

## Rectification methods for the fouling of ultrafiltration hollow-fibre membranes as a result of excessive soluble iron

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### ABSTRACT

The aim of this study is to analyse process design parameters relating to a membrane bioreactor (MBR) membrane and chemical dosing systems as well as the characteristics of membrane foulant so as to identify a remedy for implementation. A qualitative research undertaken included electron microscopy analysis of the fouled membranes through a membrane autopsy which showed major inorganic deposits to the membrane surface, mainly comprising of iron. As it currently remains, the membrane system utilises citric acid for chemical in-place cleaning of the membranes. Analysis of results identifies the impact of membrane foulant to the operational performance of the treatment works and the impairment to effluent quality discharged to local waterways. Furthermore, an assessment into economic impacts is expected to show significant opportunities for improvements to operating costs as a direct result of cleaner membranes returning transmembrane pressures to normal levels during the permeate production cycle. The study also proposes optimisation strategies of the current cleaning process that is adequate for the level of ferric chloride dosed for phosphorous removal and odour control. Consideration is given to two types of strategies for rectification.

*Keywords:* Flocculation; Fouling; Membrane bioreactor; Ultrafiltration; Wastewater

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### 1. Introduction

Following the successful commissioning and hand-over of the newly constructed sewage treatment plant (STP) in Australia, late 2007, the subsequent operation of this facility had promptly revealed premature degradation in performance of the membrane system as a result of ferric chloride, dosed upstream within the process works, contributing to a remarkable fouling of the ultrafiltration (UF) hollow-fibre membranes.

The subject of this research involves a municipal wastewater treatment process utilising a three stage

Bardenpho biological process with UF hollow-core membrane technology and UV disinfection for tertiary treatment. This process design satisfies the performance criteria required by the regulatory authority (Environment Protection Agency), and minimises land use conflicts with adjoining residential properties by virtue of the small area footprint of the membrane bio-reactor (MBR) process.

In an attempt to quash odour complaints from nearby residents, the operations team had increased ferric chloride dosing within the initial operating year so as to improve precipitation of odour-causing hydrogen sulphides. It is suspected that such a move has contributed to membrane fouling which is a major drawback for membrane

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filtration process occurring gradually and presenting in various forms such as adsorption, pore blockage, deposition or gel formation [1–4].

Ferric chloride dosing required for the effective precipitation of phosphorous as well as hydrogen sulphide odour control is passing through the treatment process resulting in excessive soluble iron content within the membrane tanks. The membranes are proving generally effective in resisting the passing of ferric through to the permeate however the ingress of iron within the fibre of the membrane is proving difficult to remove via the current chemical in-place cleaning system, which involves backwashing with 50% citric acid solution and sodium hypochlorite. The result of the problem is an increase in transmembrane pressure developed in the production of permeate. The subsequent result of this phenomenon is an increase in plant operating costs as well as other process factors as a result of the following: i) the permeate pumps are required to operate at an increased current to permeate through the otherwise congested membrane, ii) an increase in frequency of membrane backwashing and maintenance clean requires increased consumption of electricity and cleaning chemicals — citric acid and sodium hypochlorite, and iii) excessive fouling had resulted in yellow coloured effluent being discharged to the river and compromising operating licence conditions.

Whilst it should be noted that previous research by others have been undertaken within this field of study, it is worthwhile to acknowledge the novelty of this particular undertaking. Readers are directed to a similar study completed in Australia by Wang and Waite [5] where analysis is made into the transformation of iron species in activated sludge MBR. Where the study of Wang and Waite [5] is completed in an artificial lab-scale MBR, the novelty of this research is highlighted in comparison as it involves the assessment of a real life STP where the operations of the STP is adversely affecting local residential communities and ecosystems. This research involved the procurement by hand of live samples of membrane from the fully functional STP, a financial assessment of genuine electrical and chemical consumption at market prices current at the time of compiling this study and the collating of various data from plant monitoring and control systems in order to form a comprehensive assessment where findings were reported to the water authority with life cycle cost analysis and recommendations.

