

Enhanced Water Quality Monitoring for Produced Water Disposal using Cross-flow Membrane Filtration

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Abstract:

The accuracy of oil-in-water analysis for produced water is increasingly crucial as the regulations for disposal of this water are getting more stringent world –wide. According to OSPAR commission, the main component of oil and grease that are being monitored in produced water is the dispersed oil. Therefore, membrane filtration can be incorporated as part of water quality monitoring for the disposal of produced water by separating dispersed and dissolved oil so that the accuracy of measuring the dispersed oil can be enhanced. In this paper, Microfiltration and Ultrafiltration with different material and pore sizes are run at different operating pressures to investigate its effectiveness in maximising the dissolved oil in the permeate. After membrane filtration treatment, samples are analyzed with Gas Chromatography Mass Spectrophotometer (GCMS) to determine the oil content. The concentration of feed, retentate, and permeate obtained from GCMS determines the degree of dispersed and dissolved oil separation. From the study, with comparison to ultrafiltration, microfiltration of pore size 0.2µm Hydrosart® membrane can achieve a more efficient separation of dissolved and dispersed oil in permeate and retentate respectively.

Keywords: Wastewater management, Water Quality monitoring, Cross-flow filtration, Produced Water

1. Introduction:

The wastewater from oil mining industry, or produced water, consists of many constituents. Most of the constituents in the produced water are treated by ordinary mechanical treatment process except residual oils (Mondal & Wickramasinghe, 2008). The oil-in-water content is the main indicator for residual oil monitoring in produced water by operators and regulatory authorities before its disposal into the sea. The oil content can be classified into two categories: the dissolved oil and dispersed oil (Ahmadun *et.al.* 2009). The organic components in the dissolved oil can be divided into four main groups, namely aliphatic, aromatic, polar and fatty acid compounds. The relative quantities and molecular weight distribution of these components vary significantly from field to field. Low molecular weight aromatic components such as BTEX (benzene, toluene, ethylenebenzene and isomers of xylene), phenols and aliphatic hydrocarbons, carboxylic acid formed the bulk of measurable dissolved oils, are soluble in water due to their polarity. It was documented that dissolved oil leakage into the groundwater or soil will cause acute/lethal effects on health. The acute toxicity effect can be measured by LC₅₀ tests which states that the concentration where 50% of the samples are killed during the test period in 8 hours (Gbadebo *et. al.*, 2009). However, according to a study by Singer and colleagues (2001) the judgement of whether dispersed or dissolved oil is more toxic is dependent on both the oil species and oil weathering state. They concluded that weathering reduces the amount water soluble oil compounds to the point where dissolved oil elicited less than 50% effect even at unrealistically high loading rates. Therefore, acute effects of disposal of dissolved oil into the deep sea or reinjection into the oil-wells are generally negligible and relatively safe as these places are far away from land and under the sea bed and susceptible to weathering.

Besides that, aerobic and anaerobic biodegradation by microorganism takes place by changing dissolved oil from harmful organic compounds to CO₂ and H₂O. On the other hand, dispersed oil is very non-polar and hydrophobic. It does not dissolve in water and normally exists in long chains with high molecular weight. In water, it floats on top and forms a layer preventing diffusion of air into the water. This effect causes acute asphyxiation to oceanic creatures (Veil *et. al.* 2004). Therefore, the main oil component concerned by the regulatory bodies in the oil-in-water analysis is the dispersed oil content.

The standard analytical method for oil-in-water measurement has been changed from Infra-red (IR) analysis to Gas Chromatography Flame Ionisation Detector (GC-FID) analysis due to solvent restrictions to measure the dispersed oil contents in produced water by the Oslo-Paris (OSPAR) convention - signed by nations in the North Sea (Yang, 2008). However, the drawback of this GC-FID method is that without proper integration on the spectrum, it will result in measuring both the dispersed and dissolved oil concentration. As a result, the reading can be higher than the actual dispersed oil content in the analyzed produced water. This monitoring can lead to the

violation of the limit as set by the regulatory bodies. Improvement on the analysis method would be of benefits to many operators of oil exploration.

Current treatment technologies such as hydrocyclones, and gas floatation are not able to separate the soluble oil in produced water (Arnold & Stewart, 1999). Many researches for the development of inexpensive techniques for the removal of BTEX and other dissolved oil have been on-going for this reason (Ahmadun, 2009) Success have been achieved in the 90s using combined technique of clarification and reversed osmosis filtration in the removal of dissolved oil (Tao, 1993). Ultrafiltration has also been demonstrated in the removal of certain dissolved oil (Santos & Wiesner, 1997). However, there are no reports on the amount of dispersed oil acquired during filtration while removing dissolved oil.

If a cross-flow membrane (ultra/micro) filtration can be embedded as the pre-processing stage in the OSPAR (Oslo-Paris Commission) method, it is expected that the dissolved oil will be contained mainly in permeate while the retentate will contain mainly the dispersed oil. The retentate is then used for the oils-in-water analysis using the OSPAR GC-FID method. This improved produced water analysis would therefore have a more accurate measurement for monitoring purposes. Figure 1 shows the application of membrane filtration incorporated into the GC-FID test method. As can be seen from Figure 2, by incorporating membrane filtration into the test method, the colour of water are clearer.

