

Removal of TDS, Fe, Oil and Greases of Laboratory Wastewater using Ceramic Membrane Technology

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Abstract

Biochemistry Laboratory and Medicinal Chemistry as supporting facilities for practicum and research results in wastewater, which contains organic matter and suspended solids, such as TDS, Fe, oil and greases. If wastewater discarded directly to the drainage without passing sewage treatment system, then it potentially pollutes the environment and endangers the lives of living creatures. The purpose of this research is to evaluate the effectivity of the filtration system using ceramic membrane filtration technology to reduce the amount of TDS, Fe, Oil and Greases on laboratory's wastewater. The filtration process is using ceramic membrane made of clay, activated carbon from lignite, and Fe powder. Pretreatment is done with the neutralization using NaOH 5N and coagulation using aluminum sulfate 400 mg/L. Variable in this research is the operating time of 5 and 20 minutes with operating pressure 1 kg/cm². The result of this research shows that after 20 minutes operating, the reduction percentage of TDS, Fe, oil and greases after the process using ceramic membrane filtration technology were 0.97; 96.01; and 91.03 %, respectively. As the conclusion, the ceramic membrane is able to reduce the parameter of wastewater optimally.

Keywords: ceramic membrane, coagulation, laboratory wastewater, lignite coal, neutralization

Abstrak (Indonesian)

Laboratorium Biokimia dan Kimia Obat sebagai sarana penunjang praktikum dan hasil penelitian pada limbah cair yang mengandung bahan organik dan padatan tersuspensi, seperti TDS, Fe, minyak dan lemak. Jika air limbah dibuang langsung ke saluran pembuangan tanpa melewati sistem pengolahan limbah, maka berpotensi mencemari lingkungan dan membahayakan kehidupan makhluk hidup. Tujuan dari penelitian ini adalah untuk mengevaluasi efektivitas sistem filtrasi menggunakan teknologi filtrasi membran keramik untuk mengurangi jumlah TDS, Fe, minyak dan lemak pada air limbah laboratorium. Proses filtrasi menggunakan membran keramik yang terbuat dari tanah liat, karbon aktif dari lignit, dan serbuk Fe. Pretreatment dilakukan dengan netralisasi menggunakan NaOH 5N dan koagulasi menggunakan aluminium sulfat 400 mg/L. Variabel dalam penelitian ini adalah waktu operasi 5 dan 20 menit, tekanan operasi 1 kg/cm². Hasil penelitian menunjukkan bahwa waktu operasi 20 menit, masing-masing persentase reduksi TDS, Fe, minyak dan lemak setelah proses menggunakan teknologi filtrasi membran keramik adalah 0,97 %, 96,01 %, dan 91,03 %. Secara keseluruhan dapat disimpulkan bahwa membran keramik mampu mereduksi parameter air limbah secara optimal.

Kata Kunci: membran keramik, koagulasi, air limbah laboratorium, batubara lignit, netralisasi

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INTRODUCTION

Biochemistry Laboratory and Medicinal Chemistry is one of the supporting facilities for practicum and research which produces wastewater as the remnant. Wastewater comes from expired ingredients, used chemical, products from the practicum, washing water from cleaning the equipment, and the tasted sample leftover. Quantitatively, the wastewater produced is relatively small. However, based on the characteristics it can be categorized as poisonous and hazardous waste. That wastewater also contains organic matters and suspended solids. When wastewater produced directly discarded into the drainage without passing the wastewater treatment system first, then it can pollute the environment which is going to destroy either aquatic or terrestrial ecosystem [1]. From the immense impact caused by wastewater, it is necessary to apply a proper wastewater treatment technique in order to fulfil the Quality Standard according to Ministry of Environment and Forestry Regulation No.5 in 2014.

