

## **SYNTHESIS OF MEMBRANES FROM PILLARED CLAY $FeCl_3$ FOR SEAWATER DESALINATION APPLICATION**

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### **Abstract**

*Synthesis of membranes has been done using pillared clay  $FeCl_3$  for desalination application. Pillared clays  $FeCl_3$  were synthesized via a sol-gel method using  $[OH] / [Fe]$  0.25, 0.5, 1, 2, 2.5. Formed sol-gel was furnace on  $600^\circ C$  for 2 hours. The membranes which were cooled at room temperature for 3 days then tested filtration of seawater. The results showed that membrane pillared clays  $FeCl_3$  with a ratio  $[OH] / [Fe]$  0.25 reached filtration purity 99.99% with the initial TDS 27.58 ppm and resulted rejection TDS 00.22 ppm after filtration. Membrane with ratio  $[OH] / [Fe]$  0.5 reached filtration purity 80%, with initial TDS 27.58 ppm and resulted rejection TDS 11.76 ppm, while membranes with ratio  $[OH] / [Fe]$  1,2 and 2,5 was broken during the filtration proces, its resulted very small rejection.*

*Keywords: membrane, pillared clay,  $FeCl_3$ , Seawater desalination*

### **Introduction**

The membrane is a thin layer is semipermeabel that can hold and pass the movement of certain materials. Inorganic membranes have advantages, including high thermal stability, a higher separation efficiency, durable, and easy to clean and environmentally friendly [1]. Separation technology with membrane is very important, some of the benefits that can separate chemical species are species-specific, can operate at lower temperatures, saving energy, and does not cause negative impact on the environment [2]. Its role becomes very large, such as in desalination of water, ready to drink water supply, waste water treatment industry, biotechnology, pharmaceutical industry, and other separation technology [3]

$FeCl_3$  pillared clays are widely used as catalysts and adsorbents [4]. Pillared clay  $FeCl_3$  have interlayer spacing due to the inclusion pemilar agent that will cause changes in characteristics such as basal spacing, specific surface area, pore size distribution [5]. Based on pillared clays advantage  $FeCl_3$ , then in this research will be developed using  $FeCl_3$  pillared clays as seawater desalination membrane.

### **Experiment**

*Preparation of Pillared Clay.* As the host materials, natural clay samples from Banyusri Village, District Wonosegoro, Boyolali (Central Java). Natural clay shown as Figure 1. Stages of preparation done by washing using water DM, clays that have been washed, dried using an oven at  $100^\circ C$  for 2 hours to remove moisture and volatile impurities. The dried clay crushed and filtered using 170 mesh sieve to obtain the size of clay particles that were smaller and uniform.



Figure 1 Natural Clay of Boyolali

*Syntesis of Pillared Clays.*  $FeCl_3 \cdot 6H_2O$  0.02 M stirred input dropwise 0.02 M NaOH mixture was re-stirred for 24 hours at room temperature, then stirred for 24 hours at room temperature. Pillared agent  $FeCl_3$  added to the clay suspension and stirring at room temperature for 24 hours. Then the clay was intercalated with pillared

agent  $\text{FeCl}_3$  washed using distilled water until free Cl.



Figure 2 Seawater filtration process

Pillared clays  $\text{FeCl}_3$  was prepared by mixing the agent pemilar  $[\text{OH}] / [\text{Fe}]$  into an aqueous suspension with a variation lempung.menggunakan molar ratio between  $[\text{OH}]/[\text{Fe}]$  0.25; 0.5; m1; 2; 2.5. Pillared agents  $[\text{OH}]/[\text{Fe}]$  was prepared by mixing a solution of  $[\text{OH}]/[\text{Fe}]$  with NaOH solution. NaOH solution was added slowly to a solution of  $[\text{OH}]/[\text{Fe}]$ . The addition of NaOH done slowly. Pillared agent was stirred for 24 hours at room temperature and then allowed to stand and then added to the respective ratio of 30 grams was stirred for 24 hours. Habitation was done so that the particles interact and collide longer so that perfect reaction occurs.

*Syntesis of Membranes.* The homogeneous mixture then printed, with a compressive strength of 100 psi for 30 minutes. Stayed at room

temperature for 3 days, then calcined at  $600^\circ\text{C}$  for 2 hours and then test filtration [6], [7].

