

# A Mathematic Model for Removal of Heavy Metals from Wastestreams by Surfactant and Ultrafiltration

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*Abstract:* - The goal of this study is to establish a process model based on material balance for cross-flow surfactant-based UF. The amount of heavy metals bound to surfactant, sorbed/rejected by the membrane, and present as free metal ions in the permeate and retentate were modeled. The results showed that no significant level of free metal ions was present when the  $r_s$  (moles of metal sorbed per mole of surfactant) was less than 0.3 except for Cd and Ni. Membrane only sorbed/rejected a small portion of the metals. This model worked well between surfactant concentrations of 3 and 20 mM using deoxycholic acid (DCA) as surfactant.

*Key-Words:* - Ultrafiltration, Heavy metals, Modeling, Surfactant, Mass balance

## 1 Introduction

Surfactant-based ultrafiltration (UF) is a method in which large molecular weight surfactants are added into a wastestream to promote the removal of smaller ions [1-2]. Surfactant, whose molecules will aggregate beyond a certain level called critical micellar concentration (CMC), will bind to the metal and/or organic ions. This solution can then be ultrafiltered through a compatible membrane to concentrate the waste and produce a high quality of permeate that can be reused in a manufacturing process [3-4]. This process has been shown effective to separate dissolved heavy metals and organics from simulated and real wastewaters using natural surfactants in a pilot-scale membrane system [5-7]. Several methods have been developed to simulate the binding of metals [8-9] or organics [10-11] to surfactants.

The goal of this study was to establish a process model based on mass balances for cross-flow surfactant-based UF. The amount of heavy metals bound to the surfactant, sorbed and/or rejected by the membrane, and present as free metal ions in the permeate and retentate were modeled. This model was tested using a bench-scale cross-flow membrane

system.

Before each UF experiment, the mixture of surfactant and metals was well mixed and allowed to react for 30 minutes to reach equilibrium. Therefore, the metal ions in the feed tank can be divided into two portions: those present as free ions in the solution and those bound to surfactant macromolecules (Fig.1). The partition ratio of heavy metals between the aqueous phase and the surfactant aggregates can be determined by the centrifuge method which will be discussed in the Experimental section. When the metal-surfactant mixture is pumped through the membrane housing, some free metal ions will go through the membrane as permeate, some will be adsorbed by the membrane, and the rest will be rejected by the membrane as retentate. The amount of metals adsorbed and/or rejected by the membrane can be determined by running an UF experiment using pure metal solution without surfactants. The metals bound to the surfactant macromolecules are rejected by the membrane because the sizes of metal-surfactant aggregates are much larger than the pore size of the membrane. The metals rejected by the

membrane and bound to the surfactant are circulated back to the feed tank as retentate.

The following assumptions were made:

- metal-surfactant micelles are completely rejected by the membrane,
- significant amounts of surfactant do not deposit on the membrane surface,
- metal concentration in the feed tank is almost the same as that in the membrane housing, i.e. the membrane system is well mixed, and
- free metal ions are sorbed by the membrane.

If the metal-surfactant complexes are not totally rejected by the membrane, some metals will always be detected in permeate. But this was not the case. All metal ions were substantially rejected ( $\sigma > 99.9\%$ ) in the bench-scale experiments [3-4]. This shows that assumption (a) is valid. Evidence for the validity of assumption (b) is that the permeate flux remained constant during the experiment. This showed that significant amounts of surfactant did not deposit on the membrane surface. Furthermore, the ability of the surfactant to act as a biological detergent and high cross-flow velocity (4 m/sec) will clean the membrane surface during the experiment [3-4]. The membrane housing was opened after the separation trial and no surfactant was observed on the membrane surface. A high recirculation flow rate and extremely small volume in the membrane housing compared to that in the feed tank assure the validity of assumption (c). From the bottle test which will be described in Experimental section, the membrane did adsorb some heavy metals (assumption d).

