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RESEARCH PAPER

Application of Ceramic Filter and Reverse Osmosis Membrane for Produced Water Treatment

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Abstract
The effect of ceramic filter composition on improving the quality of produced water by reducing total dissolved solids (TDS), barium, and phenol for reverse osmosis (RO) treatment was investigated in the present work. The ceramic filters were fabricated using a residue catalytic cracking (RCC) unit spent catalyst with and without activation, clay, and Dioscorea hispida starch (DHS), at various compositions. The result showed that the optimum removal of TDS, barium, and phenol in produced water was achieved at a flow rate of sample 7 L/min and an operating time of 90 min. Ceramic filter with the composition of 60% spent catalyst without activation: 37.5% clay: 2.5% DHS reduced 34.84% TDS, 27.97% barium, and 71.11% phenol. While, the ceramic filter with a composition of 37.5% activated spent catalyst: 60% clay: 2.5% DHS was removed 51.44% TDS, 27.93% barium, and 85.29% phenol from produced water. The next steps of treatment of filtrates of the ceramic filter using reverse osmosis (RO) membrane showed that the permeate met the Indonesian standard for oil and gas wastewater. In addition, adsorption of TDS, barium, and phenol from produced water was dominated by clay composition in the ceramic filter.

Keywords: Adsorption, Clay, Dioscorea hispida, Oil reservoirs, Spent catalyst

INTRODUCTION

Most of the oil and gas reservoirs in South Sumatra are old wells that require more water injection into the well to extract oil. The oil and gas industry produces a large amount of water which is commonly known as produced water. Produced water is the mixture of formation water and saltwater which has a negative environmental impact due to its complex composition (Al-Ghouti et al., 2019; Al-Kaabi et al. 2019; Bezerra et al. 2019; Dickhout et al. 2017; Fakhru'l-Razi et al. 2009; Hendges et al. 2021; Lin et al. 2020; Wang et al. 2009; Yang et al. 2018; Ye et al. 2020). Produced water is characterized by parameters such as total dissolved solids (TDS), oil and grease, phenol, heavy metals, and hydrocarbons which adds to its complexity (Dudek et al. 2020). On average, produced water in oil and gas reservoirs in South Sumatra has TDS, barium, and phenol concentrations above the standard which is stipulated by the South Sumatra Governor regulation No. 8/2012 and the Ministry of Environment and Forestry of Republic Indonesia Regulation No. 5/ 2014.

TDS concentration of produced water in the oil and gas wells worldwide is ranging from 100 mg/L to 400,000 mg/L. Increased TDS due to the increased concentrations of both sodium and bicarbonate in produced water. However, the TDS concentration depends on the location

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of the well field, geological variations between basins, and the resource of the produced water (Al-Ghouti et al., 2019).

Barium is one of the higher concentration elements (0-850 mg/L) in the produced water with characteristics of high solubility in water, excellent mobility in the environment, and potentially toxic. Due to their characteristic, it needs to develop an effective process for removing barium from produced water. The sorption technique is widely used to remove barium and strontium ions from the oilfield-produced water (Reddy et al., 2021).

Phenolic compounds are present in the effluents of various industries such as oil refining, petrochemicals, pharmaceuticals, etc. The toxicity levels of phenolic compounds usually are 9–25 mg/L for both humans and aquatic life. Discharge of these compounds without treatment may lead to serious health risks to humans, animals, and marine systems. Several methods have been available to remove phenols from wastewaters, including adsorption, membrane process, and enzymatic treatment (Villegas et al., 2016; Dehmani et al., 2020).

Ceramics filters have several advantages in water filtration due to their thermal and chemical stability, durability, and high separation efficiency (Chaukura et al. 2020; Kamoun et al. 2020; Mubiayi et al. 2021). Clay is a common substance for ceramic filters manufactured, which is fine-grained soil particles of kaolinite, aluminum oxide (Al_2O_3), and silicon dioxide (SiO_2). The use of clays as adsorbent have advantages upon many other commercially available adsorbents in terms of low-cost, an abundant availability, high specific surface area, excellent adsorption properties, non-toxic nature, and large potential for ion exchange. A detailed, informative and comprehensive review of clay minerals and the chemical composition of various clay minerals as successful adsorbent has been carried out by Uddin (2017).

The residue catalytic cracking (RCC) catalysts is silica and alumina-based catalyst used in the refining processes in the oil and gas industry. The amount of RCC spent catalysts in one oil industry in South Sumatra is about 15,980 tons, and among them, 10.30 tons are only stored in the hazardous and toxic waste warehouse (Pertamina, 2017). This type of catalyst will deactivate after its life cycle (3-4 years) because of structural changes, poisoning, or deposition of extraneous materials like coke and metals (Chiranjeevi et al., 2016). Therefore, reactivation of catalysts is needed to recover the surface areas and pores (Lu et al., 2020).

Reverse osmosis (RO) is the purification technology that uses a semipermeable membrane to remove ions, molecules, and larger particles from water based on the osmotic pressure. Nowadays, RO plays an important role in water due to its wide applications in desalination and wastewater treatment (Hailemariam et al., 2020 ; Johnston et al., 2022).

The present work highlighted the application of a new ceramic filter from mixture of the RCC unit spent catalyst, natural clay, and *discorea hispida* starch (DHS) in removing the contaminant parameter of produced water. The effectiveness of ceramic filters in reducing total dissolved solids, phenol, and barium in produced water was investigated before being fed into the RO membrane.