*Testing the Performance of the Membranes.* The sea water was put into a glass beaker, and then flowed to the membrane, after jetting process finished then product was analyzed. Seawater filtration process as shown in Figure 2.

## Results and Discussions

Pillared clays  $\text{FeCl}_3$  were already in print calcination at  $600^\circ\text{C}$  for 2 hours as shown in Figure 3 which shows the color change between the clays that have not been intercalated with clay that has been intercalated. The color change also occurs in clay that has been intercalated with rising calcination temperature. According to [8] in the calcination process, polycation metal incorporated into the clay interlayer will experience dehydration (removal of water molecules) and dehydroxylation polikation structure of metal clusters of metal oxide interlayer space permanently [8].

Based on these images, can be seen the change in color that occurs at the membrane. It was caused by the evaporation of the materials used. As explained by [9] in his journal that there are some important steps that occur during the calcination process. At the time the temperature reaches  $100^\circ\text{C}$ , the membrane will experience the evaporation of the water bound to the free, at the time the samples calcined at high temperature for 2 hours, and the hardened clay particles bind to generate strong porous membrane. Calcination also can cause shrinking pore size [9].



(a)



(b)

Figure 3 Pillared clays  $\text{FeCl}_3$  yet in calcination at  $600^\circ\text{C}$  (a), Pillared clay after calcination at  $600^\circ\text{C}$

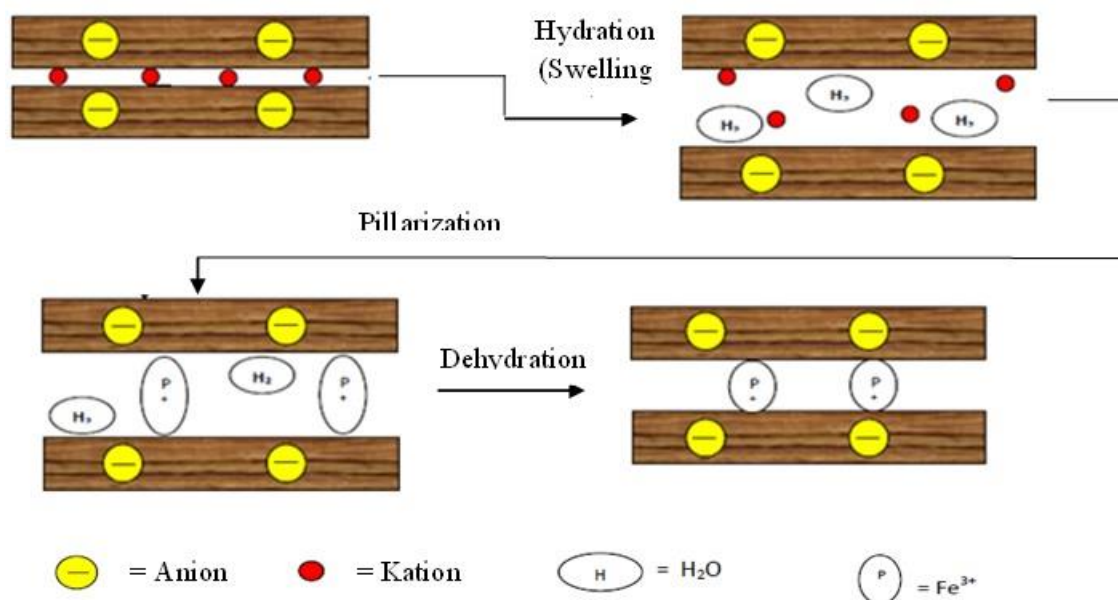


Figure 4. Mechanism of Clay Intercalation [5]

Pillared clay (Pillared Inter Layered Clay) is a clay that has porosity permanent (swelling), Pillared clay aims to improve the thermal stability, the number and type of pores, specific surface area and the acidity of the surface of the clay by adding polycation metal-hidroksi with other cations such as  $\text{Na}^+$ ,  $\text{K}^+$ ,  $\text{Ca}^+$  into the clay interlayer. Furthermore, by calcination will transform into clusters of metal oxides polycation rigid there by inhibiting damage interlayer space [5].

