7.98 – 8.55. The acidity value of the PW treatment process using a ceramic filter is still in the range of regulation, which is 6 – 9. However, there is an increase from the initial acidity of 7.80. It happens because of the difference in the ability of alumina and silica to react with bases. Aluminum oxide is amphoteric because it can react with strong acids and bases. At the same time, silica is a non-metallic oxide compound that is covalently bonded and can react with strong bases. Other causes, phenol is a weak acid [20], so when phenol is adsorbed on the ceramic filter, the acidity of the PW will increase [21]. The acidity value is close to neutral, namely 7.98 at a pressure of 2.3 kg/cm^2 and operation time 90 min using a ceramic filter D. Overall, the effect of applied pressure and operating time of each variation of the ceramic filter does not significantly affect the acidity value of the filtrate. The acidity of the RO permeate at the different compositions of the ceramic filter can be seen in Table 3.

Table 3 shows that the range of acidity values of the RO permeate obtained ranged from 7.83 to 8.53. After the PW passes through the RO membrane, the acidity value decreases. Overall, the PW treatment with various ceramic filters integrated with a reverse osmosis (RO) system obtained values that meet environmental quality standards regulation.

Table 4. The acidity of RO permeate at the different compositions of ceramic filter.

Ceramic Filter	Filtrate of Ceramic Filter (mg/L)	Permeate of RO (mg/L)
A	3.695	1.901
B	2.782	0.813
C	3.529	1.616
D	3.093	1.739

3.3. Phenol removal

Based on the regulation, the maximum permitted value of phenol in PW is 2 mg/L. The initial phenol content in the PW still exceeds the quality standard of 9.88 mg/L.

Fig. 4 shows the effect of ceramic filter variations on the percentage reduction in phenol content. The highest percentage decrease of phenol was obtained using ceramic filter B was 71.84%. Percentage reduction of phenol by RO permeate ranges from 80.76 to 83.64%. The manufacture of ceramic filters with gadung starch can produce a porous ceramic filter when the ceramic filter is burned in the furnace. The gadung starch will oxidize, leave the ceramic filter, and make the pores. The more pores in the ceramic filter will increase the surface area and adsorption capacity. Overall, PW treatment with various ceramic filters integrated with the RO system obtained phenol levels that meet environmental quality standards regulated by South Sumatra Governor Regulation No. 8 / 2012.

Table 4 shows the phenol concentration in various ceramic filters integrated with the RO system. The phenol concentration meets environmental quality standards regulated by South Sumatra Governor Regulation No. 8/2012. Percentage reduction in phenol permeate levels by membranes RO of 80.76 – 83.64%.

Table 5. Barium concentration in ceramic filter filtrate and RO permeates.

Ceramic Filter	Filtrate of Ceramic Filter (mg/L)	Permeate of RO (mg/L)
A	3.994	1.590
B	3.128	0.866
C	3.686	0.203
D	3.741	0.338

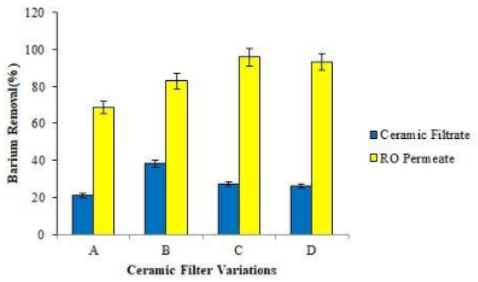


Fig. 5. Removal of barium in the filtrate of Ceramic Filter and RO Permeate (An applied pressure of 2.7 kg/cm^2 and contact time of 90 min).

3.4. Barium removal

Based on the Minister of Environment Regulation No. 5/2014 concerning wastewater quality standards. Barium concentration in PW exceeds the limit, which is 5.070 mg/L.

Table 5 shows that the concentration of barium metal ions in the ceramic filter filtrate is still above the quality standard, which is in the range of 3.994–3.128. The removal of barium metal ions of PW by the ceramic filter is ranging from 21.22 to 38.30%, as shown in Fig. 5. The highest percentage decrease of barium as 38.30% was obtained using ceramic filter B. Silica and alumina in RCC spent catalysts as aluminosilicate minerals can adsorb metal ions. Silica and alumina have a polar side with an active hydroxyl group (-OH) when in an aqueous solution. Overall the treatment of PW using a ceramic filter integrated with RO membrane produces the permeate with a concentration of barium ions that meet the quality standards of Minister of Environment Regulation No. 5 / 2014.