MATERIALS AND METHODS

The produced water was supplied by the oil and gas industry in South Sumatra, Indonesia, and placed in the polyethylene tank with a capacity of 1000 L. Ceramic filter was fabricated using a mixture of natural clay, the RCC spent catalyst with or without activation, and DHS. A ceramic filter was designed as a porous cylindrical tube shape with an outer diameter of 6 cm, an inner diameter of 3 cm, and a length of 25 cm (Nasir & Faizal, 2016). Clay and RCC spent catalyst dried naturally under sunlight for 12 h, then in the oven at 115 ^{o}C for 24 hours and grinding into 100 mesh of particle size. The RCC catalyst was activated using 1M HCl, soaked overnight, rinsed with demineralized water, and neutralized by NaOH 0.1 M. The RCC spent catalyst and clay were homogenized using 2.5% of DHS and clean water, molded, dried at room temperature for 24 h, and sintered at 900 °C for 24 h in the furnace. The average weight of ceramic filters ranged from 400 to 600 g.

Produced water was transferred into ceramic filters using a centrifugal pump. Before passing through the ceramic filter, the produced water has flowed first into polypropylene filters with a pore size of 0.5 μm, 0.3 μm, and 0.1 μm, respectively. The process variables studied were feed flow rate, operating time, and filter composition. The filtrate of the ceramic filter was taken for analysis after 30 min of operation time and pumped into the RO membrane. Both filtrates from the ceramic filter and the RO permeate were analyzed for TDS, barium, and phenol concentrations.

The ceramic filter composition was specified as follow:

- Filter A: 60% spent catalyst without activation: 37.5% clay: 2.5% DHS
- Filter B: 60% spent catalyst with activation: 37.5% clay: 2.5% DHS
- Filter C: 37.5 % spent catalyst without activation: 60% clay: 2.5% DHS
- Filter D: 37.5% spent catalyst with activation: 60% clay: 2.5% DHS

Total dissolved solids were measured using Horiba Laqua PC220-K) TDS meter. Barium analysis was conducted using APHA 3120:2017 method. While phenol was analyzed based on the Amino Antipyrine method using a spectrophotometer. The ceramic filter's morphology, microstructure, and chemical composition were investigated using a scanning electron microscope-Energy Dispersive X-Ray (SEM-EDX) Tescan Vega 3 SB instrument. The Quantachrome type Novatouch LX-4 Surface Area Analyzer was used to characterized the ceramic filter pores.

The removal of TDS, barium, and phenol can be calculated using equation (1):

$$\% \text{ removal} = \frac{C1 - C2}{C1} \text{ x } 100\% \quad (1)$$

Where: % removal is the removal percentage of TDS, barium, and phenol, C_1 is the initial concentration (mg/L) and C_2 is the final concentration (mg/L).

The experimental set-up of integrated ceramic filter-RO membrane for produced water treatment is shown in Fig. 1.

RESULTS AND DISCUSSION

Table 1 shows several parameters for wastewater standard of oil and gas exploration and

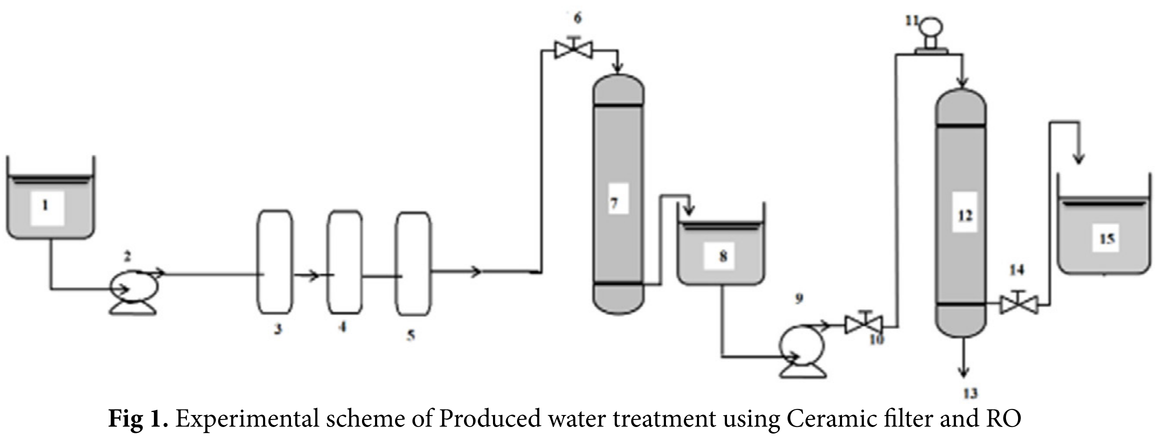

Fig 1. Experimental scheme of Produced water treatment using Ceramic filter and RO

(1. Feed tank, 2. Centrifugal pump, 3.4.5. Propylene filter (0.5, 0.3 and 0.1 μm), 6.10.14 Valve, 7. Ceramic filter, 8. Filtrate tank 9. RO membrane pump, 11. Flowmeter 12. RO membrane 13. Concentrate stream 15. RO permeate tank)

Table 1. Characterization of Produced Water

No.	Parameter	Values	Standards	Unit
1	Temperature	21.90	40	°C
2	COD	28	200	mg/L
3	Oil and grease	0.40	25	mg/L
4	Phenol	9.88	2	mg/L
5	TDS	12,500	4,000	mg/L
6	Acidity	7.80	6 to 9	-
5	Total ammonia	3.98	5	mg/L
6	Sulfide	0.24	0.5	mg/L
7	Barium*)	5.07	2	mg/L

*) The Indonesian Ministry of Environment and Forestry Regulation No 5/2014

Table 2. BET characterization of Ceramic Filter

BET characterization	Values
Specific surface area (m^2/g)	14.03
Pore size (nm)	2.75
Pore volume (cm^3/g)	0.04

production activities by South Sumatra Governor Regulation No. 8/2012 and the Indonesian Ministry of Environment Regulation No. 5/ 2014.