## 2 Process Modeling

The mass balance for heavy metals in the cross-flow system can be described by the following (Fig.1):

$$M_{total} = M_s + M_{sor,m} + M_p + M_{free} \quad (1)$$

where  $M_{total}$  = total moles of metal in the system,  $M_s$  = the moles of metal sorbed by surfactant micelles,  $M_{sor,m}$  = the moles of metal sorbed by the membrane,  $M_p$  = the moles of metal ions that have permeated through the membrane, and  $M_{free}$  = the moles of free metal present in the feed tank.

Assuming that the sorption process of metal to surfactant can be described by the Langmuir adsorption isotherm, the moles of metal sorbed by surfactant,  $M_s$ , can be expressed:

$$M_s = ([P]_{total}) \times V_t \times r_s = ([P]_{total}) \times V_t \times \frac{n_s K_s A_{t,F}}{1 + K_s A_{t,F}} \quad (2)$$

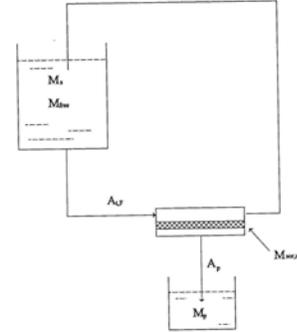


Fig.1 Schematic of metal distribution within cross-flow UF system

where  $[P]_{total}$  = surfactant concentration in the feed tank,  $V_t$  = volume in the feed tank,  $r_s$  = the moles of metal sorbed per mole of surfactant,  $n_s$  = available binding sites for metal per mole of surfactant,  $K_s$  = association constant, and  $A_{t,F}$  = free metal concentration in the feed tank.

The volume in the feed tank,  $V_t$ , can be expressed as

$$V_t = V_t^o - \int_0^{V_p} dV_p = V_t^o - V_p \quad (3)$$

where  $V_t^o$  = initial volume in the feed tank and  $V_p$  = permeate volume.

The amount of heavy metal sorbed by the membrane,  $M_{sor,m}$ , can be obtained from the membrane sorption isotherm which also can be represented by a Langmuir adsorption isotherm,

$$M_{sor,m} = S_m \times r_m = S_m \times \frac{n_m K_m A_{t,F}}{1 + K_m A_{t,F}} \quad (4)$$

where  $S_m$  = membrane area,  $r_m$  = the moles of metal sorbed by a unit membrane area,  $n_m$  = maximum moles of metal sorbed per unit membrane area, and  $K_m$  = an empirical constant.

The amount of heavy metals present in permeate,  $M_p$ , can be written as:

$$M_p = \int_0^{V_p} A_p dV_p \quad (5)$$

where  $A_p$  = permeate concentration and  $V_p$  = permeate volume.

The relationship between the  $A_p$  and  $A_{t,F}$  can be described by the absolute rejection ratio,  $\sigma_{abs}$ , as

$$\sigma_{abs} = 1 - \frac{A_p}{A_{t,F}} \quad (6)$$

where  $A_{t,F} = M_{rent}/V_t$ . If the membrane system is well mixed,  $M_{rent}$ , the moles of free metal ions rejected by the membrane as retentate, is almost equal to  $M_{free}$  or  $V_t * A_{t,F}$ . The absolute rejection ratio means that only metals rejected by the membrane are taken into account.

Substitution of Eqs. (2), (3), (4), and (5) into Eq. (1), the general material balance equation becomes

$$M_{total} = (I P_{total} J) \times (V_i^o - V_p) \times \frac{n_s K_s A_{t,F}}{I + K_s A_{t,F}} \quad (7)$$

$$+ \int_0^{V_p} A_p dV_p + S_m \times \frac{n_m K_m A_{t,F}}{I + K_m A_{t,F}} + (V_i^o - V_p) \times A_{t,F}$$

where  $A_p$  can be replaced by  $A_{t,f} \times (1 - \sigma_{abs})$  from Eq. (6). Therefore,  $A_{t,f}$  can be solved from Eq. (7).