One of the methods to process wastewater which has been mostly used and the result has fulfilled the Environmental Quality Standard is the application of ceramic membrane filtration technology [1,2]. The advantage of the ceramic membrane itself is it has excellent mechanical, thermal, and chemical stability that it is able to reduce the amount of pollutant within the wastewater [3,4,5]. Ceramic membrane made of 87.5% clay 10% rice bran and 2.5% Fe powder can reduce cadmium, COD, TDS, and TSS [6]. The carbonated tubular microfiltration membrane made from coal with phenolic resin and organic additive has been implied successfully on colored wastewater [7]. However membrane technology has a disadvantage on the decrease of permeate flux related to the operating time, because of the accumulation of feedback control system on pores at the membrane's surface [8]. So that is why, the application of neutralization and coagulation pretreatment is an effective step in reducing fouling membrane, which purpose to increase the membrane's performance [9], resulting the elongated membrane's life [10].

In the coagulation process, with the addition of positively charged chemicals, negatively charged particles of suspended solids, colloidal particles, and dissolved materials in wastewater bind to other particles to form flock so that they can be easily precipitated and filtered [11]. The process of Coagulation wastewater becomes more effective when the wastewater is neutral [12], therefore a neutralization pretreatment is needed which aims to make the pH of the wastewater neutral by adding bases

or acids. This research focused on treating laboratory wastewater using ceramic membrane technology, with neutralization and coagulation pretreatment so that it expected to reduce the content of TDS, Fe metals, oils and greases in accordance with the wastewater quality standards set by the government.

MATERIALS AND METHODS

Materials

The materials used in this study was simulated wastewater from the Laboratory of Biochemistry and Medical Chemistry, Faculty of Medicine, Sriwijaya University which was made by dissolving 45175 g of NaCl each; 28.14 g of $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$, 1607.5 mL of palm oil, and 5 L of 3M H_2SO_4 into 250 L of distilled water, in order to obtain simulated waste containing TDS, Fe, oil and greases and pH with levels of successively 180700 mg/L, 23.3 mg/L, 5851.3 mg/L, and pH of 1.2. This research was conducted from January to July 2022 at the Integrated Research Laboratory of the Postgraduate Program, Laboratory of Biochemistry and Medical Chemistry, Faculty of Medicine, UPT. Integrated Laboratory, and Chemical Engineering Laboratory of Sriwijaya University Polytechnic, Indonesia.

Lignite Coal Carbonization

Lignite coal obtained from Tanjung Enim, South Sumatra. The coal in the form of lumps is dried in the sun for 1 day. Then it is crushed into smaller size using a mortar and pestle. After that, it was carbonized using a furnace at a temperature of 700 °C for 3 hours and cooled in a desiccator. Finally, it is mashed into a size of 100 mesh.

Lignite Coal Chemical Activation

The carbonized lignite coal was immersed in 3M hydrochloric acid (HCl) and left for 24 hours. The slurry was separated using filter paper and the solid phase was washed with distilled water to pH 7 (neutral). Then the activated coal was dried using an oven at a temperature of 110 °C for 2 hours.

The Manufacturing of Ceramic Membrane

The ceramic membrane designed in a tube form, made from a mixture of clay, iron powder and activated carbon of lignite coal with an inner diameter of 5 cm, an outer diameter of 7 cm, a thickness of 1 cm and a length of 25 cm. The membrane housing is made of fiber glass with an outer diameter of 9 cm, an inner diameter of 8.5 cm and a length of 30 cm. The procedure for making the ceramic membranes is that the clay is sliced thinly and dried for 2 days, and then the clay is mashed and sieved using a 400 meshes sieve. The iron powder was sieved using a 400 mesh

sieve. The activated carbon of lignite coal was sieved using a 400 meshes sieve. The clay, activated carbon lignite coal and iron powder were mixed until smooth in the ratio of 87.5; 2.5; and 10%. Then we added the water little by little into the mixture of the membrane until it forms a paste (gel), so that it is easy to mold a ceramic membrane with a ceramic membrane molder. After molding, the dough is removed from the membrane mold, and then dried at room temperature for 7 days. After drying, the membrane is burned (sintered) at a temperature of 400 °C for 9 hours.