This study primarily aims to focus upon a current process problem within the sewage treatment plant where ferric chloride dosing is proving problematic with accumulation of foulant in the membrane treatment system. This includes the detailing of only the processes which bear an influence upon the problem experienced. This research then proposes possible remedial strategies in order to restore full operating performance.

## 2. Materials and methods

### 2.1. Wastewater characteristics

Table 1 shows the characteristics of raw sewage of the municipality and the quality of final discharge effluent from the plant. Here the loads in the below table are based on a sewage flow contribution of 210 L/equivalent population per day.

### 2.2. Biological treatment followed by UF hollow-fibre membrane processes

Fig. 1 presents the wastewater treatment processes for the sewage treatment plant indicating screening, biological processes, membrane bioreactor process and chemical dosing locations. The STP utilised a UF hollow-fibre (0.04 µm pore size) membrane treatment system located within a tank of 3.3 m (w) × 3.3 m (h) × 3.25 m (d) providing an average dry weather flow (ADWF) volume of 30.5 kL and an abnormal peak dry weather flow (APDWF) volume of 33.3 kL. The STP consisted of two parallel trains of UF membranes. Each train was sized for an average flow of 4 L/s and a maximum flow of 10.2 L/s. The bioreactor level will dictate how many ultrafiltration membranes trains will be in operation. If the bioreactor level is high both trains are in operation at maximum design flux rates. If the bioreactor level is mid-low one train is in operation at standard flux rates, with the other train switched off and in standby. If the bioreactor level is low both trains are switched off and in standby. As both bioreactor trains are connected to a

Table 1  
Characteristics of raw sewage inflow and effluent discharged from the sewage treatment plant

Inflow raw sewage parameter	Concentration (mg/L)	Load (kg/d)
BOD <sub>5</sub>	285	92
SS	310	100
COD	570	185
TKN	62	20
TP	10	3
Outflow effluent parameter	Operating licence 90‰	Operating licence 50‰
BOD <sub>5</sub>	10	5
SS	10	5
COD	—	—
TKN	11	7
TP	0.3	0.15
pH	6.5–8.5	6.5–8.5
Faecal coliforms (coliform units per 100 mL)	150	30

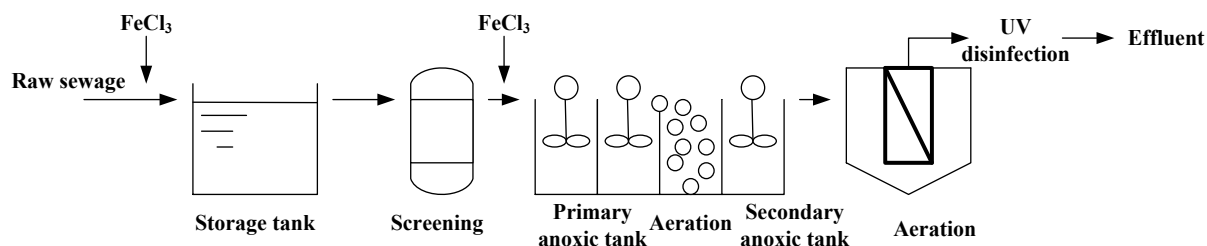


Fig. 1. Sewage treatment plant process diagram indicating membrane bioreactor process and chemical dosing locations.

common feed system, the water level reads approximately the same in each tank.

Mixed liquor was fed to the membrane tanks at 3–6 times the treated water flow rate. Low pressure air for coarse air scouring of the membranes is provided by duty/standby rotary scroll blowers at a rate of 257 L/s at a pressure of 35 kPa. The mixed liquor which was not treated by the membrane filters overflowed and returned to the biological nutrient removal process. The membrane plant also included common chemical dosing (sodium hypochlorite and citric acid), compressed air (duty/standby) and membrane air scour (duty/standby) equipment.

