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**RESEARCH PAPER**

# Investigation of direct Coagulation-Flocculation-Ultrafiltration (CFU) at lab-scale constant pressure and flux operation of copper removal

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**Abstract.** Limited heavy metal concentrations in drinking water are harmful. The size-exclusion separation process was found to be a technology for removing heavy metals and organic substances. Although promising, a single ultrafiltration process is insufficient for the separation of heavy metals. Therefore, an additional process is required. The combination of coagulation flocculation followed by ultrafiltration was investigated. In this study, water matrix was used to simulate the worst-case scenario by adding 2 mg of copper to the surface water. For the filtration process, a comparison of single ultrafiltration with and without pretreatment using aluminum sulfate was investigated. Filtration was performed in a lab-scale experiment employing a polyethersulfone (PES) membrane with an average pore size of 30 nm operated at constant flux 120 $L/m^2 \cdot h$ and constant pressure of 0.7 bar. Furthermore, TDS retention, copper concentration, and turbidity were observed. Higher Cu removal was found at filtration under constant flux compared to constant pressure (81% and 66%, respectively. In the case of treated water with coagulation (optimum coagulation of 30 mg/L), higher removal of Cu was observed at constant flux operation compared to constant pressure, with 73% and 89% removal, respectively. Additional coagulation resulted in less membrane fouling during the filtration experiment, which explained the better performance almost double that of single ultrafiltration.

**Keywords:** Copper; coagulation-flocculation; ultrafiltration; membrane performance; Membrane Retention

## 1. Introduction

Humans widely use surface water as a source of potable water for activities. However, surface water resources can be chemically polluted by industry, agriculture, mining activities (Prasad et al., 2020) and domestic wastes (Arum et al., 2019). Water pollution comes from human activity, which produces waste that contains various pollutants such as heavy metals (Briffa et al., 2020; Prasad et al., 2020). Heavy metals that accumulate in water have negative effects on surrounding ecosystems, including plants and biota, which are indirectly contaminated (Dwivedi et al., 2018; Prasad et al., 2020). Salam Lake, located in the University of Indonesia, is a lake that functions to increase groundwater absorption in the surrounding area. The University of Indonesia has a Green

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Campus programme, namely water use and water resource efforts. Salam Lake has the potential to provide clean freshwater. However, previous studies have revealed that a significant amount of heavy metal was found in Salam Lake. Copper contributed the highest concentration among the other heavy metals (Luthfi, 2021).

The World Health Organisation (WHO) and Regulation of the Minister of Health of the Republic of Indonesia No 2/2023 concerning Regulations for Implementing Government Regulation No 66/2014 concerning Environmental Health state that the concentration of copper in drinking water should be a maximum of 2 mg/L. The desorption of Cu in sediments can be disrupted by strong interactions with air, which encourages the precipitation of heavy metals into dissolved or particulate forms (Sun et al., 2014). Unbalanced copper (Cu) levels affect human health in particular in terms of heart function and metabolism, as well as inflammation and resistance to chemotherapy drugs (Bui et al., 2020).

Membrane filtration can remove heavy metals (Abdullah et al., 2019; Carolin et al., 2017). Membrane filtration has several advantages, such as its flexible filtering process, low energy consumption, and high removal efficiency (Rosman et al., 2018). Based on their size, membranes are divided into microfiltration (MF), ultrafiltration (UF), nanofiltration (NF), and reverse osmosis (RO) (Yang et al., 2019). Divalent ions can be removed using nanofiltration, whereas ultrafiltration can only remove suspended solids, colloids with a size of >0.1 µm, organic particulates, and microorganisms (He et al., 2019; Yang et al., 2019). Heavy metals and organic pollutants can be separated using nanofiltration (NF) and reverse osmosis (RO) membranes. Although efficient, these methods consume more energy at larger operating pressures than UF and MF (Muthumareeswaran & Agarwal, 2014). As mentioned before, ultrafiltration cannot remove heavy metals but can produce a higher flux than RO at low energy (Yaqub et al., 2022).