Pretreatment the Wastewater with Neutralization

The process of neutralization was carried out by adding NaOH 5N with a pretest-posttest design, by measuring the pH before treatment (pretest) and after treatment (posttest). 250 ml of each wastewater was put into 6 glass beakers. Then, 5 N NaOH was added with various doses: 0 (control), 1, 2, 3, 4, and 5 mL. After that, stirred for 2 minutes and the pH of each wastewater were measured. The addition dose that is almost neutral is applied to the wastewater neutralization process.

Pretreatment the Wastewater with Coagulation

The wastewater was coagulated using coagulant $\text{Al}_2(\text{SO}_4)_3 \cdot 14\text{H}_2\text{O}$ (alum). The dose of alum used is determined by the jar test method. At this stage, wastewater is fed into each beaker. Then the alum was added with dosage variations of 100, 200, 300, 400, and 500 ppm. After that, fast stirring was carried out for 1 minute with a rotating speed of 120 rpm and slow stirring at a rotational speed of 20 rpm for 20 minutes. Then the deposition was carried out for 15 minutes until the floc formation was completed. The value of turbidity using a turbid meter. Coagulation wastewater was analyzed for parameters of TDS, Fe metal, Oil and Greases

Wastewater Treatment with Ceramic Membrane

Wastewater from the laboratory was put into a storage tank with a capacity of 250 L. Wastewater from the storage tank was streamed using a pressure pump to housing-1 which contained a sponge filter with a pore diameter of 3 μm . The pump pressure was set to 1 kg/cm^2 by adjusting the feed flow rate using flow meter bait. Wastewater that has been filtered with a sponge filter with a pore diameter of 3 μm was streamed into housing-2, housing-3, housing-4, each of which contained a sponge filter with a pore diameter of 1 μm , manganese ferollite, and activated carbon.

Laboratory wastewater that has passed through the sponge filter, manganese ferollite, and activated carbon, the wastewater was streamed to housing-5 which contained a ceramic membrane. Laboratory

wastewater that has passed the membrane process was accommodated in a container as permeate. Wastewater samples that have passed the complete separation process are taken every 5, 10, 15, and 20 minutes. For each sampling, the volume of permeate produced is calculated. Permeate was analyzed for parameters of TDS, pH, Fe metal, oil and greases. Instrument SEM-EDS is used to determine the blocked on to the membrane wall.