Ferric chloride ( $\text{FeCl}_3$ ) dosing was utilised within the STP process for phosphorus removal by chemical flocculation and odour control by precipitation of sulphides within the odour causing  $\text{H}_2\text{S}$  compound. The amount of dosing was based upon the characteristics of the raw sewage that is common to the local community.

In addition to the normal filtration cycle, regular chemical cleaning in place (CIP) were used to maintain the performance of the membrane and to extend the time between recovery cleanings. Maintenance cleans took approximately 120 min to complete and were scheduled automatically at a frequency of approximately three times a week. Maintenance cleaning involves contacting the membrane fibres in air with sodium hypochlorite at 250 mg/L (Hypo MC), or citric acid at 2000 mg/L (Acid MC). Either process is scheduled through the plant SCADA where the operator is able to select the frequency and approximate time-of-day for each clean to take place. Only one train at a time may be selected for an MC. The PLC then automatically performs maintenance cleans on each train based on the availability of CIP resources and plant demand.

Recovery cleans (RC) were used when a more rigorous cleaning was required and are typically initiated when a train reaches a maximum transmembrane pressure (TMP) in the range  $-45$  to  $-69$  kPa. Recovery cleans are automated however it may also be initiated by the Operator from the plant SCADA. This cleaning process consists of a series of backpulse/relaxation cycles with 2,000 mg/L sodium hypochlorite (Hypo RC) or 8,000 mg/L citric acid (Acid RC) followed by soaking in the cleaning solution for a further 4–12 h at 1000 mg/L sodium hypochlorite

or 4,000 mg/L citric acid. Both the soak time and chemical concentrations are higher in a recovery clean than in maintenance clean.

During the production cycle, permeate was produced by the train and sent to the disposal system which included UV disinfection then pumping to discharge outfall into local waterways. There was aeration during the production cycles to prevent solids accumulating on the surface of the fibres and in the membrane pores. This prevented increases in trans-membrane pressure (TMP). The membranes are also back pulsed every 8–15 min with aeration to reduce TMP and maintain production flux.

### 2.3. Membrane fouling characteristics

Following continual efforts to adjust cleaning parameters and its failure to substantially clear the membranes, an autopsy of the fouled membranes was undertaken so as to profile the precise characteristics of the foulant [6,7]. Multiple strands of membranes, collected in airtight Ziploc bags obtained from both membrane train 1 and 2 and preserved in aqueous environment so as to minimise and dehydration of the foulant. Based on optical evaluations, three samples were selected for further dissection and analysis. Analytical techniques were used to determine the characteristics of membrane foulant present on or within the membrane fibre. Standard autopsy investigation involved the dissection of a fouled membrane element taken from the STP. A sample of foulant is then obtained from a known quantity of hollow fibres for further physico-chemical analysis.

The clean and fouled membrane surfaces were analysed using scanning electron microscope/energy dispersive using X-Ray (SEM/EDX, Rigaku, Japan). It has been a useful tool for investigating membrane structure during membrane fouling. SEM images of the UF hollow-fibre membrane were carried out using the scanning electron microscopy (FE-SEM, S-4700, Hitachi, Corp., Japan). The voltage was 5 kV and the working distance was 12 mm. The magnification was 20,000 times. SEM where surfaces were coated in a thin layer of iridium were studied in terms of morphology and EDX was used to identify organic and inorganic elements in foulant composition.

### 3. Results and discussion

#### 3.1. Membrane characterisation

##### 3.1.1. SEM/EDX

Fig. 2 shows the SEM images of fouled UF hollow-fibre membranes taken at varying magnification. The membrane experienced a severe fouling when wastewater after biological processes was filtered directly through the membrane. Pore blockage seems to be the major contributing type of foulant at the STP. All samples tested were covered with a thick layer of organic and inorganic fouling layer. Some samples show low or no degree of porosity in membrane fibres.