Heavy metal removal can be increased by a combination of coagulation/flocculation and ultrafiltration as well as membrane-fouling reduction (Ghadge et al., 2015; Ran et al., 2020). Combining coagulation/flocculation and ultrafiltration is widely used in water and wastewater treatment to reduce heavy metal contamination. The process of removing heavy metals using coagulation/flocculation involves the binding of heavy metals with coagulants/flocculants that combine with humic or colloidal acids (Sylwan & Thorin, 2021). Xing Du et al., (2014) showed that ultrafiltration without coagulation only removed 9% of Sb (III) metal, and the use of coagulants reached 66-96%. Sum et al., (2021) reported that the percentage of copper removed without surfactant was 26.55%, whereas the level of copper removed with surfactant was 66.31%.

In correlation with membrane performance, high membrane retention resulted in membrane fouling. Membrane fouling is a common problem in membranes and causes a decrease in membrane flux, high transmembrane pressure (TMP), and high maintenance costs (Meng et al., 2019). Fouling also occurs due to the narrowing of the membrane pores, the adsorption of solutes by the membrane, the deposition of flocs on the surface of the membrane, or the compaction of the cake layer on the surface of the membrane. It is influenced by the structure of the membrane's characteristics and operating conditions i.e. applied pressure, retention time, or cross-flow rate (Xianjun Du et al., 2020). Constant flux and constant TMP measurements have been widely reported for comparing membrane fouling due to operational modes. Lee et al., (2008) tested fouling due to natural organic matter (NOM) under constant pressure and flux conditions. The results show that constant flux filtration provides favourable conditions, namely, irreversible fouling is reduced at low flux, but the results are less clear and consistent when compared with a constant pressure of 10–90 kPa (Lee et al., 2008). The fouling results due to alginate in gel form, namely, resistance correlated well with flux at constant flux, while constant resistance was observed and with a wider range, flux decreased at constant pressure (Sioutopoulos & Karabelas, 2016). This study aims to determine the suitable performance of membrane separation processes combining direct coagulation/filtration and ultrafiltration (CFU) for Cu removal. Furthermore, the membrane performance in a constant flux and constant pressure operational mode was investigated.

## 2. Mathodology

### 2.1. Material

Surface water samples were collected from Salam Lake at the University of Indonesia. Samples characteristics were analysed before treatment with parameters of pH, temperature, turbidity, and total dissolved solid (TDS). Turbidity measurements were conducted using a turbidimeter, and a digital pH metre was used for pH measurements. Temperature measurements were conducted using a thermometer and TDS measurements were conducted using a potentiometer. All chemical reagents used in the experiments were $CuSO_4.5H_2O$ crystals, NaOH, NaOCl, and $Al_2(SO_4)_3.12H_2O$. PES flat-sheet membranes (Sterlitech, supplied by United State of America) with an effective membrane surface area of 17.35 $cm^2$ and an average pore size 0,03 µm. The virgin membrane was soaked in NaOH at pH 12 for 24 hours to remove impurities.

### 2.2. Experimental setup

#### 2.2.1. Modified water surface

The collected surface water was modified by adding a synthetic solution of $CuSO_4.5H_2O$ crystals. 20 ml of 100 ppm synthetic solution were taken and added to 1 L of collected surface water to obtain a copper concentration of 2 mg/L. turbidity, pH, total dissolved solid, and Cu parameters were analysed before the proposed treatment. The synthetic water quality is presented in Table 1.

**Table 1.** Synthetic water quality

| Parameter | Values | Unit |
|---|---|---|
| pH | 7.01±0.1 | |
| Turbidity | 12.8±4.3 | NTU |
| TDS | 128.7±8.5 | mg/L |
| Cu | 2 | mg/L |

#### 2.2.2. Jar test experiment coagulation/flocculation-ultrafiltration experiments

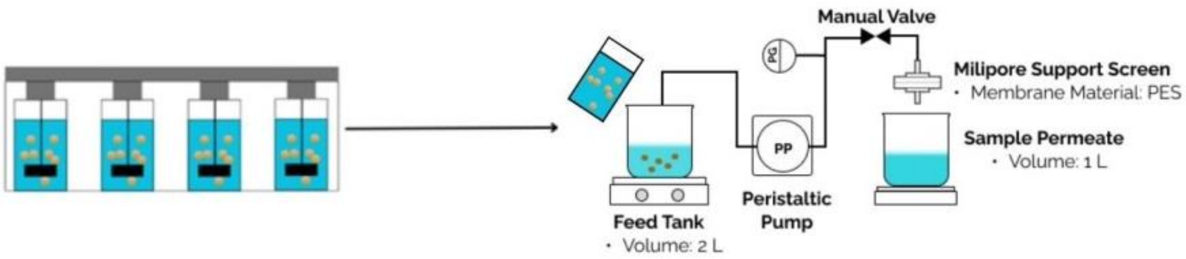
The coagulation-flocculation process was carried out in a six-jar reactor. Before performing the jar test, we prepare alum by diluting $Al_2(SO_4)_3.12H_2O$ with distilled water. A feed sample containing Cu was placed in a 1-L glass beaker. Alum was then placed in a pipette according to the coagulant dosage into a beaker glass. The jar test was first mixed rapidly at 100 rpm for 1 min, followed by slow mixing at 40 rpm for 15 min and 20 min. After settling, the samples were carefully filtered using filter paper so that there were no disturbing flocs, and turbidity, pH, and TDS were measured.