Table 1 summarizes some parameters of produced water such as TDS, barium, and phenol, which are exceeding the limits stipulated by the Ministry of Environment and Forestry, Republic of Indonesia regulation.

The Brunauer Emmett Teller (BET) analysis shows the surface area, pore size, and pore volume of ceramic filter. The BET of ceramic filter filter D with the composition of 37.5% spent catalyst with the activation: 60% clay: 2.5% DHS is illustrated in Table 2.

Based on the BET analysis, the surface area of ceramic filter D is 14.03 m^2/g and the pore size is 2.75 nm. Therefore, the pore size of ceramic filters D is classified as mesoporous (between 2 – and 6 nm) (Ngoc Dung et al., 2019). The relatively larger surface area is associated with the adsorption capacity of the adsorbents. The increase in surface area is due to the opening of the pores of ceramic filter constituent materials, which were initially covered by impurities. The release of impurities of the spent catalyst occurs during the activation and sintering process of the ceramic filter.

Adsorption is a surface phenomenon that occurs as a result of the interaction forces between the surface of the adsorbent, the solvent, and the dissolved various chemical species in the produced water to be treated. Adsorption can be classified as chemisorption and physical adsorption, depending on the nature of surface forces. However, a suitable adsorbent must have a good affinity to target compounds and must ensure maximum removal of the compounds at a low cost (Amakiria et al., 2022). In physical adsorption, the process was controlled by weak intermolecular forces between adsorbate and adsorbent. In chemisorptions, there is significant electron transfer due to chemical bond between adsorbate and the adsorbent surface (Annan et al., 2018). The contaminants in produced water such as TDS and metal ions including barium are removed by adsorbing onto the solid surfaces of ceramic filter using the ability of solids to selectively concentrate specific substances from produced water. Therefore, this mechanism is

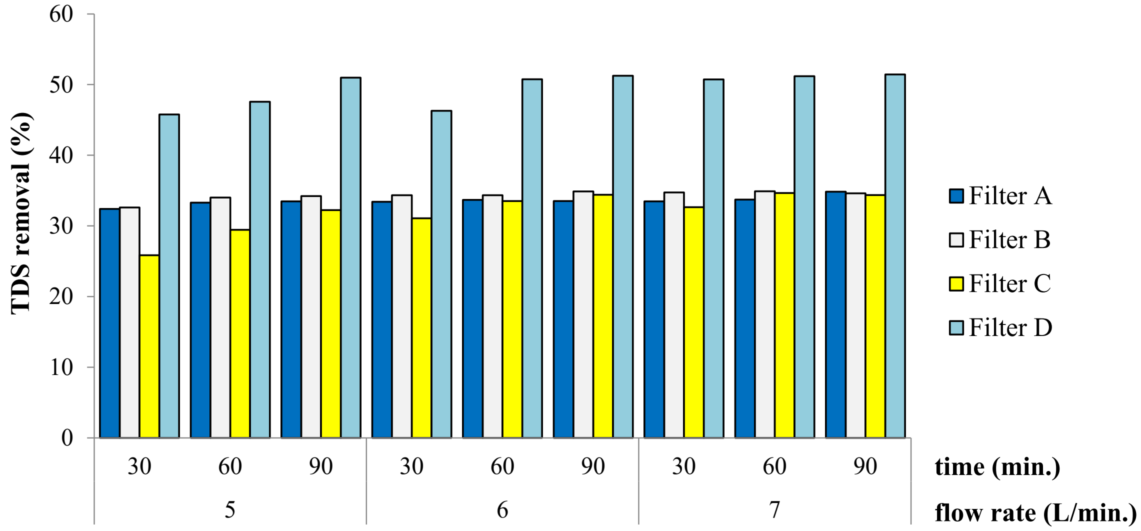

Fig 2. TDS removal by ceramic filters

affected by the pore distribution and the ceramic filter characteristics such as surface area and pore size. In addition, the adsorption of phenol onto ceramic filter surface is provided by the hydrogen bonding of the water molecules (Quallal, 2019). According to (Dehmani et al., 2020) clay is one of low-cost and effective adsorbent for phenol removal from wastewater.

Total dissolved solids (TDS) describe the content of inorganic salts and small amounts of organic compounds present in water and wastewater. High concentrations of TDS in produced water can cause scale in wells or contamination of surrounding water and soil (Jang et al., 2017).

Fig 2 shows a significant decrease in TDS for each variation of ceramic filter. The TDS removal by filters A and D at a flow rate of 7 L/min and operation time of 90 min are 34.84% and 51.44%, respectively. While, for filters B and C at a flow rate of 7 L/min and operation time of 60 min are 34.89% and 34.65%, respectively. Among these filters, ceramic filter D shows the higher removal of TDS. However, the TDS removal for all-ceramic filter reached an average of 37.35%. Filter D is clay-based ceramic filter where its composition was dominated by clay. In the adsorption, clay is a good adsorbent for TDS and heavy metal ions. The results considered that the TDS removal effectively using the clay-based ceramic filter. Increase of clay and spent catalyst ratio in the ceramic filter will increase the TDS removal. This is due to the activation of spent catalysts and sintering process will open the pores and releasing the impurities covering the spent catalyst surface. This result also considers that ceramic filters from clay and spent catalysts can be used as a pretreatment of produced water before fed into RO membrane. RO membrane technology can be applied to the treatment of produced water to reduce the concentration of high TDS content (Jang et al., 2017). Produced water can reduce TDS volume by using RO membranes up to 80% (Gregory et al., 2011).