3.5. SEM-EDX of Ceramic Filter

Scanning electron microscopy (SEM) is a helpful method for showing various properties related to surface topography, analyzing surface features, shape, and particle size of samples [15]. While energy-dispersive X-ray spectroscopy (EDX) is an elemental analysis technique associated with electron microscopy based on the generation of characteristic X-rays that reveal the presence of elements present in the specimen [22].

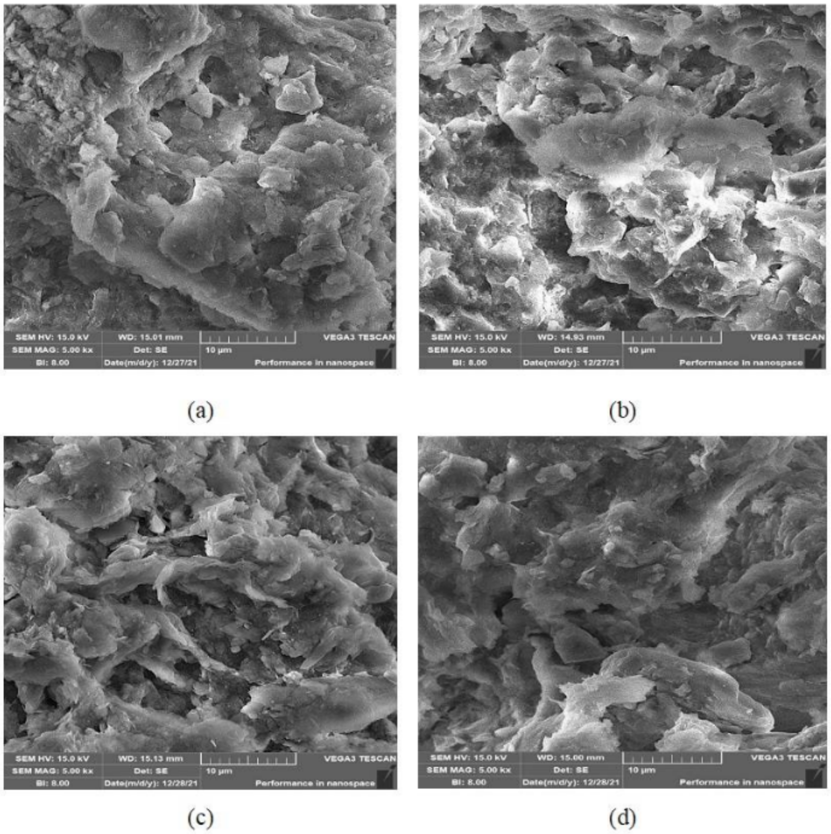
The combined use of electron microscopy with energy-dispersive X-ray spectroscopy (SEM-EDX) provides detailed information on the elemental composition of ceramic filters with additional details on the inorganic additives. The ceramic filters surface analyzed using SEM were ceramic filters C and D. Fig. 6 shows that two types of ceramic filters show the rough surface, non-homogenous with random pore size. Ceramic filters C and D are prepared with activated spent catalysts. However, the composition of those filters is dominated by clay. The uses of hydrochloric acid as an activated agent only for the RCC spent catalyst. Therefore, it is suggested that ceramic filters' impurities originate from clay. The ceramic filter pores size ranges from 1 to 10 μm, and the ceramic filters can be categorized as microfiltration filters [17].

Figs. 7 and 8 show the EDX spectrum of ceramic filters C and D before and after use for PW filtration.

Figs. 7 and 8 show the presence of ions on the surface of ceramic filters C and D. Barium ions are found in ceramic filters C and D before and after being used in the filtration. Chemical analysis by EDX revealed higher amounts of barium in ceramic filters C and D after use as filters. It can be seen in Fig. 7 that the increase of barium by ceramic filter C from 0.36% to 0.66%. In Fig. 7 (b), there is N (2.54% of mass) as a inorganic element were observed on the membrane surface. Its source was most probably from PW containing ammonia (NH3N) of 3.980 mg/L. Fig. 8 shows an increase in % mass of barium ions from 0.45% to 2.97% by ceramic filter D. It indicates that the ceramic filter has an adsorption capacity to adsorb barium from the PW.