#### 2.2.3. Flocculation-Ultrafiltration Experiments

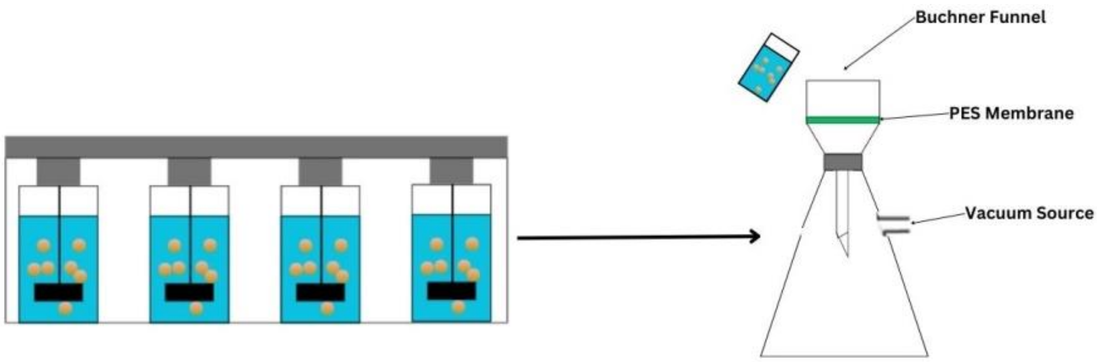
The experimental setup for the constant flux is illustrated in Figure 1. After the jar test (without settling), each coagulated solution was slowly placed into a glass beaker containing a magnetic stirrer to create weak agitation and prevent floc settling. After that, the feed water flowed through the membrane, and the sample was filtered. A peristaltic pump was used to maintain constant flux at 120 liters per square meter per hour (LMH) or 2 rpm. During filtration, the volume of filtrate produced on an analytical balance reading was recorded at 1-min intervals for the first 10 min and every 5 min after 10 min. Turbidity, pH, and TDS were measured. The direct ultrafiltration process was named CF, coagulation-flocculation and ultrafiltration with 30 and 50 mg/L alum were named CF-30 and CF-50, respectively.

The experimental procedure for constant pressure was carried out using a vacuum filter. The coagulation solution without sedimentation is stirred with a magnetic stirrer. Then, a sample of

25 ml was taken and filtered through the membrane in the Buchner funnel. A vacuum pump provided a constant pressure of 0.7 bar. During the filtration process, a stopwatch was used to record the time until 25 ml of water was completely filtered. The experimental setup for constant pressure testing is illustrated in Figure 1. Turbidity, TDS, and Cu were measured. The direct ultrafiltration process was named CP, coagulation-flocculation and ultrafiltration with 30 and 50 mg/L alum were named CP-30 and CP-50, respectively.




(a)




(b)

**Figure 1.** Experimental setup: a) constant flux; b) constant pressure

The final turbidity, TDS, and Cu concentrations were calculated using Eq.(1), where membrane retention (%), Cp is the solute concentration in the permeate, and Cf is the solute concentration in the feed solution:

$$R = \left(1 - \frac{C_p}{C_f}\right) \times 100\% \quad (1)$$

The volume of permeate collected for a specific time was calculated from the permeability of the membrane using Eq.(2), where W is the permeability of the membrane, Jw is the permeate flux, and ΔP is the transmembrane pressure:

$$W = \frac{J_w}{\Delta P} \quad (2)$$

N Normalised permeability, W' was calculated using Eq.(3), where Wvsp is the specific-filtered volume permeability and Wo is the permeability of the pristine membrane:

$$W' = \frac{W_{Vsp}}{W_o} \quad (3)$$

The specific-filtered Volume, Vsp, was calculated using Eq.(4), where t is the filtration time in hours:

$$V_{sp} = J_w \times t \quad (4)$$

## 3. Result and discussion

### 3.1. Coagulation-floculation proses

The coagulation/flocculation process was performed using a general jar test procedure. During the experiments, analytical parameters such as turbidity, TDS, and pH were measured. the coagulation test results for alum doses of 10, 30, 50, and 70 mg/L are shown in **Error! Reference source not found.**. During the experiment, the pH of the water was measured and revealed at pH at normal condition (6,8±0,1). At a coagulant dose of 10 mg/L, the turbidity and TDS results were 89% and 5%, respectively. In parallel, at a dose of 30 mg/L, turbidity and TDS were removed in the 90% and 2% range, respectively. At a coagulant concentration of 50 mg/L, turbidity removal and TDS were 97% and 4%, respectively. At the highest coagulant concentration (70 mg/L, the removal of turbidity and TDS was 87% and 1%, respectively.

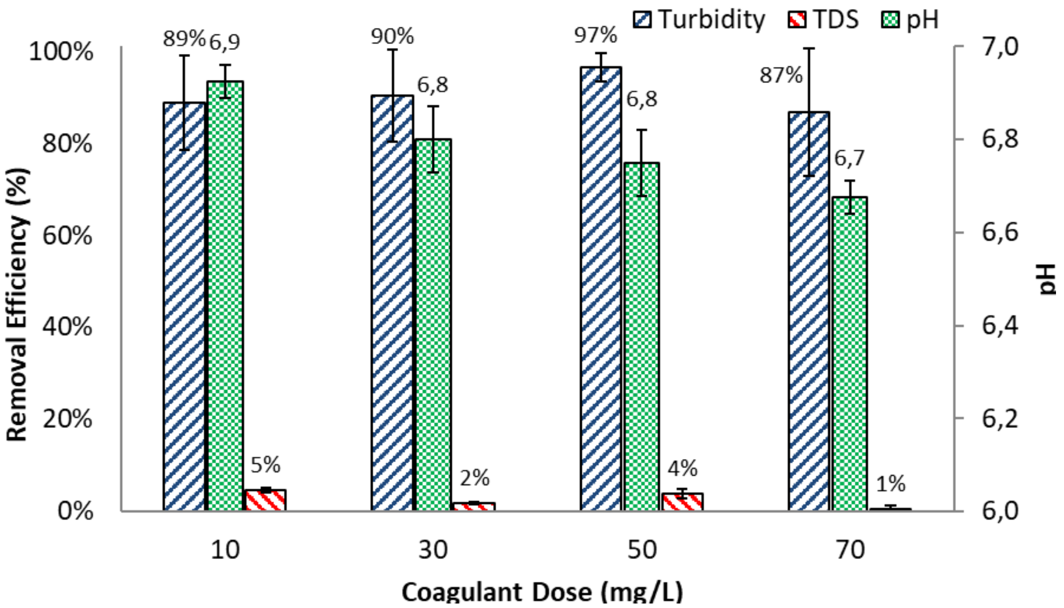



**Figure 2.** Effect Jar Test on removal efficiency (%) of Turbidity, TDS, and pH value

Focusing on water turbidity, the removal of this parameter showed linear correlation. Within the mentioned coagulant doses, the removal rate increased from 89% to 97 %. Interestingly, at 70 mg/l, the removal efficiency tended to decrease by approximately 87%. The decreased removal efficiency might indicate overdosing of the coagulant. This process can lead to particle de-stabilisation due to incomplete particle neutralisation which increases turbidity (Karnena et al., 2022). Despite a clear decrease in efficiency, considering the deviation in the measurement, one may consider that the removal was comparable. In parallel, the TDS parameter resulted in an insignificant reduction and revealed that all coagulant doses only reduced TDS by 1%–5%. Similar results were obtained (Bergamasco et al., 2011) in which TDS was removed via coagulation/flocculation-sedimentation (CFS) with an alum content of 7.5%.