Fig. 3 shows the EDX results with elemental compounds that form the ingredients of the membrane foulant. The inserted images show that the sample was completely covered by a thick fouling layer. The fouling layer is a mixture of organic and inorganic matter and was developed with little or no degree of porosity. Dwelling bacteria were also observed at several locations and the original skin. EDX results showed that various elemental compounds originated from organic and inorganic foulants were found. Fe, P, Ca and Si were the major components on membrane foulants. Other elemental foulants were recorded such as Mg, S, Mn and Cl at low

weight percentages. Especially, the majority of elements with Fe and P were detected. This may be due to the overdose or underdose of ferric chloride, which leads to no optimal concentration and results in non-settable aggregated flocs. The P compound may be due to complexed colloidal Fe compounds which may not be settled down. The presence of Fe in the bore samples indicates severe iron contamination and lack of cleaning.

#### 3.2. Mass balance of ferric chloride dosing

Ferric chloride ( $\text{FeCl}_3$ ) dosing is utilised within the STP process for odour control by precipitation of sulphides within the odour causing  $\text{H}_2\text{S}$  compound and phosphorus removal by chemical flocculation. The amount of dosing is based upon the characteristics of the raw sewage that is common to the local community.

Table 2 shows the designed and actual balances of  $\text{FeCl}_3$  dose at the first dosing point for the removal of  $\text{H}_2\text{S}$ . The chemical reaction between  $\text{FeCl}_3$  and  $\text{H}_2\text{S}$  is as follows:



The sulphur reduces iron(III) salts to iron(II) salts. The addition of ferric chloride to wastewater results in the reaction of ferric chloride with hydrogen sulphide to