In the next stage, filtrates of ceramic filter are treated using RO membrane as a further process to obtain the optimum values for TDS, barium, and phenol removals. The results of the TDS removal obtained after passing through the ceramic filter and followed by RO can be seen in Fig. 3.

Fig 3 shows the removal of TDS by combining ceramic filter with an RO (herein stated as A-RO Filter). The TDS removal by the A-RO, B-RO, C-RO, and D-RO Filter is 63.17%, 71.08%, 71.35%, and 71.96%, respectively. The higher TDS removal after 90 min operation is shown by combine of ceramic filter and RO at flowrate of 7 L/min. Increase in contact time and flowrate will increase the TDS removal. However, there is no significant effect of ceramic filter composition on the TDS removal in RO permeates. The results shown, the TDS removal of

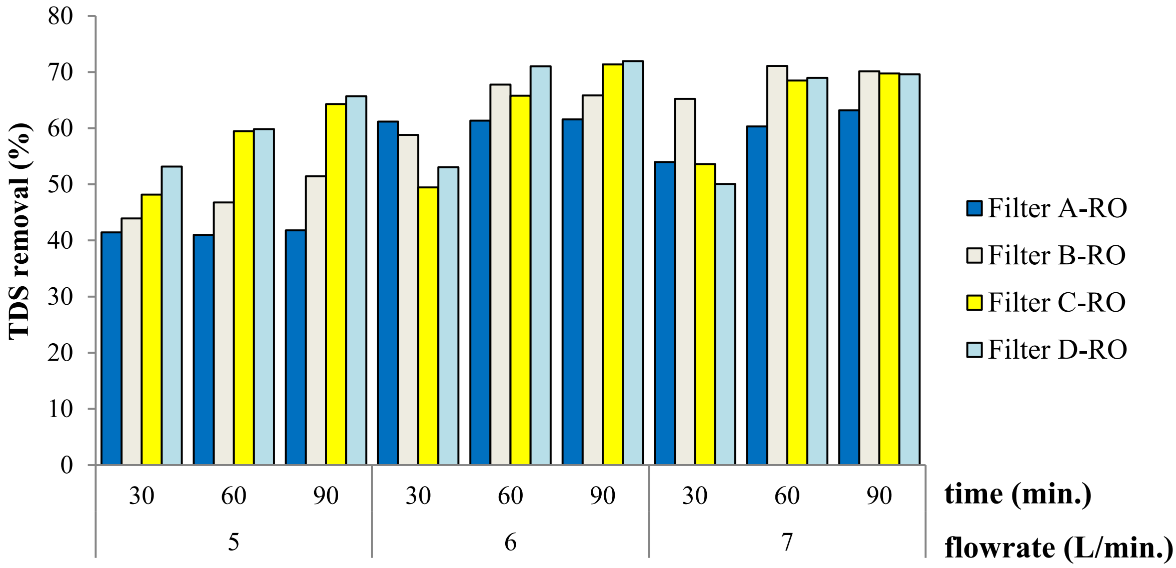

Fig 3. TDS removal by combined ceramic filters and RO

Table 3. Barium and phenol concentration after ceramic filter and combined ceramic filter-RO

No.	Concentration (mg/L)					
	Ceramic filter	Barium	Phenol	Ceramic filter-Reverse Osmosis	Barium	Phenol
1	A	3.65	2.85	A-RO	0.15	1.03
2	B	3.49	3.05	B-RO	0.17	0.80
3	C	3.74	3.01	C-RO	0.28	1.53
4	D	3.65	1.45	D-RO	0.23	1.03

combined ceramic filter and RO met the quality standard of regulation.

Barium is the most common and abundant metal ion found in produced water. Based on the regulation of the Ministry of Environment and Forestry of Republic Indonesia No. 5/2014, the maximum barium ions concentration in oil and gas wastewater is 2 mg/L. Table 3 shows the results of barium concentration from the ceramic filter and RO membrane. Barium concentration decreased from 5.07 to 3.49 mg/L in the filtrate of ceramic filter B, and to 0.17 mg/L in the RO permeate.

Fig 4 illustrates that ceramic filters can reduced barium concentration in produced water. For instance, ceramic filter B was reduced 31.15% of barium in produced water. Among the filters, ceramic filter B shown the highest barium removal. Nevertheless, all ceramic filters cannot reduce barium concentration to standard stipulated by the Ministry of Environment and Forestry of Republic Indonesia No. 5/2014 regulation. Ceramic filter B has the dominant composition of activated spent catalyst than clay. Activation of spent catalyst using HCl and NaOH removed impurities covering the surface of the spent catalyst thus increase ceramic filters surface area and active sites and adsorb metal ions including barium on the pores.

The results have also shown a significant decrease in barium concentration after RO treatment. It can be seen that ceramic filters A, B, C, and D combined with RO decreased the barium by more than 92%. All permeates RO indicated the barium values met the wastewater standard.