Based on these results, the CFS process with aluminium sulphate could not significantly remove TDS. The pH of the solution tended to decrease as the alum dose increased. The decrease in water pH is caused by sulphuric acid due to the presence of alum, which reacts with water and lowers the pH. Despite the various decreases in the pH values for all variations in the alum doses, one might consider that the pH was found to be comparable. Furthermore, based on the obtained results, the jar test result showed the highest turbidity removal (97% at a dose of 50 mg/L alum. Therefore, a coagulant dose of 50 mg/L alum was found to be the optimum dose and was further employed in subsequent experiments.

### 3.2. Performance of The Combined Coagulation/Flocculation (CFU) System

The combined coagulation/flocculation and ultrafiltration (CFU) processes for Cu removal in modified surface water. The coagulant concentration was based on a prior experiment, as explained in the previous chapter. Additionally, a direct ultrafiltration process under different operation conditions (i.e., Constant Pressure and Constant Flux) was also carried out for comparison with the combination process. The doses of 30 and 50 mg/l alum were also carried out in this experiment.

The results of the removal of the combine process at certain parameters (i.e., turbidity, Copper, and TDS) are shown in **Error! Reference source not found.**. During the filtration e xperiment under constant pressure (CP) condition without pretreatment of coagulation process, removal of turbidity, copper, and TDS was observed at 96%, 3%, and 66%, respectively. In parallel, for filtration constant pressure pretreated with coagulant 30 mg/L coagulant, the removal of turbidity, copper, and TDS was estimated to be 97%, 5%, and 73%, respectively. At the highest concentration of 50 mg/l coagulant, turbidity, copper, and TDS were removed at 97%, 1%, and 55%, respectively. At Constant Flux (CF) operation without coagulation pretreatment process, removal of turbidity, Copper, and TDS concentrations were calculated as 95%, 3%, and 82%, respectively. Moreover, filtration of constant flux with a coagulant dose of 30 mg/l resulted in removal efficiencies of 97%, 3%, and 89% for turbidity, copper, and TDS, respectively. Furthermore, removal efficiencies of 93%, 0%, and 72% for turbidity, copper, and TDS were observed under constant flux operation, respectively. From all the experiments conducted, it can be seen that the turbidity removal for all experiments reached ±96%. On the other hand, the removal of TDS for experiments only reaches 1-5%. This could be explained by the fact that ultrafiltration could remove suspended solids, colloids with sizes > 0.1 μm, and organic particulates (Yang et al., 2019).

Comparing filtration with different concentrations and without treatment, interesting results were revealed. The constant pressure filtration shows that the copper removal efficiency increased from 66±3% to 73±2% and decreased to 55±3% with CP, CP-30, and CP-50, respectively. The Cu removal increased with constant flux filtration. The efficiency of copper removal increased from 82±2%, 89±2% and decreased to 72±3% with CF, CF-30, and CF-50, respectively. The results showed that an additional 30 mg/L alum was able to effectively remove copper. It can be explained that a positive charge contained in coagulants is attached to other heavy metals, which have a negative charge (Zheng et al., 2022). The presence of Cu in lake water can undergo reactions with organic compounds, resulting in the formation of flocs when combined with alum. These flocs can subsequently accumulate on the membrane surface. However, removal of Cu using CP-50 and CF-50 decreased Cu removal. Differences in coagulant dosage cause floc characteristics such as floc size and strength. Feng et al., (2015) showed that flocs of large size and loose structures can be damaged when the pressure on the cake layer increases, accelerating membrane fouling. Floc damage during filtration can decrease the Cu removal efficiency. Ran et al., (2020) also found that fouling can experience cracks due to larger flocs, such that the coagulated water comes in direct contact with the surface of the membrane through the gaps, thereby reducing the removal of heavy metals. Al ions also affect the efficiency of heavy metal removal. Wang et al., (2020) also showed that the coagulation-flocculation process reduced the efficiency of Cu removal compared to direct ultrafiltration due to Al ions. Al has stronger electrostatic attraction with negatively charged membranes than heavy metal cations. Excessive doses of alum result in a higher interaction of aluminium (Al) and copper (Cu) ions. This behavior leads to easier passage of unreacted Cu ions through the membrane.