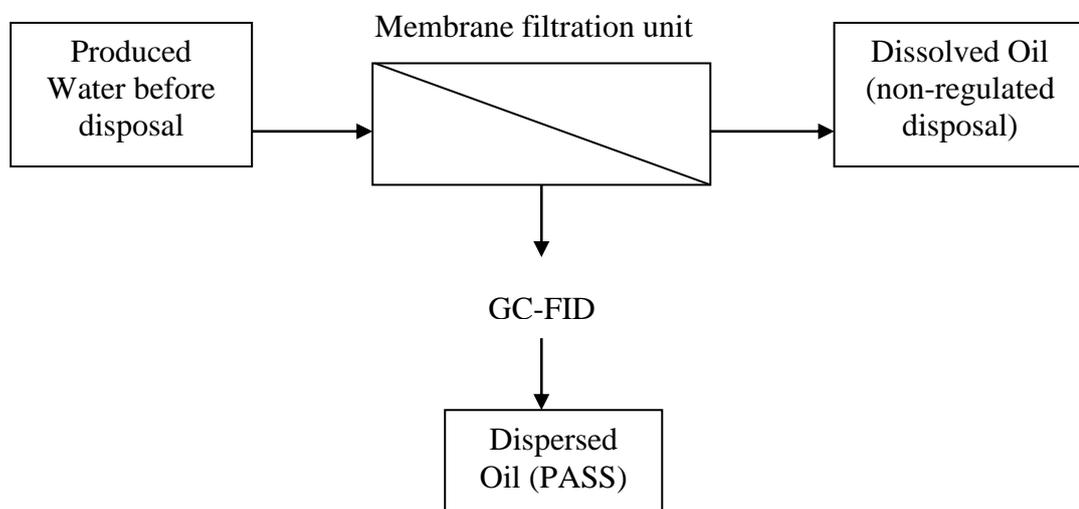


Fig. 1: Integrated oil-in-water analysis process



Fig. 2: Produced water before membrane filtration (left) compared with produced water after membrane filtration (right).

This paper serves to discuss the feasibility of incorporating membrane filtration as part of oil-in-water analysis to separate dispersed and dissolved oil so that the accuracy of measuring the dispersed oil concentration can be enhanced. As mentioned earlier, microfiltration (MF) and some ultrafiltration (UF) can both be potential tools to separate dissolved oil while maintaining dispersed oil in the produced water due to their pore sizes. Therefore besides the feasibility check, this paper also discussed about the efficiency of the separation by exploring different types and sizes of membrane with different operating pressures. To achieve an effective separation, two objectives that need to be addressed are: maximizing the amount of dissolved oil in permeate and maximizing the amount of dispersed oil in retentate. Both the amount of dissolved oil and dispersed oil content are measured by their concentrations using Gas Chromatography with Mass Spectrophotometer detector (GCMS). The efficiency of separation and the interactions among the factors for these objectives are then statistically analyzed.

2. Materials and methods

2.1. Calculation for the Efficiency of Dissolved and Dispersed Oil Separation

As mentioned, there are two types of oil in the produced water namely: dispersed and dissolved oil. The objective is to achieve 100% dissolved oil in permeate; and 100% dispersed oil in retentate. However, some quantity of oil is likely to remain attached or adsorbed on the membrane surface, resulting in unaccounted losses (*error*). In this study, the efficiencies of MF and UF for the separations are being compared. The oil contents are identified and measured by using gas chromatography and mass spectrophotometer detector (GCMS).

The efficiency in the separation of oils is calculated based on percentage as shown in Figure 3. The total dispersed oil concentration from the feed is denoted as 100% dispersed oil and the rest of the concentration will be the dissolved oil concentration which is also 100% dissolved oil. Dispersed oil is assumed not to pass through the membrane therefore it will only be found in the retentate. Therefore two objective functions need to be met for the study of efficiency of the separation: maximizing $z\%$ and maximizing $y\%$. To meet both the objective functions 1 and 2, the error, which is the deposition on the membrane surface, must be as low as possible. Accordingly, qualitative and quantitative GCMS analyses were repeated with retentate and permeate after the filtration experiments.