3.6. Flux

The flux of a membrane is an important measure for its permeation capability. In Fig. 9, At 30 min the flux values are 4.01 L/m^{2}h, 4.81 L/m^{2}h and 5.61L/m2h for pressure 2.3 kg/cm^2, 2.5 kg/cm^2 and 2.7 kg/cm^2, respectively. The membrane flux value is highest at the lower time, where pressure has little role, namely the higher the pressure, the higher the flux value. Increasing the contact time will increase the fouling risk of ceramic filters caused by high time [17]. The membrane flux decreases because of the deposition particles on the membrane surfaces, the membrane pores become denser resulting in the decrease of the membrane pore size [23, 24]. As shown at Fig. 9 , increases in the contact after 90 min of contact time.



(a) (b) (c) (d)

Fig. 6. SEM image of the ceramic filter at a magnification of 5000 X (Ceramic filter C: (a) before (b) after, and ceramic filter D: (c) before (d) after).

4. Conclusion

The following conclusion can be drawn from the current work:

1. The RCC spent catalytic cracking can be applied as a material for the ceramic filter for produced water treatment. Ceramic filters with various material compositions can be used to reduce the TDS, phenol, and barium concentration in produced water.

2. The optimum removal of TDS, phenol, and barium are 43.21%, 68.69%, and 26.21% respectively was obtained using ceramic filter D with the removal in RO permeate are 86.68%, 82.40%, and 93.33% respectively. Ceramic B was reduced TDS, phenol, and barium in the filtrate 34.11%, 71.84%, and 38.30%, respectively and RO permeates 67.93, 91.77%, and 82.92%, respectively.

3. The removal efficiency of TDS, phenol and barium in decreasing order is Filter D > Filter C > Filter A > Filter B, for phenol is Filter B > Filter D > Filter C > Filter A, and for barium is Filter B > Filter C > Filter D > Filter A.

4. The permeate produced from the ceramic filter integrated with the RO membrane system are met the wastewater quality standards regulation.

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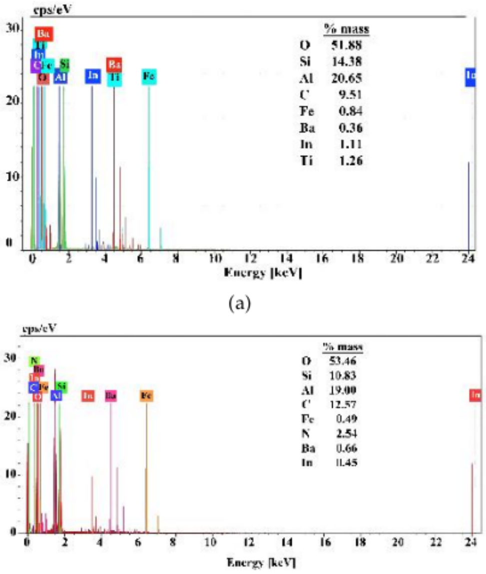
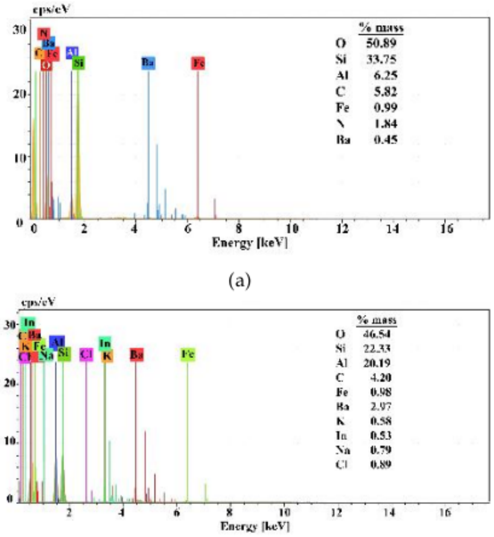


(a)

(b)

Fig. 7. EDX spectrum of ceramic filter C (a) before (b) after used.



(a)

(b)

Fig. 8. EDX Spectrum of Ceramic Filter D (a) before used (b) after used.

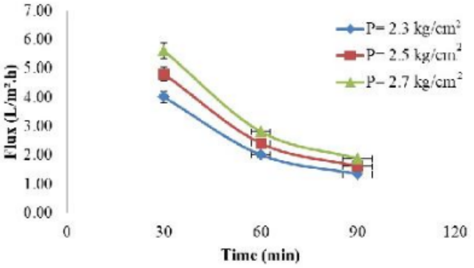


Fig. 9. Flux of ceramic filter D.

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