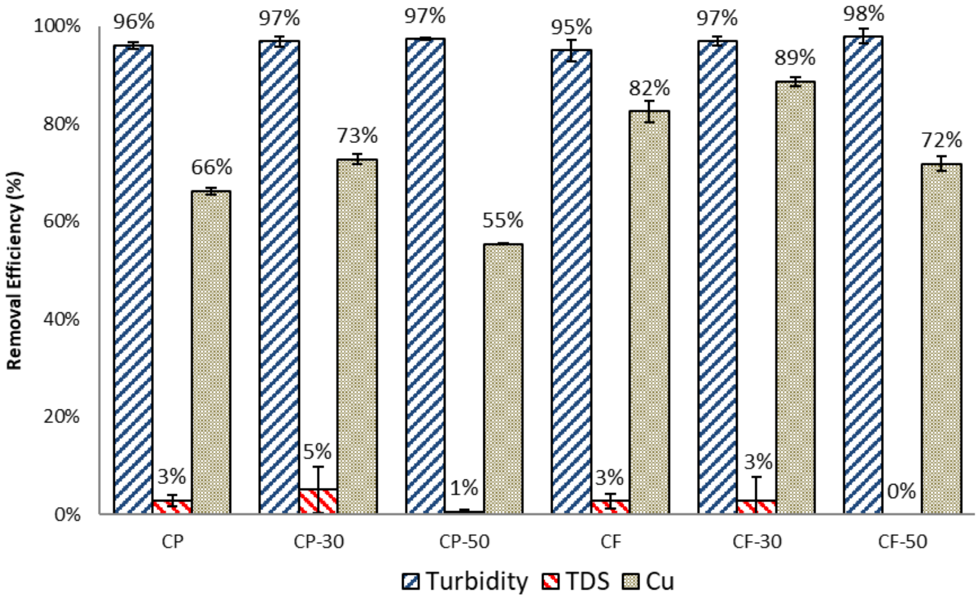
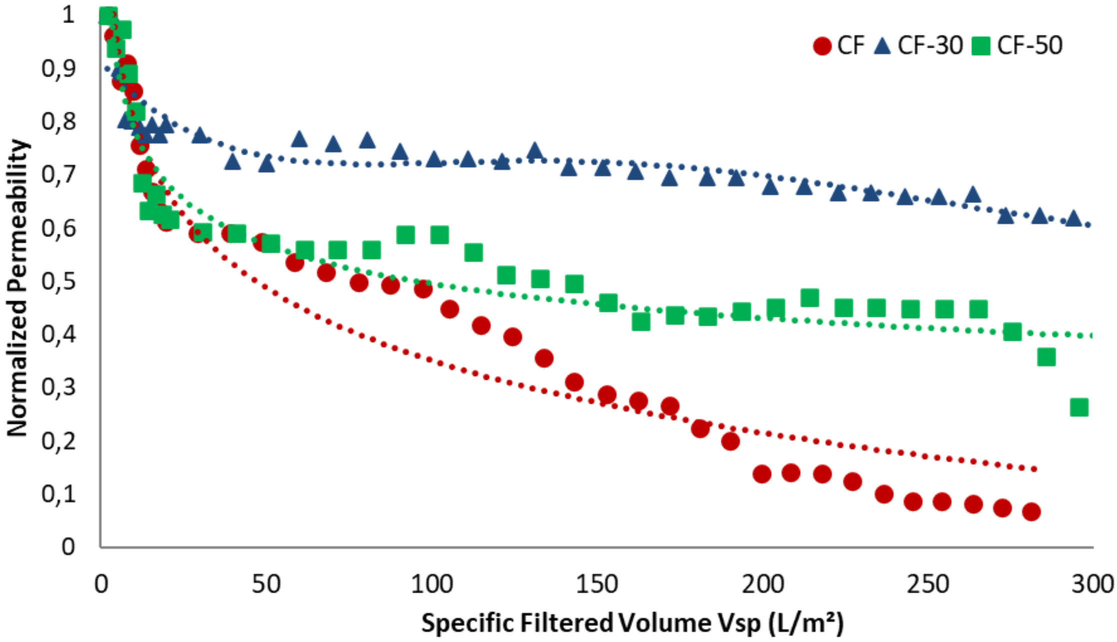