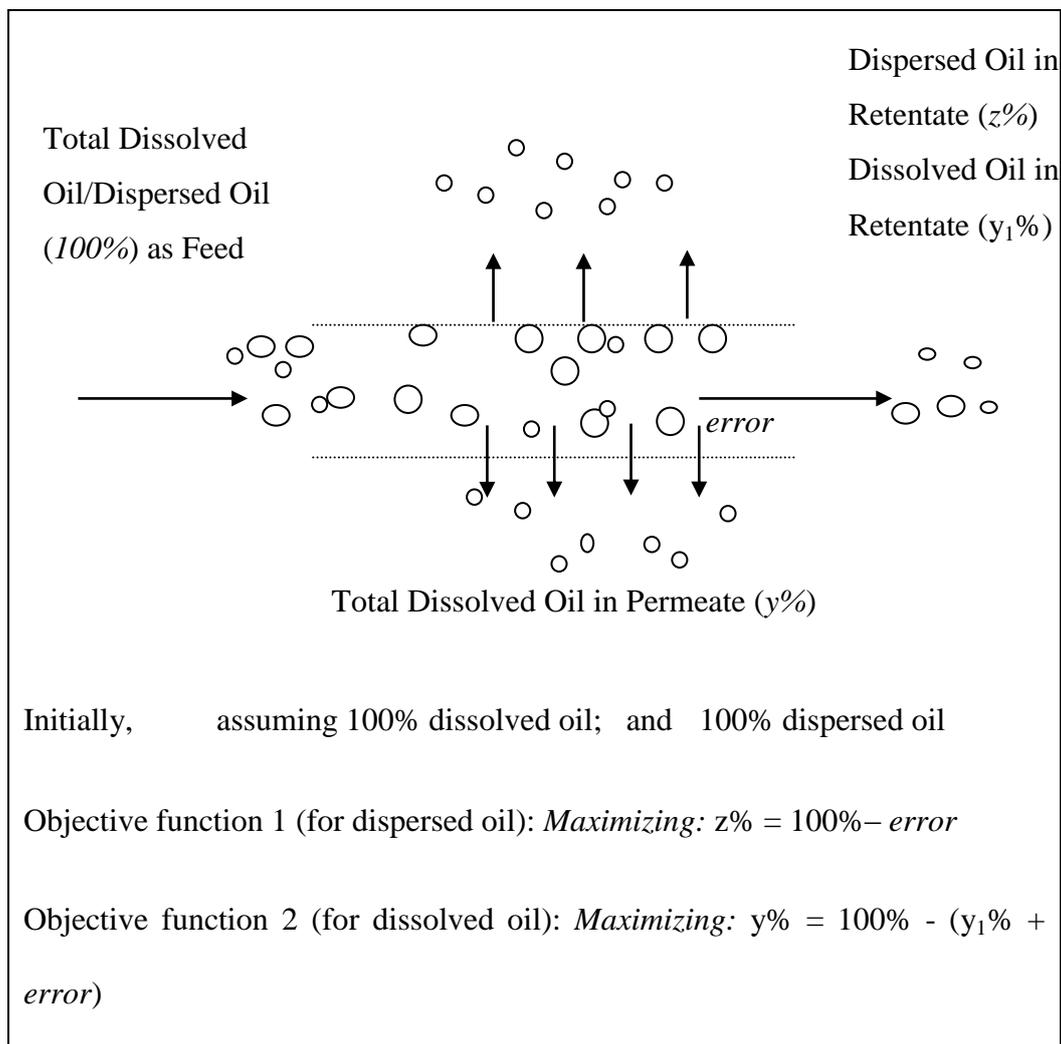


Fig. 3: Definition of efficiency in the separation of dispersed and dissolved oil

2.2. Determination of Optimum Trans-membrane Pressure (TMP) for Membrane Filtration

Concentration polarization is the cause for flux reduction and therefore there is a need to increase effective trans-membrane pressure (TMP) to maintain a constant permeates flux (Choi *et. al.*, 2005). A high TMP is required due to the increasing extent of fouling on membrane surface over time where the optimum TMP is dependent on the cycling time of filtration process as well. As the TMP is governed by Eq. 1, that is:

$$TMP = \frac{(P_F + P_R)}{2} - P_P \quad (1)$$

The filtration system was operated in a fully recycle mode where both retentate and permeate channels were recycled back to the feed beaker containing the sample solution of produced water in order to determine the optimum TMP (Harrison *et. al.*, 2002)

The optimum TMP is being determined by measuring the flux ranging from 0.35 to 3.25 bar, corresponding to four differential pressures (DP). The differential pressure is being governed by the equation (2):

$$DP = P_F - P_R \quad (2)$$

The feed pressure (P_F) was manipulated through the pump power while the retentate pressure (P_R) was set by adjusting the control valve on retentate line as shown in Figure 4.

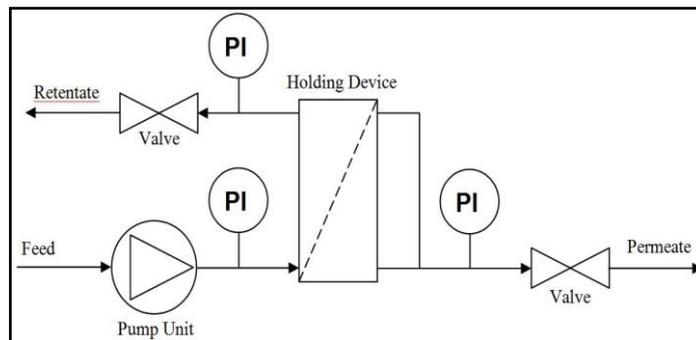


Fig. 4: A schematic diagram of the filtration unit

The system was left to run for a couple of minutes until the pointer inside the pressure gauges stabilized, or steady state was achieved. The time taken to collect about 200 ml of permeate flow was measured and recorded. For consistency, the readings were taken at least three times. As a result, one could calculate the permeate flow rate and ultimately the permeate flux (J) under

different values of TMP. To estimate the optimum values of TMP, the permeate flux was plotted against increasing TMP at different DP.

2.3. Membranes

All the membranes used in this research are made by Sartorius AG. Microfiltration membrane with pore size 0.2 μm and ultrafiltrations membranes with pore sizes 0.01 μm (50kDa) and 0.02 μm (100kDa) are used in experiments. The type of membrane material was added as another function to be studied. Two types of material was used, Polyethersulfone (PESU) and Hydrosart®. The Hydrosart® membrane is a patented product with similar properties as cellulose-based membrane (Sartorius, 2006). According to the manufacturer, it can give good separation and prevent fouling.