### 3 Experimental

#### 3.1 Chemicals

A sodium salt of deoxycholic acid (DCA) was obtained from Sigma Chemical Company (St. Louis, Missouri) and used as received. Cadmium chloride, zinc chloride, nickel sulfate, copper sulfate, and lead acetate were obtained from Sigma. Deionized water was used in preparing all solutions and reagents. RGO3 membrane made from anisotropic acrylonitrile has a 2,000 MWCO for the flat-sheet type. All membranes were provided by Osmonics Inc., Minnetonka, Minnesota.

#### 3.2 Methods

UF experiments: Cross-flow UF runs were made by using a bench-top system with an effective membrane area of 138.7 cm<sup>2</sup> (flat-sheet type). Membranes were soaked in deionized water to remove the protective glycerol layer which was applied before shipment. A new membrane was used for each bench-scale experiment while the membrane was reused for each pilot-scale experiment after it had been cleaned thoroughly. Membrane cleaning was considered satisfactory if the deionized water flux after cleaning was

higher than 95% of that of a new membrane. The pressure drop across the membrane was maintained at 276 kPa for the bench-scale system and 552 kPa for the pilot-scale setup. The temperature was set at 30°C. All the S/M ratios used in this research are calculated using the molar concentration of surfactant and metal.

For the bench-scale system, the feed tank initially contained a 3-liter solution of metals and surfactant. After collection of 250 mL of permeate, 2 grams of surfactant was added into the feed tank. The collected permeate at each interval was put back into the feed solution to keep the concentration of each metal as constant as possible. Therefore, the S/M ratio in the feed tank increased during the experiment.

Partition (centrifuge) experiments: Partition experiments were conducted to determine the membrane interference with the binding of surfactant and metal ions. Various amounts of surfactant were added into several solutions with fixed metal concentrations. After a 30-minute reaction time, the samples were centrifuged for 10 minutes at 10,000 rpm (Sorvall, Du Pont Instruments, Newtown, Connecticut) and then filtered through a 0.45-micrometer filter (Whatman Filter Company, Maidstone, England). The macromolecules containing absorbed metals and metal precipitates were separated from the liquid phase containing free metal ions and surfactant monomers. The liquid-phase samples were analyzed for their metal contents using an atomic adsorption spectrophotometer (AAS).

Membrane sorption experiments: Amounts of metals sorbed by the membrane were determined by the bottle test. A piece of membrane (RGO3, 2,000 MWCO) was placed between the top of a bottle and the cap which sealed the bottle completely. An opening within the cap, which had the same size as the opening of the bottle, was cut to allow the metal solution to permeate through the membrane and collected by another bottle on the bottom. No metal solution was collected in the bottom bottle after duration of 10 hours. Therefore, the metals sorbed by the membrane could be determined by measuring the metal contents in the bulk solution and applying a material balance for metal before and after the experiment.

Metal concentrations in the feed and permeate samples were determined by using a Varian 975 AAS. A new calibration curve was prepared for each analysis to ensure the accuracy of readings.

### 4 Results and Discussion

The sorption isotherms of metals to surfactant are shown in Fig.2. No significant level of free metal ions is present when the  $r_s$  is less than 0.3 except for Cd and Ni. The curves shown in Fig.3 are determined by the least squares method which minimizes the square errors between the measured  $r_s$  and expected  $r_s$  calculated from the Langmuir isotherm. Table 1 lists the values of  $n_s$  and  $K_s$  for the Langmuir sorption isotherms. Pb has the highest  $K_s$ , 26.8  $\text{mM}^{-1}$ , followed by Cu, Cd, Zn, and Ni. That shows that Pb has the highest affinity to the DCA.

The sorption isotherms of metals to membrane are close to each other and are shown in Fig.4. The curves shown in Fig.3 are also determined from the least squares method to fit the calculated data to those obtained experimentally. Table 2 lists the values of  $n_m$  and  $K_m$  in the Langmuir sorption isotherms for metals sorbed by the membrane. Pb has the highest  $K_m$ , 0.37  $\text{mM}^{-1}$ , followed by Cu, Cd, Ni, and Zn.