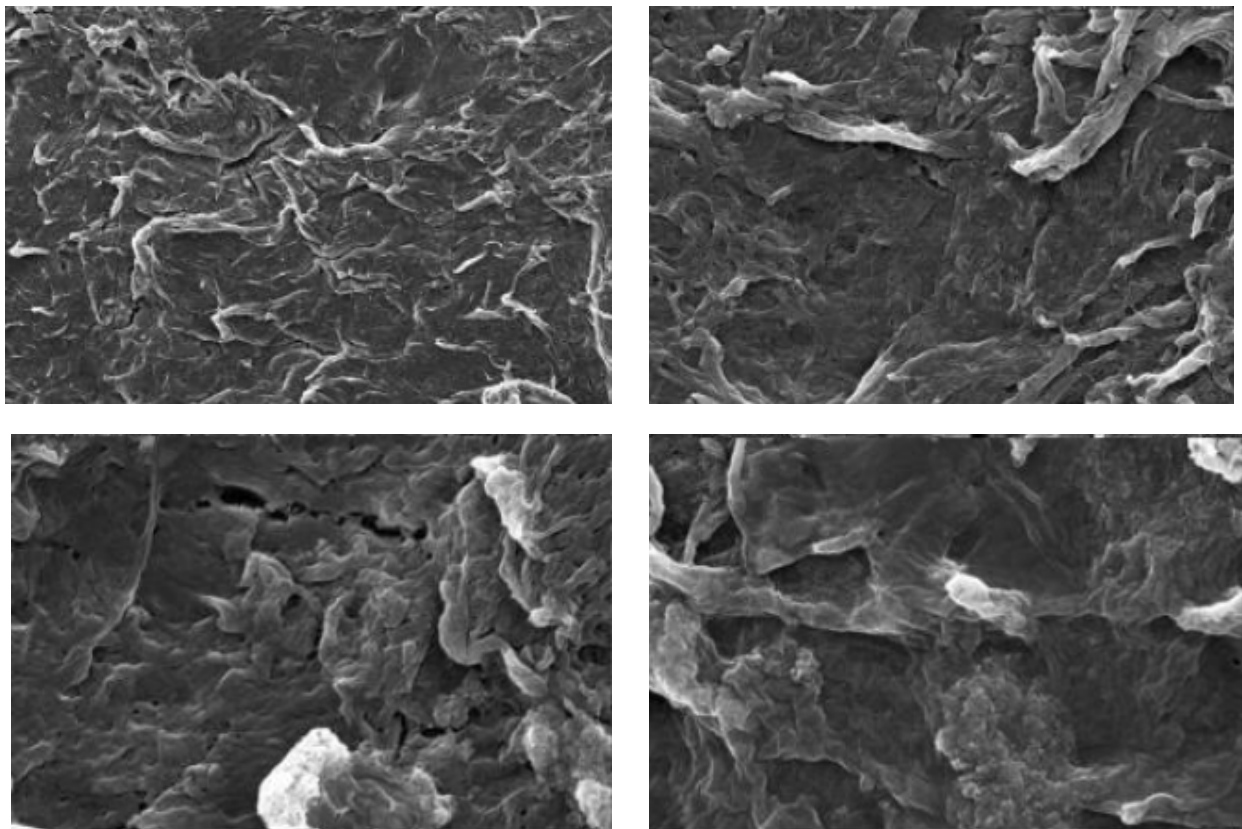
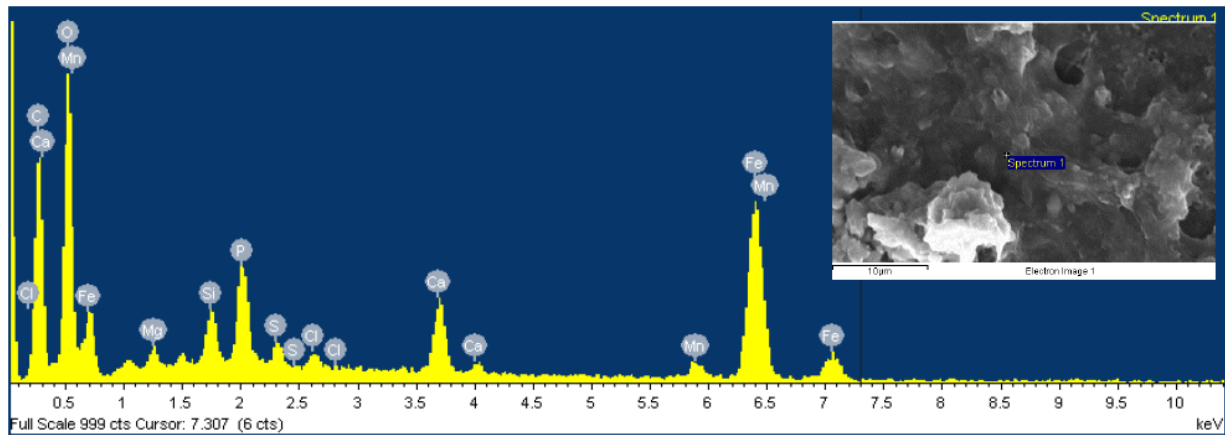


Fig. 2. SEM images of the membrane specimen taken at varying magnification. Magnification levels: 1000× (top left); 5000× (top right); 10000× (bottom left); 50000× (bottom right).



Element	Weight (%)	Element	Weight (%)
Mg	3.64	Cl	1.86
Si	6.04	Ca	9.44
P	12.19	Mn	3.92
S	2.95	Fe	59.95

Fig. 3. EDX results with compositions of elemental compounds that form the ingredients of the membrane foulant.

Table 2  
Designed and actual balances of FeCl<sub>3</sub> dose at the first dosing point

Designed factors	Balance
Average influent flow, ML/d	0.61 (plant design)
Peak influent flow, ML/d	2.59 (plant design)
Influent H <sub>2</sub> S, mg/L	9.0 (from design data)
Total average H <sub>2</sub> S inflow/d, kg/d	5.49
FeCl <sub>3</sub> concentration, mg FeCl <sub>3</sub> /mg solution	0.42
Required Fe dose, mmol/L	0.48
Design dose rate of FeCl <sub>3</sub> , kg/h	1.96 (for odour removal)
Actual factors	Balance
Total FeCl <sub>3</sub> consumed, L/d	36 (from plant SCADA*)
Current inflow, ML/d	0.275
Calculated FeCl <sub>3</sub> consumption, L/d	79.85 (per inflow of 0.61 ML/d)
Actual dose rate of FeCl <sub>3</sub> , kg/h	4.42 (for odour removal)