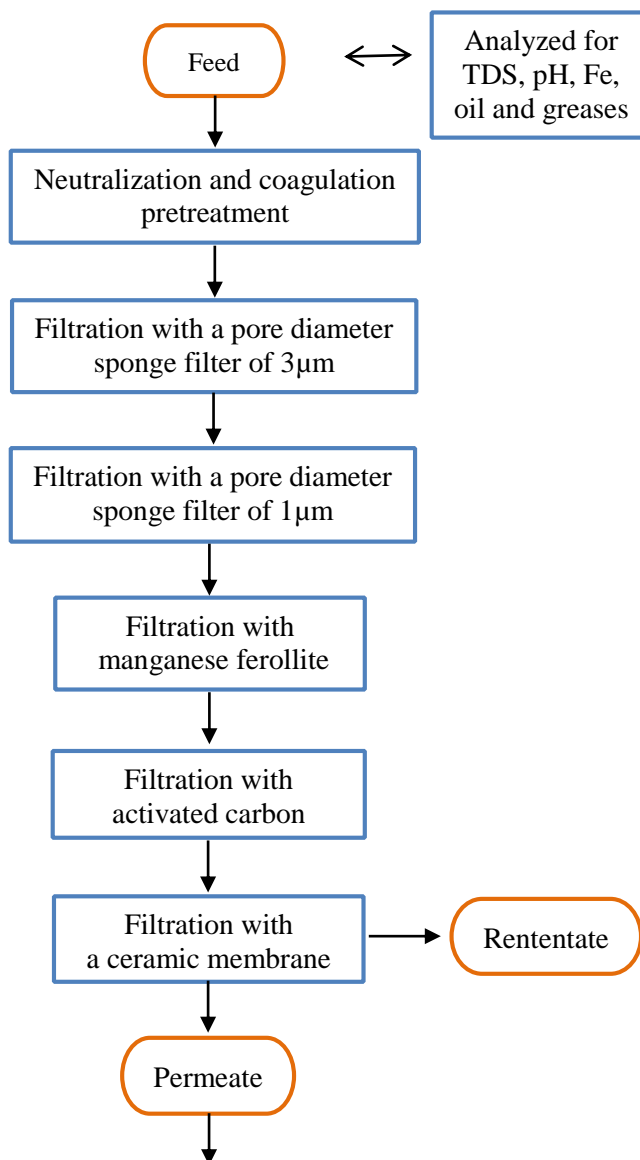


Figure 1. A Flowchart of the Process

Analysis Data

Data analysis was done by using comparison. The data obtained will be presented in the form of numbers, descriptions, tables and graphs. The flux value can be calculated based on the equation:

$$J = \frac{v}{(A \times t)} \quad (1)$$

Where J is flux ($L/m^2 \cdot h$); V is volume of the permeate (L); A is area of the membrane (m^2); and t is time (h).

RESULTS AND DISCUSSION

The Analysis Results to the Initial Sample

The wastewater used in this research was from the Biochemistry and Medical Chemistry Laboratory, Faculty of Medicine, Sriwijaya University, South Sumatra. Before the filtration process carried out, the wastewater was first analyzed with the parameters of TDS, Fe metal, oil and greases. The results of the initial sample analysis of laboratory wastewater are shown in **Table 1**.

Table 1. Results of Analysis of Initial Laboratory Wastewater Samples

Parameters	Unit	Concentration	Quality Standards
pH		1.2	6-8
TDS	mg/L	182.5	50
Fe	mg/L	81.4	20
Oil and Greases	mg/L	248.3	200

Quality Standards as per PERMEN-LH RI No.5 of 2014

The Characteristics of the Ceramic Membrane

In this research the characteristics of ceramic membranes using Scanning Electron Microscopy (SEM) and Energy Disperse Spectroscopy (EDS) as seen in **Figure 2**.

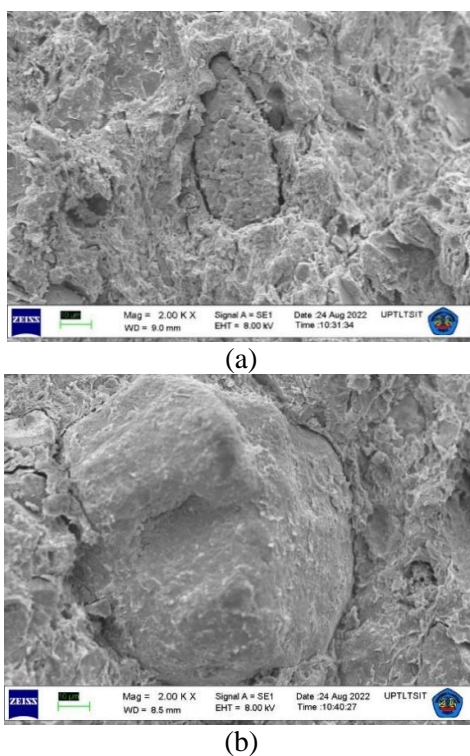


Figure 2. SEM Image of Ceramic Membrane (a) Before and (b) After Processing

In the analysis using SEM, it is very important to determine the morphological structure of the membrane, where the surface structure and cross-section are seen microscopically on the photos produced by SEM. The membrane surface and pore size affect the performance of the membrane in the filtration of a material [13].