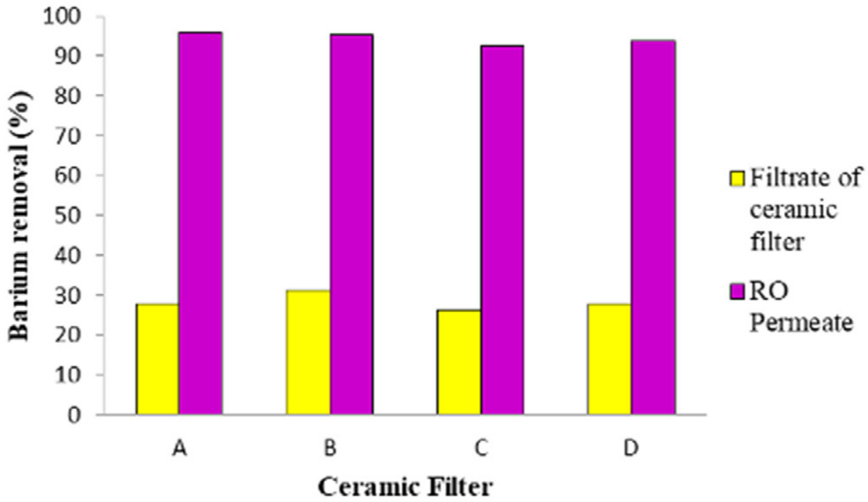

Fig 4. Barium removal by combined ceramic filter-RO

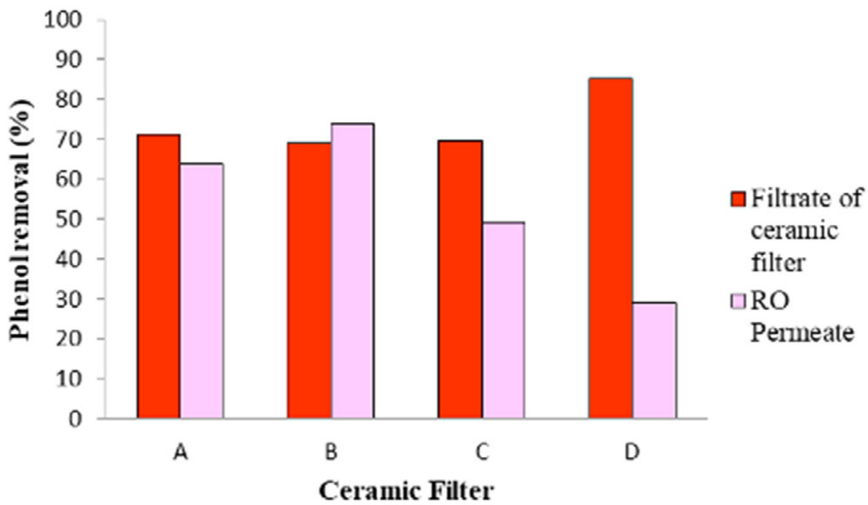

Fig 5. Phenol removal by combined ceramic filter-RO

Therefore, to reduce the fouling of RO by barium ions, a ceramic filter from RCC spent catalyst and clay is potential as a pretreatment before RO.

Phenol is an organic compound in the produced water sample that does not meet the wastewater quality standards. Table 3 also shows the phenol analysis from the filtrates of ceramic filter and RO membrane permeates. Among all-ceramic filters, filter D decreased the phenol concentration of produced water to 1.45 mg/L. This value is under the limit of standards regulation of the South Sumatra Governor No. 8/2012. The phenol removal by the combined ceramic filter and RO membrane can also be seen in Fig. 5.

Fig 5 shows that the ceramic filter can reduce phenol in the produced water. The average removal percentage of phenol by ceramic filter is between 69.14% and 85.29%. Phenol removal was similar to activated ceramic from crushed coal, bentonite and kaolin which is achieved 84.8%. Phenol can be removed more efficiently than metal ions because ceramic filter material has more affinity to organic than inorganic pollutants (Hamad, 2021). It is seen that phenol removal by RO membrane was varied for all ceramic filters. The concentration of phenol in RO permeate was about 28%. It is suggested that ceramic filters can be use as a pretreatment for