2.4. Produced Water Sample

A produced water of 100 ppm of oil-in-water was used in all the experiments as feedstock. The synthetic produced water was mixed by adding 0.5g of crude oil with 5000ml of distilled water. The mixture was mixed thoroughly to form a uniform dispersed and dissolved oil mixture. Their oil contents are identified and measured by using GCMS analytical method.

2.5. Dissolved and Dispersed Oil Identification and Separation by Pore Sizes

Calibration curves comprise of Eicosane, Ethyl-benzene and Xylene which of these are used as the standard reference components representing dispersed (Eicosane) and dissolved oil (both Ethyl-benzene and Xylene) contents, were constructed to quantify the unknown concentrations of respective components in feed, retentate, and permeate. The calibration was done by using Mass Selective Detector (MSD) software. An example of the spectrum of 200 ppm Eicosane standard is shown in Figure 5. Each of these 200 ppm standard solutions consists of Eicosane, Ethyl-benzene and Xylene is also verified with MSD library. Filtration process was performed at DP 0.5 bar with TMP 2.75 bar. This preliminary test is to prove the hypothesis of dispersed oil found in retentate only, whereas dissolved oils are found in both retentate and permeate. The results are tabulated in Table 2.

Table 2 Comparison of membrane sizes towards the separation of dispersed and dissolved oils

| Oil Type | 0.01µm (50kDa) membrane | Feed (ppm) | Retentate (ppm) | Permeate (ppm) |
|-----------|-------------------------|------------|-----------------|----------------|
| Dissolved | Xylene | 3.03 | 2.82 | ND |
| Dissolved | Ethylenebenzene | 10.32 | 1.96 | ND |
| Dispersed | Eicosane | 5.82 | ND | ND |

| Oil Type | 0.02µm (100kDa) membrane | Feed (ppm) | Retentate (ppm) | Permeate (ppm) |
|-----------|--------------------------|------------|-----------------|----------------|
| Dissolved | Xylene | 2.84 | 1.42 | 0.39 |
| Dissolved | Ethylenebenzene | 12.62 | 0.42 | 0.07 |
| Dispersed | Eicosane | 1.64 | ND | ND |

| Oil Type | 0.2µm membrane | Feed (ppm) | Retentate (ppm) | Permeate (ppm) |
|-----------|-----------------|------------|-----------------|----------------|
| Dissolved | Xylene | 5.85 | 2.34 | 2.46 |
| Dissolved | Ethylenebenzene | 9.88 | 1.14 | 2.28 |
| Dispersed | Eicosane | 7.36 | 1.11 | ND |

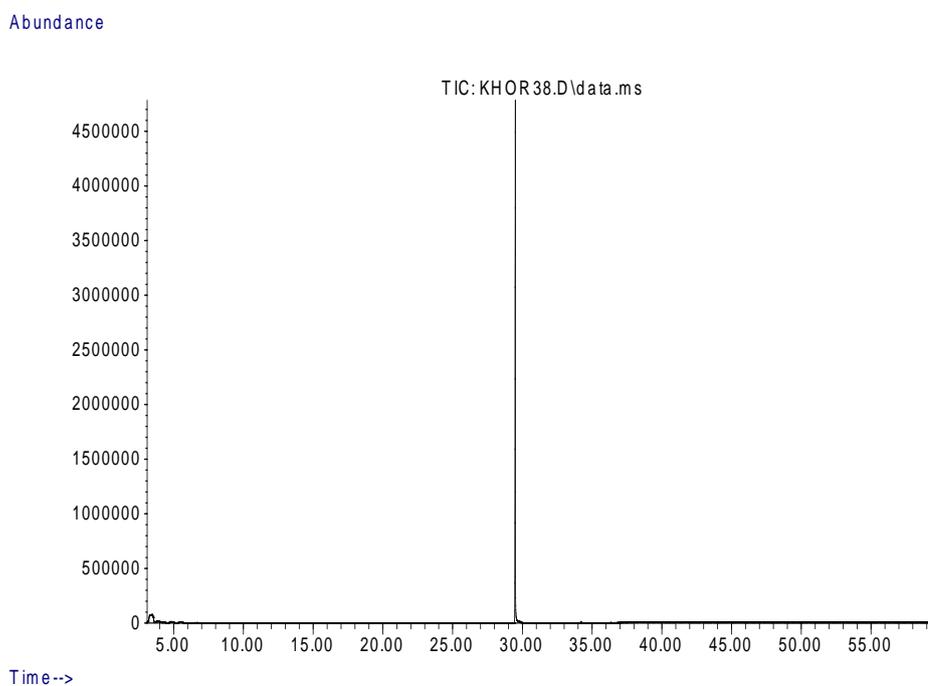


Fig. 5: Spectrum of Eicosane standards at 200ppm (Time@29.5mins)

2.6. Factorial Design Experiments

Factorial design has many advantages over the normal One-Factor-A-Time (OFAT) approach. It does not only offer efficient and economical ways to study the effect of several factors but also the interaction between the factors, thus a better conclusion can be drawn. Three factors that are used in this experiment are membrane pore sizes, membrane types and crossflow pressure/differential pressure (DP). Since 2 out of the 3 factors are discrete, the approach is to screen the main categorical factors and then establish the best operating numerical factors. Firstly, one categorical factor with one numerical factor (i.e. Size with DP) was analyzed. Next, the other categorical factor with the same numerical factor (i.e. Type with DP) was analyzed. The 2³ full factorial designs with high and low level for the factors within the operating range are shown in Table 1. There are two objectives for these experiments namely maximizing dispersed oil in retentate and maximizing dissolved oil in permeate. In this work, the results were systematically analyzed using analysis of variance (ANOVA) to determine the main and interaction effects on process behavior (Johnson, 2005). The interaction graphs were studied to estimate the differential pressure to be operated. At a later stage, this analysis results will be used in finding the optimum condition for the operation.