In **Figure 2a**, it can be seen that the surface morphology of the membrane is porous which is uneven and asymmetrical (not homogeneous), and some resembles a rock (sponge). This uneven pore structure can be influenced by temperature, type, size and composition of the membrane constituent [14].

In **Figure 2b** showed a cross section with gravel-like grains and a relatively thicker cake layer formed on the membrane surface after the filtration process. The surface was irregular with large lumps and agglomerates formed in the filtration process [9]. From the results of this SEM, most of the fouling mechanism by the fouling layer is thought to be related to the pores which were the active areas of the membrane.

The impurities particle from the feed which are larger than the membrane pores will be retained and pass components that are smaller than the membrane pores. Impurity particles will clog the pores and accumulate on the surface of the membrane, causing a decrease in the effectiveness of the membrane which is accompanied by a decrease in the effectiveness of the membrane with a decrease in membrane flux [10]. There has been a change in the morphological structure of the membrane surface to be nonporous after being used as a filter.

In addition, the element analysis used EDS (**Table 2**). The results showed the constituent elements of the membrane. This was indicated by the spectra of each element in **Figure 3**.

Table 2. EDX Analysis of Ceramic Membrane Before and After Processing

Element	Mass (%)	Atom (%)
Before Processing		
O	46.95	57.72
Si	32.42	22.71
Al	15.64	11.40
C	4.99	8.17
Total	100.00	100.00
After Processing		
O	40.46	47.64
Si	25.64	17.19
Al	9.88	6.89
C	16.68	26.16
Zn	7.35	2.12
Total	100.00	100.00

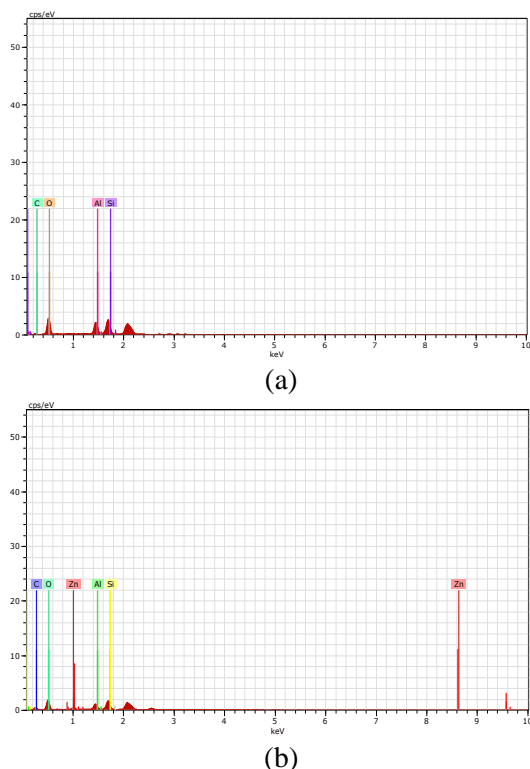


Figure 3. EDX Analysis of Ceramic Membrane (a) Before and (b) After Processing, where X axis is energy; Y axis is signal intensity

In the ceramic membrane which was previously used for wastewater treatment, several elements were seen, namely Carbon (C), Oxygen (O), Aluminum (Al), and Silica (Si). These elements come from the membrane-forming material, namely clay. The absence of Fe was thought to be due to the imperfect mixing process in the manufacture of ceramic membranes. The mass of each element before and after wastewater treatment changed, where Oxygen from 46.95 to 40.46%, Silica from 32.42 to 25.64%, Aluminum from 15.64 to 9.88%, and Carbon from 4.99 to 16.68%. Because the sampling was only from one area and only shows the dominant element, the results of the analysis did not represent all the elements that make up the membrane [15].