Table 1 Values of  $n_s$  and  $K_s$  in Langmuir adsorption isotherms for metals and surfactant (DCA)

Metal	$n_s$	$K_s$ ( $\text{mM}^{-1}$ )
Cd	0.443	14.4
Cu	0.484	18.6
Ni	0.511	10.0
Pb	0.532	26.8
Zn	0.521	14.0

Note: Langmuir adsorption isotherm: 
$$r_m = \frac{n_m K_m A_{t,F}}{1 + K_m A_{t,F}}$$

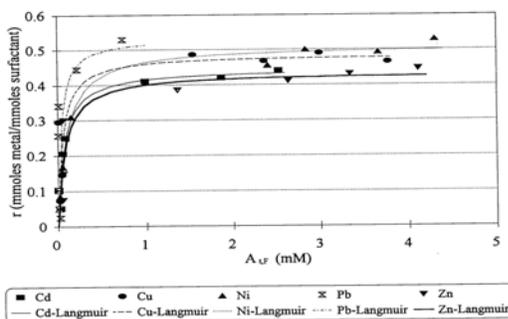


Fig.2 Langmuir sorption isotherms for metals and surfactant

Table 2 Values of  $n_m$  and  $K_m$  in Langmuir adsorption isotherms for metals and membrane (RGO3)

Metal	$n_m$	$K_m$
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	(mM <sup>-1</sup> )	
Cd	1.14	0.22
Cu	0.814	0.34
Ni	1.07	0.16
Pb	0.911	0.37
Zn	1.44	0.12

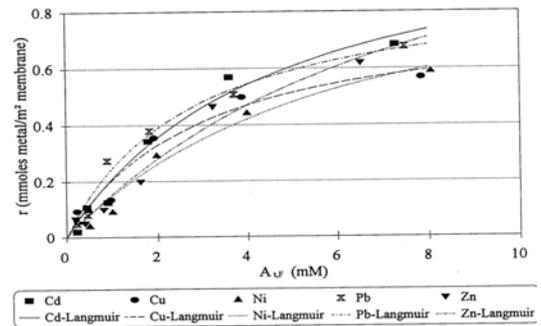


Fig.3 Langmuir sorption isotherms for metals and membrane

Table 3 to table 7 summarizes the metal distributions for the bench-scale UF experiments. The amounts of heavy metals sorbed by the surfactant initially increased with increasing surfactant concentration and then reached a plateau which indicated that surfactant was saturated by the heavy metals. Most of the metals were sorbed by the DCA at the S/M ratio around 3. Only small amounts of heavy metals were sorbed by the membrane. This indicates that surfactant (DCA) is the key for the success of surfactant-based UF. This model seems to underpredict the permeate concentrations at lower DCA concentration, and overpredict the permeate concentrations at higher DCA concentrations. Overall, this model worked well between surfactant concentrations of 3 mM (9.6 mmoles / 3 liters) and 20 mM. One of the reasons for overprediction at higher DCA concentrations is that the Langmuir sorption isotherms do not match the data points very well at extremely low  $A_{t,F}$ . The other reason is formation of a gel layer on the membrane surface which increases the amount of metals rejected by the membrane. The discrepancy between modeled and measured permeate concentration can be minimized by using multiple types of binding between surfactant and heavy metals [8-9].

Table 3 Cadmium distribution in membrane system

DCA <sup>a</sup> (mM)	DCA <sup>a</sup> (mmole)	Cd <sup>b</sup> (mmole)	$M_s$ (mmole)	$M_p$ <sup>b</sup> (mmole)	$M_{sor,m}$ <sup>c</sup> (mmole)	$M_{rent}$ <sup>d</sup> (mmole)	$A_p$ (mM)	Measured $A_p$ (mM)
1.6	4.80	7.50	2.06	0.368	0.0455	4.93	1.47	1.88
3.2	9.60	7.50	4.02	0.233	0.0322	3.12	0.930	0.920
4.8	14.4	7.50	5.70	0.117	0.0181	1.57	0.457	0.549