Table 3  
Designed and actual balances of FeCl<sub>3</sub> dose at the second dosing point

Designed factors	Balance
Average influent flow, ML/d	0.61 (plant design)
Peak influent flow, ML/d	2.59 (plant design)
Influent phosphorus, mgP/L	12.7 (from design data)
Mass of influent P average, kgP/d	5.80
Target effluent P concentration, mgP/L	0.10 (operating licence)
Required Fe dose, mmol/L	0.48
FeCl <sub>3</sub> solution concentration, mg FeCl <sub>3</sub> /mg solution	0.42
Design dose rate of FeCl <sub>3</sub> , kg/h	1.54 (for P removal)
Actual factors	Balance
Bioreactor 2 flow setpoint, L/h	1.43 (from SCADA* parameters)
Actual dose rate of FeCl <sub>3</sub> , kg/h	2.07 (for P removal)

\*Supervisory control and data acquisition

form an insoluble precipitate that is black in colour. The designed dose of FeCl<sub>3</sub> was 1.96 kg/h, whereas the actual dose rate of FeCl<sub>3</sub> was 4.42 kg/h. It should be noted that FeCl<sub>3</sub> can be lost through screenings 44% (from design data) and the total FeCl<sub>3</sub> is transferred to anoxic tank

(2.49 kg/h). The percentage increase of FeCl<sub>3</sub> is 126% compared to design dose rate. However, the actual dose rate is higher than the designed requirement.

Table 3 shows the designed and actual balances of FeCl<sub>3</sub> dose at the second dosing point for the removal of phosphorous. The second dosing point for FeCl<sub>3</sub> is at the entrance to the bioreactor primary anoxic tanks and serves the purpose of chemical flocculation for the

Table 4  
Financial impact assessment of permeate pumps with/without fouled membrane

Parameters	Consumption
Permeate pumps, kW	5.5 (9.2 A full load current motor, 0.85 power factor, 2 pumps running at any one time 24 h)
Total run time/y, h	8,760
Permeate pumps motor performance during commissioning	
Operating current drawn, A	4.7
Motor Watts drawn, kW/h	$\sqrt{3} \times 415V \times 4.7A \times 0.85\Phi = 2.872$
Total cost operating 2 pumps/y	$2 \times 2.872 \times 8760 \times 0.1724 = \text{AUD}\$8,674.73$
Permeate pumps motor performance with fouled membranes	
Operating current drawn, A	7.1
Motor Watts drawn, kW/h	$\sqrt{3} \times 415V \times 7.1A \times 0.85\Phi = 4.338$
Total cost operating 2 pumps/y	$2 \times 4.338 \times 8760 \times 0.1724 = \text{AUD}\$13,102.60$
Calculated operating cost increase	$\text{AUD}\$13,102.60 - \text{AUD}\$8,674.73 = \text{AUD}\$4,427.87/y$

Note: calculated value does not include costs associated with increased frequency of backwashing

phosphorous removal. The following is an assessment of the amount of iron required at dosing location 2 for flocculation of phosphorous:



It should be noted that the percentage increase of  $\text{FeCl}_3$  was 34% compared to design dose rate. The maximum  $\text{FeCl}_3$  allowable was 2.39 kg/h (design allowable at membranes). The total  $\text{FeCl}_3$  arriving at membranes was 4.56 kg/h combined with residual ferric. The total  $\text{FeCl}_3$  overdose at membranes was 91%.

### 3.3. Financial impact assessment

The effect of membrane foulant at the STP impacts on the economies of two systems: i) electrical consumption by mechanical equipment: this is a result of electrical drive operating at greater current and processes occurring at an increased frequency and ii) chemical costs resulting from excessive consumption of chemicals. An assessment of the costs associated with the two economies listed above is shown in Tables 4 and 5. The combined cost of chemical and electrical was AUD\$17,183.57 per annum (AUD\$12,755.70 + AUD\$4,427.87). This cost impact per household (225 properties) is AUD\$76.37 per property per annum. This significant amount of cost can be reduced by a rectification method.