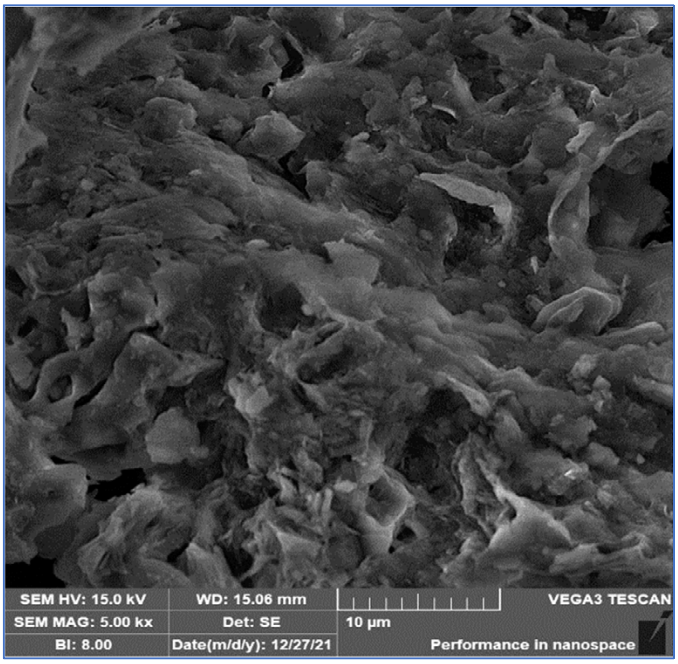

Fig 6A. SEM images of raw filter C at magnification 5000X

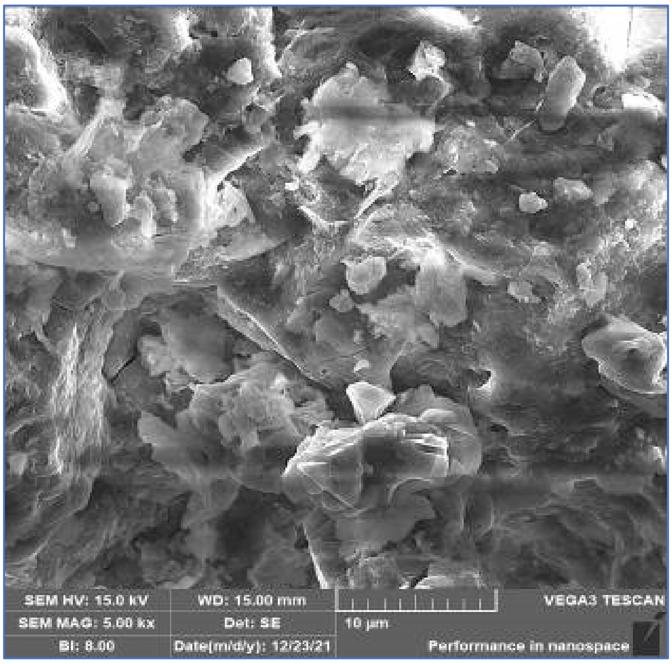

Fig 6B. SEM images of adsorbed filter C at magnification 5000X

reducing phenol and barium before feeding into RO membrane.

The SEM-EDX method is used to observe morphology and surface of ceramic filters before and after the produced water treatment process. Filters C and D were chosen for SEM-EDX characterization.

Fig 6A and 6B represent the SEM images of the raw and adsorbed ceramic filter C, whereas Fig 7A and 7B show the raw and adsorbed ceramic filter D with the magnification of 5000 X. Fig 6A and Fig 7A show the morphology of the ceramic filter structure where the non-activated spent

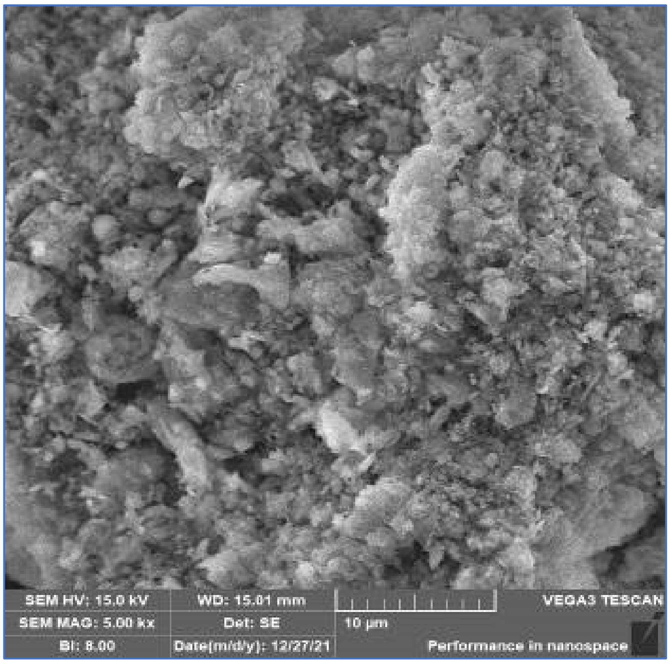

Fig 7A. SEM images of raw filter D at magnification 5000X

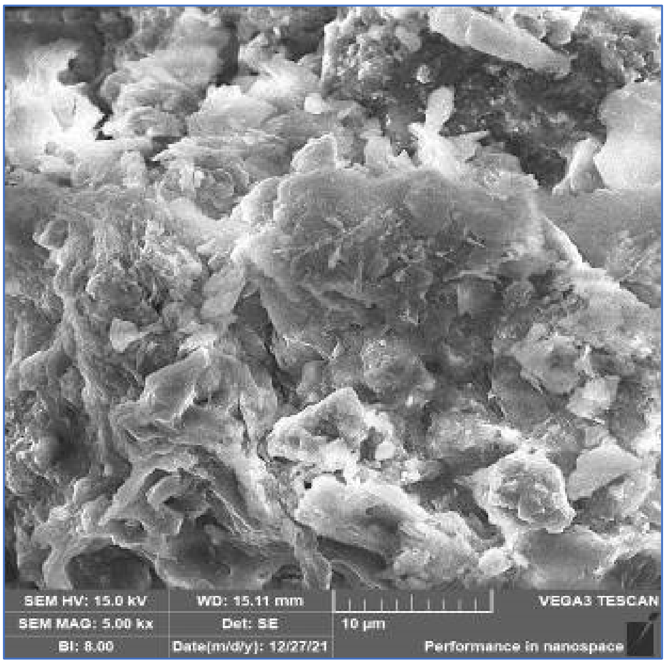

Fig 7B. SEM images of adsorbed filter D at magnification 5000X

catalyst is added into ceramic filter composition. The ceramic filter surface is rough and compact. A different result was founded in Fig 6B and Fig 7B, where the particles are evenly distributed and homogenous. The filter surface is indicating an adsorption of ions from produced water by the ceramic filter. The adsorption of ions on the pore and surface of filters is strengthened by the Energy Dispersive X-Ray (EDX) analysis as shown in Figures 8a, 8b, 9a, and 9b.