**Figure 3.** Effect Experiment on removal of Turbidity, TDS and Cu

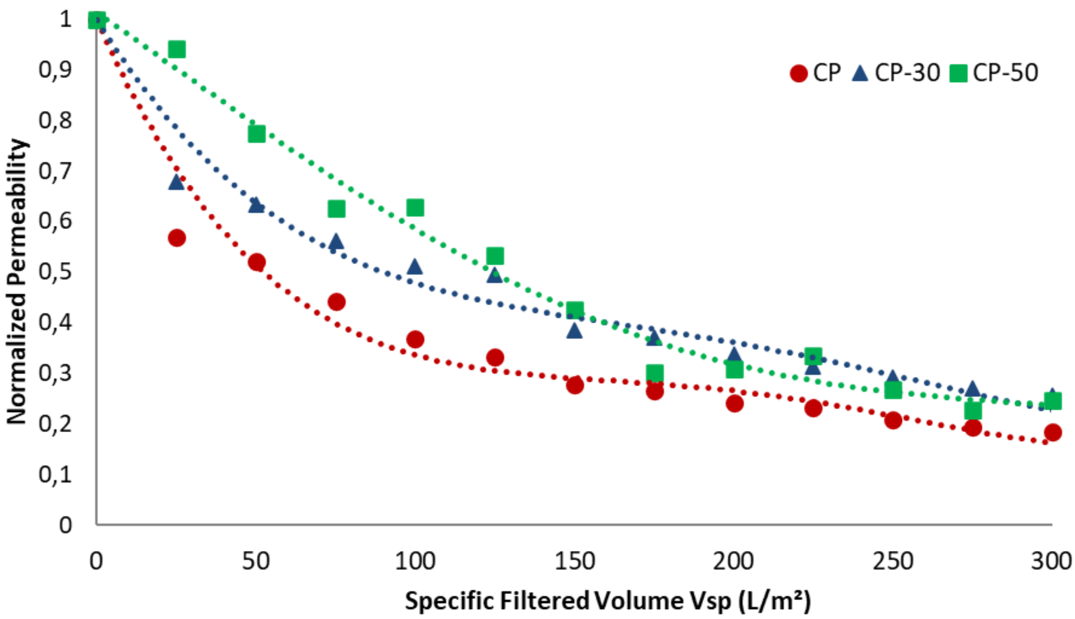
Fouling formed as a result of the experiment can also affect the Cu removal efficiency. The initial pressure difference between CP and CF, which is greater than CF, makes the cake layer more compact and stagnant. Decreased removal by a thick compact fouling layer due to polarisation of cake concentration (Ng & Elimelech, 2004). The compact cake layer interferes with pollutant permeation, thereby increasing the pollutant concentration within the fouling layer. The increase in the cake content increases the pollutants across the membrane and thus lower removal. Miller et al., (2014) showed that increasing TMP can reduce organic rejection due to increased concentration polarisation compared to constant flux.

### 3.3. Performance of combined coagulation

Further analysis was performed on the membrane performance of the direct CFU process. In General, different membrane fouling behaviour was observed in the CP and CF (Figure 4). The comparison between normalised permeability and filter volume specific is shown in Figure 4(a). The results showed a decrease in the normalised permeability of CF, CF-30, and CF-50 by 0,06, 0.61, and 0.24, respectively, when the specific filtered volume reached 300 L/m$^2$. From the results, better membrane performance was observed in case of filtration pretreated with coagulation/flocculation process. The coagulation/flocculation process increased floc size, retaining larger particles on the membrane surface. Not only floc size but also floc characteristics, including structure, have a significant influence on membrane fouling. Floc with loosely structured agglomerates reduces resistance to membrane filtration, whereas flocs that compact into a structured cake layer may increase the retention of ultrafiltration and exacerbate flux reduction (Nan et al., 2015). The CF-50 experiment caused more compact fouling than the CF-30 experiment. The addition of high concentrations of alum will cause more hydrolysis of $Al(OH)_3$ and greater polarisation of the concentration formed due to precipitation of $Al(OH)_3$, so membrane fouling will be severe and the permeate will not increase too much. The permeability of the CF-30 membrane did not substantially decrease, indicating that the resulting flux was greater than that of the CF-50 membrane. An excessive amount of coagulant forms a positive charge, making the cake layer formed more compact due to the positively charged floc sticking to the negatively charged surface of the membrane (Wu et al., 2019). Similar results were also obtained by Wang et al., (2020), aluminium ions could combine with small organics, which causes organic rigidity, resulting in the blockage of membrane pores and hard cake layers due to Al-organic.