The Connection between Fluxes to Operation Time

Permeate flux was one of the factors that determined the optimal working of a membrane. In this study, at 5 minutes the processing produced a flux of $23.34 \times 10^2 \text{ L/m}^2 \cdot \text{hour}$, then there was a decrease in flux at an operating time of 10 minutes with a flux of $7.11 \times 10^2 \text{ L/m}^2 \cdot \text{hour}$ and at an operating time of 15 minutes produced a flux of $4.25 \times 10^2 \text{ L/m}^2 \cdot \text{hour}$. The flux decreased until the operating time was 20 minutes with a flux value of $3.03 \times 10^2 \text{ L/m}^2 \cdot \text{hour}$. The Flux can be seen in Figure 4.

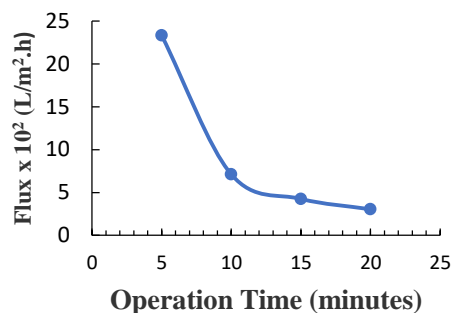


Figure 4. The Relationship of Flux to Operation time

Figure 4 shows a decrease in the value of the permeate flux with the longer the wastewater treatment process. This is due to the increasing number of polluting particles that are retained in the pores of the membrane and accumulate on the surface of the membrane, resulting in the formation of a gel or fouling layer. The fouling layer will clog the membrane pores and increase the resistance (barrier) on the membrane surface [16].

This fouling came from colloidal particles, dissolved salts, dissolved organic solvents, microorganisms, protein molecules, and other particulates. The presence of this source of fouling caused loss of productivity and quality of membrane degradation, so that the effectiveness of the membrane as a selective barrier was getting smaller. Therefore, it was very important to evaluate the performance of the membrane before using it in real applications, namely by measurement of flux, rejection factor, separation factor, capacity, membrane activity, membrane fouling, membrane compaction, discharge rate, and barrier properties [10].

Analysis of pH, TDS, Fe Metal, Oil and Greases

The analysis of all important parameter were shown in Figure 5 and Table 3.

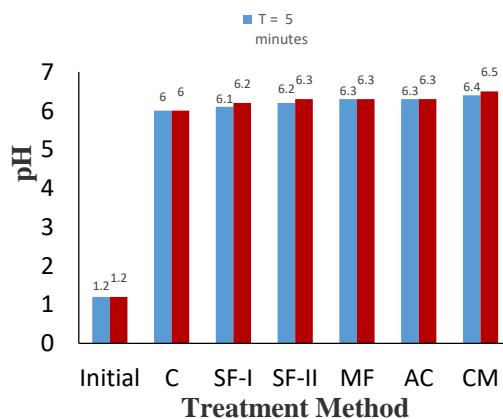


Figure 5. pH of Laboratory Wastewater on Treatment Method

Based on **Figure 5**, the pH before processing is still very acidic, i.e. 1.2. After the neutralization pretreatment process using NaOH 5 N solution was done, there was an increase in pH of 7.6. Because pH can affect the solubility of metals, the addition of NaOH causes some metals to precipitate in the form of metal hydroxides. The addition of NaOH will precipitate Fe³⁺ metal as ferric hydroxide Fe(OH)₃ which is insoluble with a reddish color [12]. The addition of pH also aims to improve the performance of alum as a coagulant because alum will work more effectively to precipitate impurities dissolved in wastewater in the pH range of 6-8 [17]. The pH of the wastewater after the coagulation process became 6.0 and continued to increase from 6.3 on the activated

carbon filter to 6.5 after passing through the ceramic membrane process. This is because the source of acidic materials (containing H⁺ ions) in wastewater has been blocked on the surface of the membrane wall, so that the pH of the permeate has met environmental water quality standards.

From **Table 3**, it can be seen that the effectiveness of alum coagulant in reducing TDS in laboratory wastewater is still at a concentration of 179.133 mg/L with a reduction percentage of 0.87%. The TDS level after the filtration process is still high, namely 178.955 mg/L with a reduction percentage of 0.95%, so it still does not meet the quality standards set by the government.