Based on the EDX results, barium concentration was significantly increased in the ceramic filter surface after the adsorption process. As shown in Figures 8A and 8B, barium in the ceramic

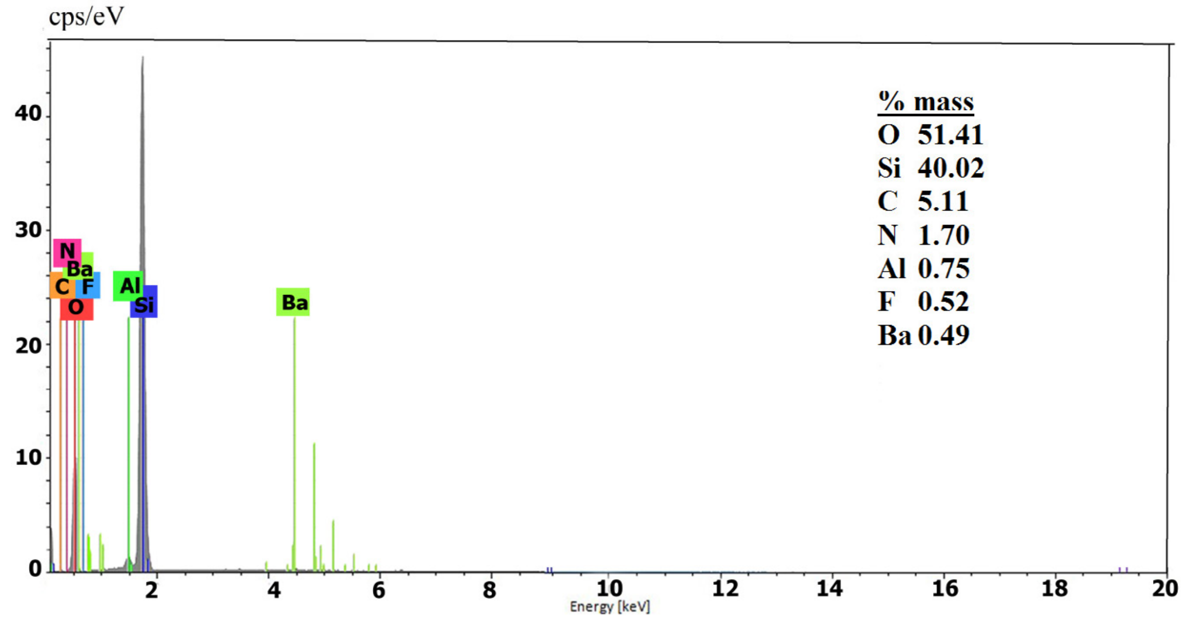

Fig 8A. Energy dispersive X-Ray (EDX) raw filter C

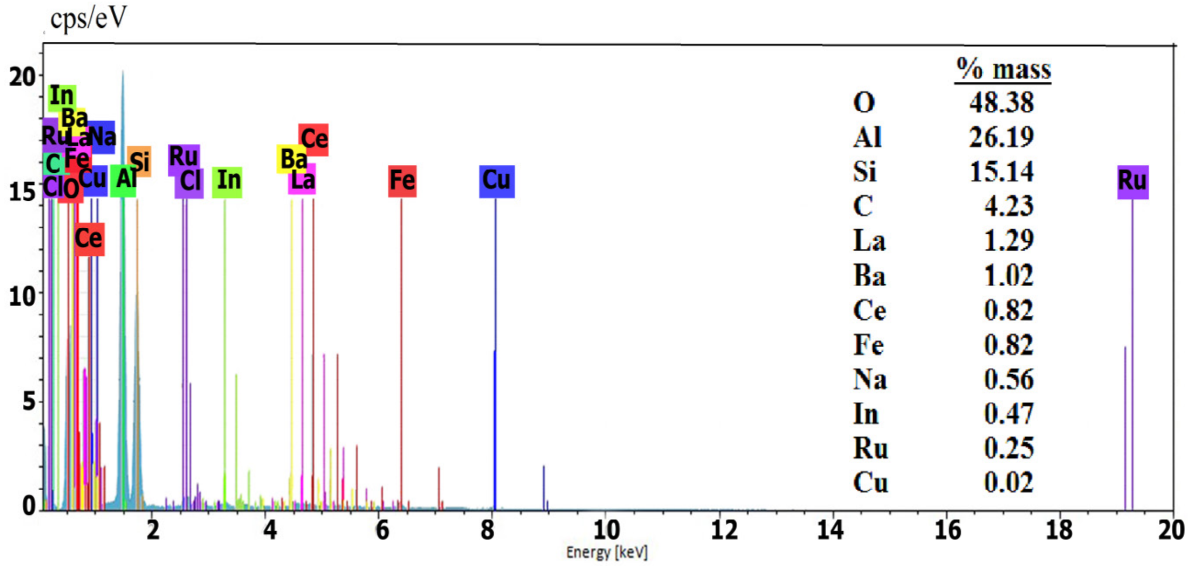

Fig 8B. Energy dispersive X-Ray (EDX) adsorbed filter C

filter C increased from 0.49% to 1.02%. A similar increase in barium from 0.34% to 0.88% using a ceramic filter D is shown in Figures 9A and 9B. It indicates that the ceramic filter effectively reduced barium ions. However, the results still exceed the standard, so it is necessary to carry out the following stage of treatment using an RO membrane. Barium absorbed by ceramic filter D appears to be slightly higher than those of filter C because the composition of filter D is dominated by an activated spent catalyst. The spent catalyst's activation can increase the surface area and enhance ion adsorption on the ceramic filter. In addition, SEM images show that all-ceramic filters have random pore structures with pore sizes ranging from 1 to 10 μm, and ceramic filter can be categorized as microfiltration filters (Nasir & Faizal, 2016).