Table 1 2³ factorial design for efficiency of dissolved oil separation

| Variables | Code | Low level (-1) | High level (+1) |
|-----------|------|----------------|-----------------|
| DP | A | 0.5bar | 2.0bar |
| Size | B | 0.02μm | 0.20μm |
| Type | C | Hydrosart® | PESU |

3. Results and Discussions

3.1 Determination of Optimum TMP

The bigger the pores size of the membrane, the higher the TMP (Madaeni *et. al.*, 1999). Therefore, the TMP for limiting flux for the smallest pore size membrane is shown in this paper. Figure 6 shows the permeate fluxes vs. TMP at various DPs for 50 kD (0.01 μm) PESU membranes. According to the graph, optimum TMPs occurs beyond the instrumental operating limit (the limit was at 3.5 bar). This was due to the low solute concentration of 100 ppm feed sample which resembles the sample from treated produced water before disposal. Therefore in this study, limiting flux was assumed to occur at a standardized 2.75 bar of TMP for all DPs.

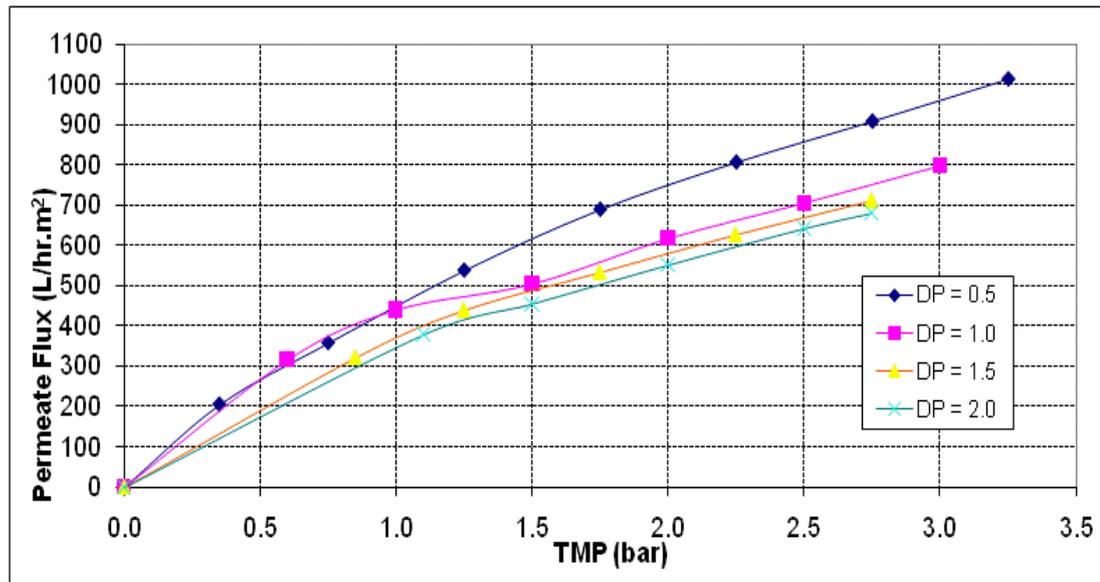


Fig. 6: Flux vs. TMP for various DPs

3.2 Effect of Pore Sizes on the Separation of Dissolved and Dispersed Oils

This is a preliminary experiment to study whether the dispersed and dissolved oils can be separated by the membranes. In Table 2 these oil components are identified and quantified in “ppm”. The qualitative analysis was done by using standard Eicosane, Ethylenebenzene and Xylene, purchased. Any concentration loss in components before and after filtration is assumed to be trapped within membrane sheet. Dispersed oil is non-detectable (ND) in permeate channel because of its larger particle size where the possibility to be retained on membrane surface or to leave the retentate channel are higher. For dissolved oil, a reduction in concentration after membrane filtration could be seen, but the extent might not be that significant if it is compared with dispersed oil.

Figure 7 shows that the amount of xylene, ethylenebenzene and eicosane measured in the feed were different due to the method of sample treatment preparation. Multiple –ratio method sample preparation employed in this study represents the constant changing effluent from treatment process (Singer *et. al.*, 2001).