6.4	19.2	7.50	6.66	0.0510	0.00843	0.684	0.201	0.206
9.6	28.8	7.50	7.14	0.0180	0.00309	0.242	0.0721	0.0621
12.8	38.4	7.50	7.25	0.0104	0.00180	0.140	0.0422	0.00511
16.0	48.0	7.50	7.30	0.00735	0.00127	0.0986	0.0290	0.00224
19.2	57.6	7.50	7.32	0.00567	0.00101	0.0761	0.0231	0.000751

<sup>a</sup>Feed volume ( $V_f$ ) = 3 liters; <sup>b</sup>Permeate volume ( $V_p$ ) = 0.25 liters; <sup>c</sup>Membrane area = 0.139 m<sup>2</sup>; <sup>d</sup>Retentate volume ( $V_{rent}$ ) = 2.75 liters.

**Table 4 Copper distribution in membrane system**

DCA <sup>a</sup> (mM)	DCA <sup>a</sup> (mmole)	Cu <sup>a</sup> (mmole)	M <sub>s</sub> (mmole)	M <sub>p</sub> <sup>b</sup> (mmole)	M <sub>so,r,m</sub> <sup>c</sup> (mmole)	M <sub>re,n</sub> <sup>d</sup> (mmole)	A <sub>p</sub> (mM)	Measured A <sub>p</sub> (mM)
1.6	4.80	12.6	2.29	0.778	0.0609	9.51	3.11	3.39
3.2	9.60	12.6	4.56	0.607	0.0539	7.42	2.43	2.52
4.8	14.4	12.6	6.78	0.439	0.0449	5.37	1.76	1.78
6.4	19.2	12.6	8.91	0.279	0.0334	3.41	1.12	1.09
9.6	28.8	12.6	11.8	0.0653	0.0101	0.798	0.261	0.222
12.8	38.4	12.6	12.3	0.0233	0.0038	0.285	0.0931	0.0171
16.0	48.0	12.6	12.4	0.0138	0.0023	0.169	0.0550	0.0132
19.2	57.6	12.6	12.5	0.0101	0.0016	0.119	0.0391	0.0113

<sup>a</sup>Feed volume ( $V_f$ ) = 3 liters; <sup>b</sup>Permeate volume ( $V_p$ ) = 0.25 liters; <sup>c</sup>Membrane area = 0.139 m<sup>2</sup>; <sup>d</sup>Retentate volume ( $V_{rent}$ ) = 2.75 liters.

**Table 5 Nickel distribution in membrane system**

DCA <sup>a</sup> (mM)	DCA <sup>a</sup> (mmole)	Ni <sup>a</sup> (mmole)	M <sub>s</sub> (mmole)	M <sub>p</sub> <sup>b</sup> (mmole)	M <sub>so,r,m</sub> <sup>c</sup> (mmole)	M <sub>re,n</sub> <sup>d</sup> (mmole)	A <sub>p</sub> (mM)	Measured A <sub>p</sub> (mM)
1.6	4.80	14.7	2.39	0.870	0.0596	11.4	3.48	3.82
3.2	9.60	14.7	4.76	0.703	0.0522	9.21	2.81	3.03
4.8	14.4	14.7	7.08	0.539	0.0436	7.06	2.16	2.44
6.4	19.2	14.7	9.30	0.382	0.0338	5.01	1.53	1.87
9.6	28.8	14.7	12.8	0.138	0.0143	1.80	0.551	0.546
12.8	38.4	14.7	14.0	0.0510	0.0056	0.668	0.204	0.175
16.0	48.0	14.7	14.3	0.0290	0.0033	0.380	0.116	0.0791
19.2	57.6	14.7	14.4	0.0201	0.0023	0.263	0.0802	0.0153

<sup>a</sup>Feed volume ( $V_f$ ) = 3 liters; <sup>b</sup>Permeate volume ( $V_p$ ) = 0.25 liters; <sup>c</sup>Membrane area = 0.139 m<sup>2</sup>; <sup>d</sup>Retentate volume ( $V_{rent}$ ) = 2.75 liters.