Intercalation was done by replacing the cations in the interlayer region with other metal cations that have a charge and large size. In this pemilaran, the greater the coating so it can be used for filtration of seawater. The process of intercalation by polikation consists of three parts, namely A) polymerization of polycation such as Al (III), Ga (III), Ti (IV), Zr (IV), Fe (III), Cr (IV) and others. Intercalation polikation into the interlayer clays resulting in substitution of cations naturally (Na and Ca) and C) calcination at high temperatures which is essential for the calcination polikation metal entered will experience dehydration and dehydroxylation clusters of metal oxides that keep the room interlayer permanently [10]. Mecanism of clay intercalation as shown in Figure 4.

Homogeneity and strenght of pillared clay were affect by pillared clays synthesis condition such as

concentration of metal ions, temperature of preparation, the standing time and temperature, the ratio of metal and clay, and calcination temperature.

In this research, membrane pillared clays  $\text{FeCl}_3$  will be used as a means of sea water filtration. The sea water that will be used has an 2758 ppm TDS. The results of the filtration rejeivity measured by comparing the results of rejection before and after filtration of seawater.

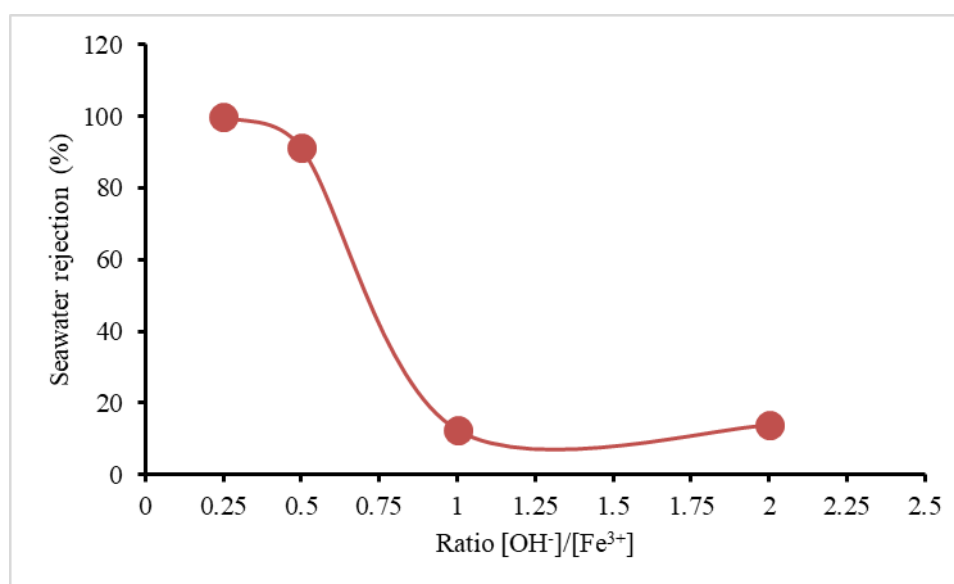
From the table 1 can be in the know of the still one ratio  $[\text{OH}] / [\text{Fe}]$  the most good, namely membrane pillared clays  $\text{FeCl}_3$  with a ratio of 0.25 which is 99.20232%.

Rejeivity obtained was 99.20232%; 57.36041%, 8.629442%; 7.940537% and the concentration of salt solution as shown in Figure 4. Rejeivity was decreased caused by difference increasing of ratio  $[\text{OH}]/[\text{Fe}]$ .

The filtration process generally can occur due to the membrane pore size smaller than the particle size that will be filtered. The aim is that the filtered particles can not pass through the filter pores so only the particles were relatively small can run through the pore filters.

Table 1. Seawater rejection

Seawater rejection			
The ratio [OH]/[Fe]	The concentration early	The concentration end	Rejection
0,25	2758	22	99,20232 %
0,5	2758	1176	57,36041 %
1	2758	2539	7,940537 %
2	2758	2520	8,629442 %
2,5	0	0	0

Figure 5. Rejektivty Chart of membrane pillared clays FeCl<sub>3</sub>

## Conclusion

Membranes have successfully created pillared clays pillared clays based FeCl<sub>3</sub> using simple mixing method for memfiltrasi sea water drinkable. Sea water filtration process showed different results depending with rejection valor. The best rejektivty [OH] / Fe at the ratio of 0.25 which the purity was 99%.

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