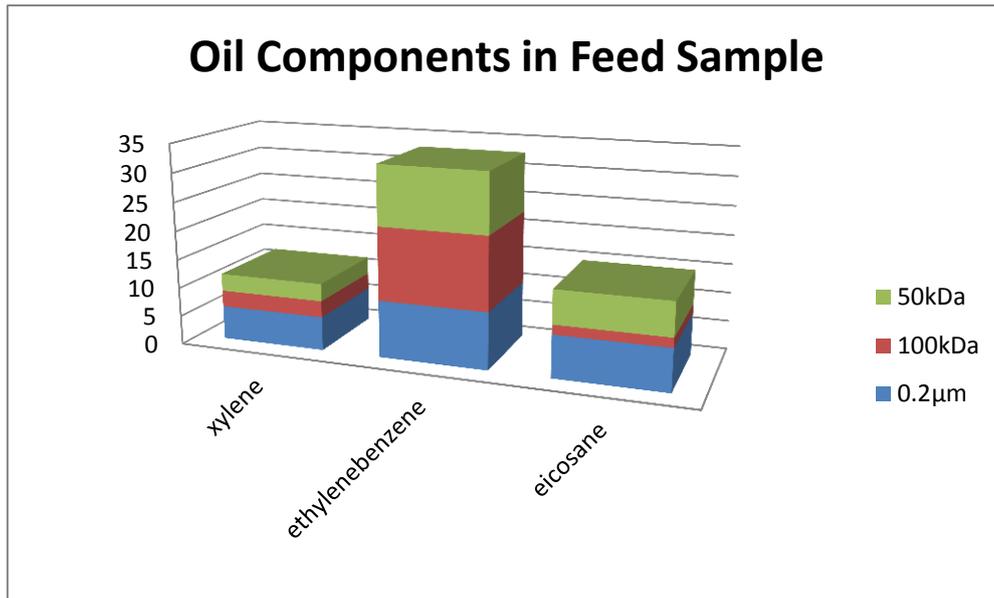


Fig. 7: Oil components in feed sample

In Table 3, a total of dissolved and dispersed oils are identified by using GCMS and their concentrations are measured and reported in percentage. These results show that only 0.02 µm UF and 0.2 µm MF membranes are able to separate the dispersed and dissolved oil in the produced water. Dissolved oil could not pass through 0.01 µm membrane pore size. Therefore, 0.01 µm UF membrane will be excluded in the comparison in the following experiments.

Table 3 Comparison of sizes towards the separation of total dispersed and dissolved oil

| Separation Results and Size | 0.01µm - Ultrafiltration | 0.02µm - Ultrafiltration | 0.2µm- Microfiltration |
|--------------------------------|-----------------------------|-----------------------------|---------------------------|
| Dispersed oil in retentate (%) | 1.3 | 14.1 | 23.1 |
| Dissolved oil in permeate (%) | 0 | 0.93 | 17.3 |

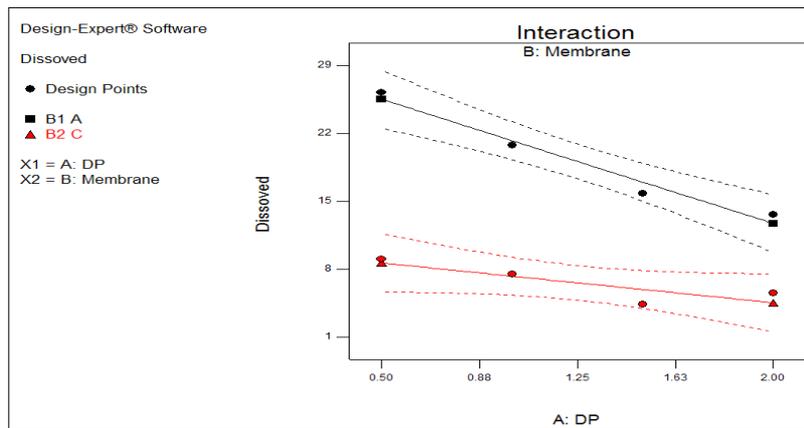


Fig. 8: Interaction graph for dissolved oil at the permeate (Membrane pore size vs. DP)

3.3 Comparison of Different Factors

Factorial design is used to generate design of experiment for comparing two factors i.e. membrane size and DPs and for the matter of effective separation the interactions of the two factors are studied. In Figure 8 & 9, Membranes A (MF) and C (UF) are of the same type (Hydrosart®) of membrane with different sizes, i.e. 0.2 μm and 0.02 μm (100 kDa) respectively. The lines are the best fit for the model, whereas the dotted lines above and below show the range of the data.

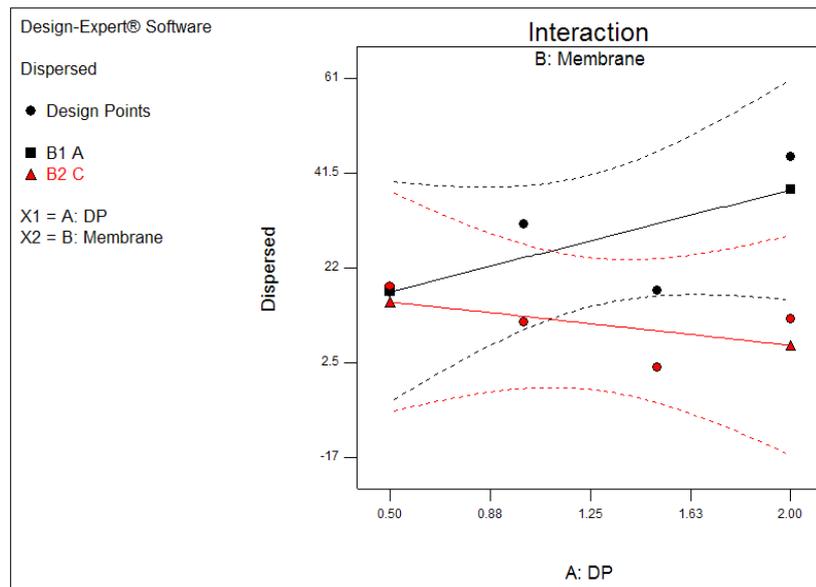


Fig. 9: Interaction graph for dispersed oil at the retentate (Membrane pore size vs. DP)