### 3.4. Rectification method

A prospective method for the rectification of the membrane foulant caused by  $\text{FeCl}_3$  complexes is considered by implementing a selection of the following possible remedies: i) substituting the current citric acid agent with potassium permanganate ( $\text{KMnO}_4$ ) and ii) modification of the membrane surface by application of argon plasma.

Table 5  
Excessive chemical consumption costs

Parameters	Consumption
Excess dose rate of $\text{FeCl}_3$ , kg/h	2.99
Hours in year	8,760
Total excess $\text{FeCl}_3$ dosed in 1 y, kg	26,192
Cost of bulk $\text{FeCl}_3$	AUD\$487.00/tonne
Total cost of excess $\text{FeCl}_3$	AUD\$12,755.70 per annum

Table 6  
Cost of  $\text{KMnO}_4$  dosing for membrane cleaning

Parameters	Consumption
Residual $\text{FeCl}_3$ at membrane tanks, kg/h	2.99
Residual Fe at membrane tanks, kg/h	1.03
Residual Fe at membrane tanks, kg/d	24.71
Total plant influent, ML/d	0.61
Fe influent to membrane rate, g Fe/L	0.041
Amount of $\text{KMnO}_4$ required for Fe removal	23.23 kg $\text{KMnO}_4$ /d (AUD\$8,478.95 kg/y)
Cost of $\text{KMnO}_4$ dosing	AUD\$44,763.85 per annum

### 3.5. Potassium permanganate

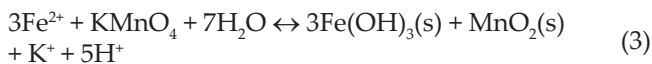
Potassium permanganate ( $\text{KMnO}_4$ ) is a highly effective cleaning agent that is commercially available and is

relatively safe to handle. As such, an investigation has been conducted into the suitability of this agent within the defined problem at the sewage treatment plant. Potassium permanganate is only available in dry form and is found to be highly reactive under conditions typically within the wastewater industry as it will oxidise a wide variety of inorganic and organic substances [8]. In order to utilise potassium permanganate, a concentrated  $\text{KMnO}_4$  solution is generated on-site by mixing with water at typically 1–4% concentration to produce a pink coloured solution ready for dosing into wastewater treatment applications. Solubility of  $\text{KMnO}_4$  is found to be 6.5 g/mL at 20°C.

Table 6 shows the cost of  $\text{KMnO}_4$  dosing for membrane cleaning.  $\text{KMnO}_4$  is currently distributed by Orica Chemicals and is sold in bulk dry container at a cost of AUD\$5.28/kg (2010 list price) per 25 kg drum. Permanganate will oxidise iron to convert ferrous (2+) iron into the ferric (3+) state. The oxidised forms will precipitate as ferric hydroxide. The precise chemical composition of the precipitate will depend on the nature of the water, temperature, and pH.

In considering potassium permanganate as a viable cleaning agent, care should be taken to prevent overdosing in which case, potassium permanganate may pass through filtration. It is important to monitor and maintain correct dosing to ensure all of the  $\text{KMnO}_4$  is reduced to  $\text{MnO}_2$  solids and hence removed by filtration and consequently treated through the sludge dewatering system. If excessive  $\text{KMnO}_4$  is passed through membranes the result is reduction to  $\text{MnO}_2$  solids downstream of membranes where the reduced solids may cause a black discoloration of the discharged effluent and any reuse of the effluent, as is currently practised onsite, may cause the solids to precipitate on heat exchange surfaces such as hot water heaters. Consequently, operating licence conditions may be breached by excessive discharge of manganese. In general, potassium permanganate does not influence other treatment processes or plant conditions.