**Table 6 Lead distribution in membrane system**

DCA <sup>a</sup> (mM)	DCA <sup>a</sup> (mmole)	Pb <sup>a</sup> (mmole)	M <sub>s</sub> (mmole)	M <sub>p</sub> <sup>b</sup> (mmole)	M <sub>so,r,m</sub> <sup>c</sup> (mmole)	M <sub>re,n</sub> <sup>d</sup> (mmole)	A <sub>p</sub> (mM)	Measured A <sub>p</sub> (mM)
1.6	4.80	15.5	2.53	0.974	0.0780	11.9	3.90	3.99
3.2	9.60	15.5	5.05	0.784	0.0713	9.58	3.14	3.15
4.8	14.4	15.5	7.55	0.595	0.0627	7.28	2.32	2.34
6.4	19.2	15.5	10.0	0.410	0.0511	5.02	1.63	1.63
9.6	28.8	15.5	14.1	0.100	0.0180	1.23	0.392	0.386
12.8	38.4	15.5	15.2	0.0235	0.00473	0.287	0.0940	0.00652
16.0	48.0	15.5	15.3	0.0124	0.00254	0.151	0.0496	0.00481
19.2	57.6	15.5	15.4	0.00836	0.00172	0.102	0.0334	0.00242

<sup>a</sup>Feed volume ( $V_f$ ) = 3 liters; <sup>b</sup>Permeate volume ( $V_p$ ) = 0.25 liters; <sup>c</sup>Membrane area = 0.139 m<sup>2</sup>; <sup>d</sup>Retentate volume ( $V_{rent}$ ) = 2.75 liters.

**Table 7 Zinc distribution in membrane system**

DCA <sup>a</sup> (mM)	DCA <sup>a</sup> (mmole)	Zn <sup>a</sup> (mmole)	M <sub>s</sub> (mmole)	M <sub>p</sub> <sup>b</sup> (mmole)	M <sub>so,r,m</sub> <sup>c</sup> (mmole)	M <sub>re,n</sub> <sup>d</sup> (mmole)	A <sub>p</sub> (mM)	Measured A <sub>p</sub> (mM)
1.6	4.80	11.3	2.44	0.631	0.0528	8.17	2.52	2.64
3.2	9.60	11.3	4.84	0.460	0.0414	5.95	1.84	1.87
4.8	14.4	11.3	7.14	0.296	0.0288	3.83	1.19	1.14
6.4	19.2	11.3	9.11	0.155	0.0162	2.01	0.621	0.456

9.6	28.8	11.3	10.8	0.0385	0.00428	0.498	0.154	0.133
12.8	38.4	11.3	11.0	0.0184	0.00207	0.238	0.0736	0.0623
16.0	48.0	11.3	11.1	0.0121	0.00136	0.156	0.0482	0.0153
19.2	57.6	11.3	11.2	0.00895	0.00101	0.116	0.0358	0.00762

<sup>a</sup>Feed volume ( $V_f$ ) = 3 liters; <sup>b</sup>Permeate volume ( $V_p$ ) = 0.25 liters; <sup>c</sup>Membrane area = 0.139 m<sup>2</sup>; <sup>d</sup>Retentate volume ( $V_{rent}$ ) = 2.75 liters.

## 5 Conclusions

The results showed that no significant level of free metal ions was present when the  $r_s$  (moles of metal sorbed per mole of surfactant) was less than 0.3 except for Cd and Ni. Most of the metals are sorbed by the surfactant. Only small amounts of metals are sorbed/rejected by membrane. This model seems to underpredict the permeate concentrations at lower DCA concentration, and overpredict at higher DCA concentrations. The discrepancy between modeled and measured permeate concentration can be minimized by using multiple types of binding between surfactant and heavy metals. This model can adequately estimate the permeate concentration for surfactant concentrations between 3 and 20 mM using deoxycholic acid (DCA) as surfactant.

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