From the interaction graph for DP and size of membrane (Figure 8), Membrane A (black line) can achieve higher dissolved oil in permeate than Membrane C (red line). Similarly, for high dispersed oil in retentate (Figure 9), Membrane A (black line) is more efficient than Membrane C. To get a higher dissolved oil in permeate, DP has to be set at low pressure for both UF and MF membranes so that with the lower crossflow rate, there will be enough time for dissolved oil to pass through the membrane pores and would not be carried away into retentate. However, to have higher dispersed oil in retentate, DP has to be set at high pressure for MF membrane. This indicates that dispersed oil are mostly deposited on the MF membrane surface if the pressure is set low, and when DP was increased, the shear force from the crossflow lifted the dispersed oil on the surface into retentate. However, for the UF membrane, DP is not a significant factor for dispersed oil as has been shown in the ANOVA of dispersed oil in Table 4. Table 5 shows a summary of operating condition (DP) for maximum dissolved and dispersed oil in permeate and retentate respectively.

Table 4 ANOVA for factorial model of DP and pore size for dispersed oil

| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F | |
|------------|----------------|----|-------------|----------|------------------|---------------|
| Model | 866.1496 | 3 | 288.72 | 3.0388 | 0.1555 | |
| A-DP | 40.27724 | 1 | 40.28 | 0.423926 | 0.5505 | Insignificant |
| B-Membrane | 577.9007 | 1 | 577.90 | 6.082523 | 0.0692 | |
| AB | 247.9716 | 1 | 247.97 | 2.609952 | 0.1815 | |

Table 5 Summary table for operating condition (DP) for maximum dissolved and dispersed oil

| | MF | UF |
|-------------------------------------|------|-----------------|
| Maximizing oil concentration | DP | DP |
| Dissolved oil in permeate | Low | Low |
| Dispersed oil in retentate | High | Not significant |

Another experimental design was created to compare two factors i.e. membrane type and DPs so that their interactions for the separations can be studied. In Figure 10, Membranes A and C have the same pore size (0.02 μ m) but different material type, i.e. PESU and Hydrosart® respectively.

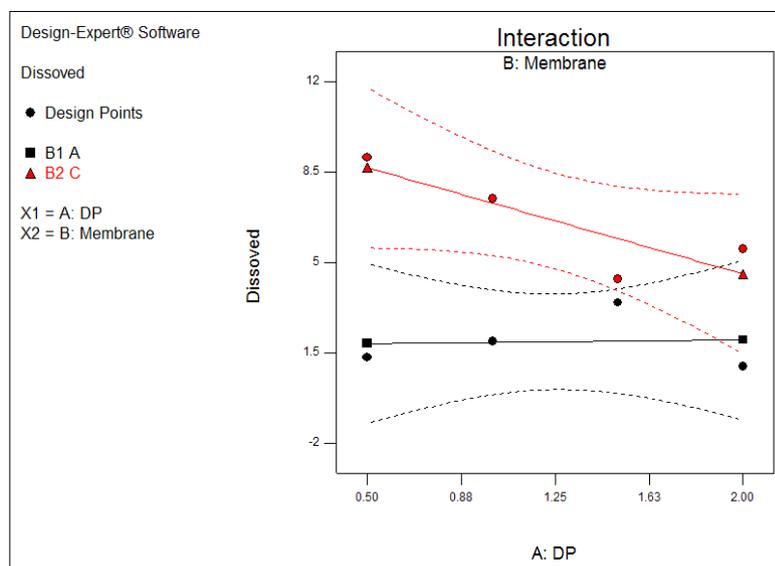


Fig. 10: Interaction graph for dissolved oil in permeate (Membrane Type vs. DP)

From the interaction graph (Figure 10), for high dissolved oil in permeate, Membrane C, Hydrosart® (red line) is better than Membrane A, PESU (black line), however the effect of the type of membrane for dispersed oil is not significant (Figure 11). The ANOVA in Table 6 also confirmed the insignificance of the factor by giving a p-value of 0.8990. As dissolved oil passes through the membrane, more of it was plugged inside the PESU membrane, while lesser dissolved oil plugged the pores inside the Hydrosart® membrane. This shows that Hydrosart® membrane material has non-fouling properties as claimed by the manufacturer. To get higher dissolved oil in permeate, DP has to be set low for both types of membrane. Similarly, DPs

have to be set low for both types of membrane to obtain higher dispersed oil in retentate.