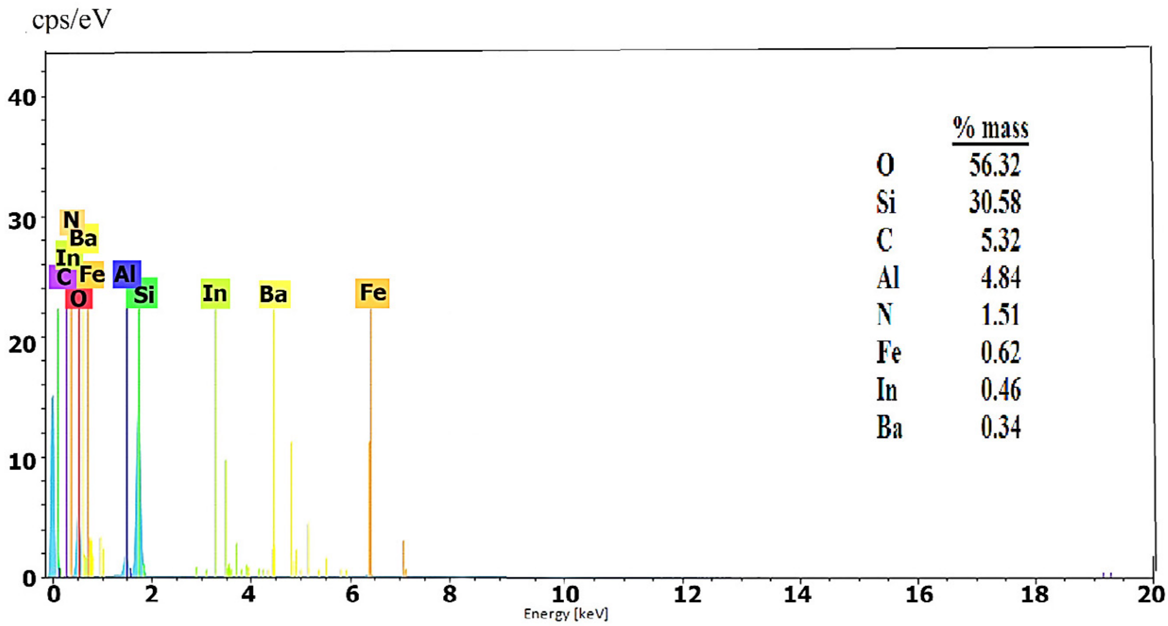

Fig 9A. Energy dispersive X-Ray (EDX) raw filter D

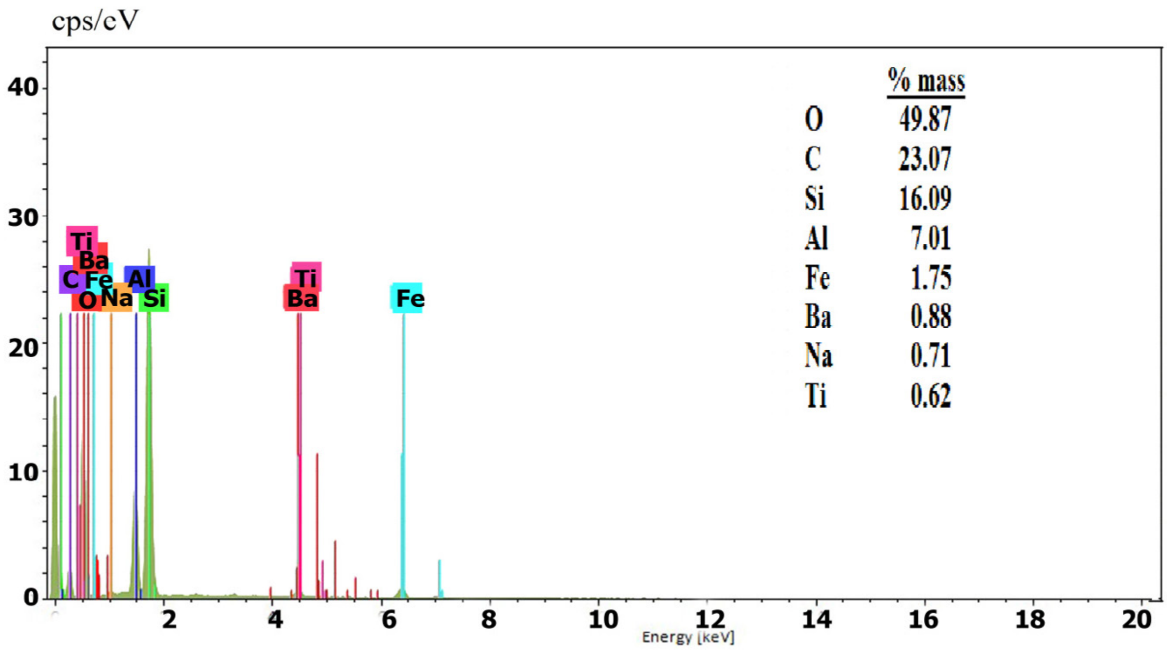

Figure 9B. Energy dispersive X-Ray (EDX) adsorbed filter D

CONCLUSION

Clay and activated spent catalyst composition in ceramic filter significantly affected the removal of TDS, barium and phenol from produced water. The TDS, barium, and phenol removal from produced water is in decreasing order as follows; TDS: filter D> filter A> filter B> filter C, barium: filter B> filter A> filter D > filter C, and phenol: filter D>filter A> filter C> filter B. It can conclude that the ceramic filter from the spent catalyst and clay followed by the RO membrane was suitable to reduce TDS, barium, and phenol concentration from produced water. Produced water that has been treated using a ceramic filter followed by an RO can be reinjected into oil and gas reservoirs or directly discharged into the environment.

GRANT SUPPORT DETAILS

The present research did not receive any financial support.

CONFLICT OF INTEREST

The authors declare that there is not any conflict of interests regarding the publication of this manuscript. In addition, the ethical issues, including plagiarism, informed consent, misconduct, data fabrication and/ or falsification, double publication and/ or submission, and redundancy has been completely observed by the authors.

LIFE SCIENCE REPORTING

No life science threat was practiced in this research.

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