The typical chemical reaction equation for iron and permanganate is as follows:



This reaction shows that alkalinity is consumed through acid production at the rate of 1.49 mg/L as  $\text{CaCO}_3$  per mg/L of  $\text{Fe}^{2+}$ . The potassium permanganate dose required for oxidation is 0.94 mg/mg Fe. The oxidation time ranges from 5 to 10 min, provided that the pH is over 7.0 [9].

An assessment into the possibility of Mn fouling on the UF membrane as a result of  $\text{KMnO}_4$  dosing does not form part of this study however a research study undertaken in Korea by Choo et al. [10] showed a remarkable manganese removal performance by dosing chlorine at a rate of 3 mg/L. Such a method would be compatible within the context of this municipal wastewater treatment plant as

its readily available liquid form, inexpensive costs and already established onsite chemical handling and dosing systems means that chlorine dosing may be experimented should  $\text{KMnO}_4$  dosing lead to Mn membrane fouling.

### 3.6. Membrane surface modifications

It has been identified in previous research undertaken globally, that membrane surface modifications provide a significant improvement for membranes to maintain good flux performance and product longevity. In specific applications to hollow core UF membranes, a recent study undertaken in a Chinese research group [11] explored the application of argon (Ar) plasma as an attempt to modify surface characteristics. It should be noted however that the application of this surface modification was not implemented as part of this study.

In the study undertaken, results show that 1 min of argon plasma treatment would result in etching of the membrane surface which found to decrease pore diameter and membrane porosity whilst increasing surface roughness. This contributed to an improvement to the hydrophilicity, biocompatibility and adhesion of membrane surface. The plasma application not only generated modifications during the plasma exposure but also retained energised site on the membrane surface which were subject to post-reaction. More specifically, results showed that 1 min treatment of membrane surface with Ar plasma improved flux recovery after water cleaning by 20% and flux ratio after fouling was boosted 143%, in comparison to unmodified membrane samples within a membrane bioreactor application.

This technique shows that the antifouling characteristic of hollow fibre micro porous membranes is greatly improved. This process may be investigated for its suitability on the existing UF membranes and the costs associated with the undertaking of such modifications as current information commonly available for public viewing is limited and fails to explore undesired impacts. Should such a study prove favourable then such modifications may provide a further degree of anti-fouling improvement.

## 4. Conclusions

It is recommended that i) current dosing levels of  $\text{FeCl}_3$  are excessive at both dosing locations 1 and 2. It is an imperative ingredient of rectification to return levels down to design specifications of 1.96 kg/h for odour control precipitation of sulphides and 2.08 kg/h for phosphorous removal by way of chemical flocculation. Process design indicates the need for less ferric chloride dosed for a greater influent flow. Currently as it remains, the sewage treatment plant is dosing greater ferric chloride for less influent and phosphorous/hydrogen sulphide loading, ii)

potassium permanganate can be implemented as a suitable additive to the current cleaning agent citric acid. This should ensure full oxidation of inorganic foulants such as Fe whilst proving to be an easily sludge-digested process if dosed in correct amounts and iii) we may also consider modern membrane treatment technologies to be applied to the membranes in order to reduce the likelihood of membrane fouling – such as surface modification by argon plasma treatment

In recommending the establishment of a new chemical cleaning agent, consideration is given to the capital investment required for the erection of an appropriate facility, which would include dosing pumps, dry potassium permanganate hopper and batching tank to develop dosing solution. A similar chemical dosing skid already installed within the plants chemical dosing facility for the dosing of acetic acid would be a similar representation of equipment necessary and at a cost of AUD\$65,000.00. Based on this amount, the economy of implementing potassium permanganate over a ten year period is as follows: i) capital Investment and Construction: AUD\$65,000.00, ii) annual  $\text{KMnO}_4$  dosing (year): AUD\$44,763.85 per annum, iii) total cost over 10 year period: AUD\$512,638.50, iii) less cost currently exhausted on  $\text{FeCl}_3$ : AUD\$17,183.00 per annum, iv) cost for  $\text{KMnO}_4$  dosing per year: AUD\$171,830.00 and v) net cost for implementation: AUD\$340,807.50.

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