Table 6 ANOVA for factorial model of DP and Membrane Type for dispersed oil

| Source | Sum of Squares | df | Mean Square | F Value | p-value Prob > F | |
|------------|----------------|----|-------------|----------|------------------|-----------------|
| Model | 181.8171 | 3 | 60.61 | 0.981559 | 0.4853 | |
| A-DP | 168.0075 | 1 | 168.01 | 2.721019 | 0.1744 | |
| B-Membrane | 1.128258 | 1 | 1.13 | 0.018273 | 0.8990 | Not significant |
| AB | 12.68137 | 1 | 12.68 | 0.205385 | 0.6739 | |

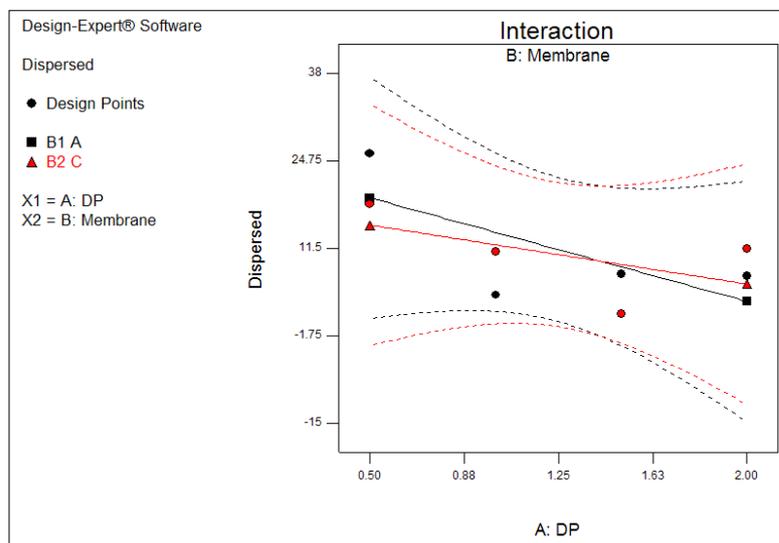


Fig. 11: Interaction graph for dispersed oil in retentate (Membrane Type vs. DP)

Figure 12 shows a summary of the efficiency of dissolved oil separation in permeate for different types and sizes of membranes and the different operating DP conditions. The legend which shows 0.2 μ m is the 0.2 μ m Hydrosart membrane; 100P is the 100kDa (0.02 μ m) Polyethersulfone membrane and 100H is the 100kDa (0.02 μ m) Hydrosart membrane. With membrane filtration incorporated in the standard test method, more than 25% of dissolved oil can be separated from the treated produced water by using 0.2 μ m membrane operating at DP 0.5 bar. Without membrane filtration, these 25% of dissolved oil will be measured as dispersed oil thus the chances of violating the disposal regulation limit increases. As DP increases the efficiency

of separation dropped. For a similar pore size, efficiency can be improved by using Hydrosart® membrane material.

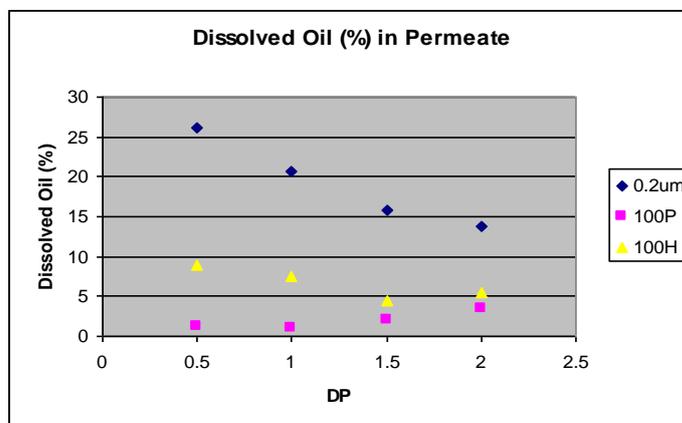


Fig. 12: Dissolved oil (%) in permeate vs. DP

4 Conclusions

In this paper, the incorporation of membrane filtration into the current standard test method has shown improvement in the oil-in-water analysis by eliminating 25% of the dissolved oil from the test sample. The effectiveness of the membrane in terms of pore size and the type of membrane and operating pressure to achieve high dispersed oil in retentate and high dissolved oil in permeate are analysed. From Table 2, conclusions can be drawn that 0.01 μm (50kDa) membrane pore size is not suitable for dissolved oil removal. This is because the pore size is too small for the dissolved oil to be removed as permeate. Table 2 also shows that dissolved oil is lower in molecular weight than dispersed oil as dispersed oil such as Eicosane is not detectable (ND) in permeate. From the experimental data, it was found that for high dissolved oil in permeate, the pore size and the type of membrane are critical. On the other hand, for high dispersed oil in retentate, the pore size is critical but not the membrane type. Moreover, the polarity of the oil has an effect towards the membrane type where in the case of dissolved oil, cellulose-based Hydrosart® membrane prevent the adsorption of oil plugging the membrane wall. In conclusion, Microfiltration using 0.2 μm Hydrosart® membrane can achieve a more efficient separation of dissolved and dispersed oil in permeate and retentate respectively.

5 Further work

In the determination of suitable DP for separation of dissolved and dispersed oil, the results give conflicting suggestion: low DP is needed for high dissolved oil but high DP is needed for high dispersed oil. Thus, to obtain a good separation by maximizing both the oil contents, it is important to get the best optimum setting of the crossflow pressure.

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