Table 3. Results of Analysis of TDS, Fe, Oils and Greases in Laboratory Wastewater

Parameters	Operation		Initial	C	SF-I	SF-II	MF	AC	CM	QS*	Reduction (%)
	Time (minutes)	Unit									
TDS	5	mg/L	180,70	179,133	179,092	179,078	179,066	179,047	178,986	2000	0.95
	20	mg/L			179,088	179,071	179,053	179,038	178,955	2000	0.97
Fe	5	mg/L	23.30	2.52	2.49	2.46	2.32	2.13	1.55	5	93.35
	20	mg/L			2.33	2.29	2.15	1.91	0.93	5	96.01
Oil and Greases	5	mg/L	5851.3	8.2	7.9	7.7	7.7	7.6	7.5	10	90.79
	20	mg/L			7.7	7.6	7.5	7.5	7.3	10	91.03

Where SF-I is sponge filter size 3µm; SF-II is spoon filter size 1µm; MF is manganese ferollite filter; AC is activated carbon, CM is ceramic membrane; QS* is quality standards as per PERMEN-LH RI No.5 of 2014; % Red is reduction percentage

The decrease in dissolved solids occurs because the active substances collide and interact with colloidal particles in the wastewater. The interaction that occurs causes a decrease in colloid stability because the charge is neutralized and unites to form a micro flock colloid and then settles [9].

The TDS elimination by coagulation is influenced by pH, so not all metal ions can undergo precipitation. Metal ions that do not undergo precipitation will remain soluble in water, causing the TDS to remain high. The value of the TDS concentration is still high also because it is not diluted before processing so that the pollutant load is still high [10]. Through dilution in the filtration process it will reduce the performance of the membrane, where contaminants are gradually retained, so that it will slow down the occurrence of fouling [16].

Meanwhile, Fe metal showed a very significant decrease after the coagulation process. The effectiveness of coagulant to decrease Fe in laboratory wastewater is very good, reaching 89.18%. This is happening due to the binding reaction between the coagulant and Fe metal. Fe element is generally positively charged and is equivalent to Aluminum sulphate, resulting in ion exchange between Fe and Al

[9]. After passing through the ceramic membrane, the concentration of Fe metal has met the environmental quality standards of PERMEN LH No. 5 of 2014 which is 0.93 mg/L.

In contrast to the change in concentration of Fe, the concentration of Oil and Greases experienced a stable change in concentration after ceramic membrane filtration, but experienced a significant decrease in concentration after the coagulation process. The addition of Aluminum sulphate (Alum) as a coagulant to the concentration of Oil and Greases is very effective reaching 89.93%. Positively charged Al interacts with negatively charged particles resulting in charge neutralization. There is destabilization of the oil droplets and breakdown of the emulsion [17]. There is a difference in the density of the two fluids (Oil and Water), so that the oil droplets will rise to the top and will float due to the density of oil being smaller than water [18].

In the filtration process, a thin layer of Oil is adsorbed onto the membrane surface, where the Oil layer will make the membrane surface hydrophobic. Oil adsorption will be dispersed into the gel. The presence of gel formation will inhibit the flow of effluent and cause a decrease in flux [8,18]. The

concentration of oil and fat has met environmental quality standards with a reduction percentage of 91.03%.

CONCLUSION

The concentration of TDS, Fe metal, oil and greases decreased to 178,955 mg/L, 0.93 mg/L, and 7.3 mg/L with increasing operating time. The percentage of reduction after processing for the TDS pollutant parameter reached 0.97%, while the percentage reduction for the Fe parameter was 96.01% and for the oil and greases parameter it was 91.03%. The metal parameters of Fe, oil and greases after processing have met the quality standards of the Decree of the Minister of the Environment of the Republic of Indonesia No. 5 of 2014 concerning the quality standards of liquid waste, while TDS has not met environmental standards so that further processing is needed.

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