(a)




(b)

**Figure 2.** Normalized permeability curve: a) Constant Flux; b) Constant Pressure

Fouling formation is further studied by dividing the phases of the fouling that are formed—normalised permeability to the mathematically specific filtered volume of constant flux. At the beginning of filtration, the membrane permeability decreases rapidly with a steep decline in the membrane filtration curve. When the clean membrane is covered with a thin layer of fouling, which increases markedly with the volume generated, pore blocking contributes to this stage (Sioutopoulos & Karabelas, 2016). During the initial stage of filtration, large particles in water quickly settle on the surface of the membrane and further clog the pores (Garba et al., 2019). Furthermore, the decrease in membrane permeability gradually decreased in all experiments. The cake layer prevented pollutants in the feed water from entering the membrane pores, resulting in a relatively gentle increase in TMP (S. Wu et al., 2023). After fouling, the cake layer covered the membrane with a sufficient layer showing almost constant fouling/cake resistance (Laksono, 2021). Coagulation/flocculation combination reduces membrane fouling. In the coagulation/flocculation process, the particle size that can be enlarged can be retained by the UF to form a concentration polarisation, namely, reversible fouling. At the same time, direct filtration of samples with UF causes adsorption on the membrane pores, resulting in a faster decrease in flux (Bergamasco et al., 2011). The coagulation/flocculation process reduces the formation of membrane pore blockages, and permeability can be increased to produce more filtered volume.

The fouling rate at constant pressure was also studied. A comparison between normalised permeability and volume-specific filter is shown in Figure 4(b). The results showed a decrease in the normalised permeability of CP, CP-30, and CP-50 by 0,18, 0,26, and 0.25, respectively, when the filter volume specific reached 300 L/m2. As can be seen from the graph, the membrane permeabilities of CP-50 and CP-30 are greater than that of CP. In the CP-30 and CP-50 processes, CP-50 increased the membrane permeability to a greater extent than CP-30. However, when the specific filtered volume reached 150 L/$m^2$, the membrane permeability of CP-50 was the same as that of CP-30 and did not differ much from that of CP. After the filter volume reached 150 L/$m^2$, the fouling layer formed by constant pressure filtration was almost identical. At the beginning of filtration, the CP membrane performance exhibited a generally faster decrease in membrane permeability, indicating pore blockage due to the smaller particle size. In the case of CP-30, the membrane permeability decline became less extensive compared with that of CP due to the increase in the size of the floc; it could be retained by the membrane and pore blocking was reduced. Furthermore, the best membrane permeability performance was observed in the early phase of CP-50 filtration. However, during the filtration process in later stages, the membrane permeability declined faster than CP-30 or CP. Initial CP-50 filtration can produce more flux because the initial floc layer is more significant, making the membrane pores remain clean. During the filtration process, the cake layer formed due to compact floc causes a thicker cake layer, making it difficult for water to pass through the membrane due to the presence of a floc layer causing permeability to decrease more rapidly.

Different results were obtained for constant pressure filtration and constant flux. In CP, CP-50 increased membrane permeability better than CP-30 when the volume reached 150 L/m2h, while constant flux filtration was better for CF-30 than for CF-50. Differences in the CF and CP values may occur due to different operating conditions. Sioutopoulos and Parabola (2016) conducted an experiment with constant pressure and constant flux filtration, where the constant pressure decreased the flux faster than the constant flux. This is due to the initial pressure between the constant flux and the constant pressure being different from that of the constant flux having a smaller initial pressure. The pressure difference at the CP caused a faster decrease in membrane permeability compared to CF-30 and CF-50. However, based on the curve line formed for CF filtration, a more significant decrease in membrane permeability was observed than in CP. The experimental results of Miller et al. (2014) show that membrane fouling under constant flux can occur faster than under constant pressure. Constant flux occurs when membrane pores are blocked, and the blockage continues to increase to maintain the same flux in the membrane area.

Once the membrane pores are sufficiently blocked, the cake layer starts to increase. However, the addition of the coagulation-flocculation process reduces pore blockage, so the cake layer formed on CF is better than that formed on CP.

## 4. Conclusion

The coagulation/flocculation effect on UF performance was investigated for copper removal and membrane-fouling reduction using modified surface water. Higher Cu removal was found at filtration under constant flux compared to constant pressure (81% and 66%, respectively. In the case of water treated with coagulation (optimum coagulation of 30 mg/L), the results revealed a higher removal of Cu concentration at constant flux operations compared to constant pressure operations (73% and 89%, respectively. Related to the membrane filtration process, additional coagulation resulted in reduced membrane fouling due to an increase in membrane permeability compared to untreated ultrafiltration.

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