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Micellar-Enhanced Ultrafiltration of Heavy Metals Using Lecithin

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ABSTRACT

Conventional treatment methods for removal of heavy metals from metal finishing operations are usually energy-intensive and costly. Micellar-enhanced ultrafiltration (MEUF) with synthetic surfactants is a recently developed technique which can remove heavy metals and other small molecular weight ions from wastestreams at relatively lower costs and without a phase change. Lecithin, a natural, inexpensive, nontoxic, and biodegradable surfactant exhibits emulsifying characteristics which can be used in a MEUF. The binding of various lecithins to cadmium, copper, lead, nickel, and zinc—in a mixture and individually—was studied using a continuous diafiltration method. This technique uses small volumes of toxic waters and produces an entire isotherm with just one experiment. In the presence of all five heavy metals, the lecithin in this study showed the following affinity: Cu > Cd ~ Zn > Ni. In experiments when only one metal was present, lecithin exhibited the following affinity: Ni > Cu ~ Zn > Cd. Lead was not bound significantly in either scenario.

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INTRODUCTION

Surface treatment of metals is a major source of pollution when compared to some other manufacturing operations. The toxic nature of many of the chemicals involved in such treatment, and sometimes even the harmful nature of the metals being coated, means that any release of materials has the potential of serious effects on the environment (1). Effluents from metal finishing plants contain a variety of toxic substances. Among these are heavy metals and other inorganic compounds as well as organic compounds. Traditional methods of separating soluble compounds from a stream often involve a phase change, as in distillation, or extraction followed by distillation. These procedures are energy-intensive and therefore alternative low-energy separation processes are desirable.

Water is used throughout metal finishing facilities for rinsing work places, washing away spills, air scrubbing, process fluid replenishment, cooling and lubrication, washing of equipment and work places, quenching, spray booths, and assembly and testing (2).

Solids removal methods for metals and other pollutants include sedimentation, diatomaceous earth filtration, membrane filtration, granular bed filtration, and flotation. The common treatment techniques for the recovery of metals are evaporation, ion exchange, electrolytic recovery, electrodialysis, and reverse osmosis (2).

Membrane processing has been used in various industries including water desalination, wastewater treatment, gas separations, and petroleum, food, and beverage processing. Ultrafiltration and reverse osmosis techniques have also been applied in aluminum forming, iron and steel manufacturing, battery manufacturing, nonferrous metals manufacturing, and metal finishing. Ultrafiltration requires little energy and relatively low pressures, and it is generally much less expensive than other separation techniques per unit volume of solution processed.

The main limitations of conventional membranes in waste treatment processes are 1) membrane life and durability, 2) sensitivity of membranes to chlorine, and 3) stability of membranes at high and low pH values. In recent years, new membranes have become commercially available, providing for higher operating temperatures and pressures, and wider pH ranges. They have high flow rates and are relatively durable. However, they are limited to treatment of pollutants with molecular weights in the range of 1000 to 500,000; therefore, heavy metal ions and smaller molecular weight organic compounds cannot be removed without additional processing techniques.

Micellar-enhanced ultrafiltration (MEUF) is a recently proposed method of treating wastewaters containing heavy metals and toxic organic compounds (3). It combines the high selectivity of reverse osmosis with the higher flow rate of ultrafiltration. The underlying principle is to increase the size of the pollutant molecules so they can be removed when passed through a membrane with an appropriate pore size.

In MEUF, Fig. 1, a large molecular weight surfactant is added to a wastewater to promote the removal of smaller contaminants, such as heavy metals, which have molecular weights of 60–210.

Beyond a certain concentration, the surfactant molecules will attach to each other and form macromolecules or micelles. The metal ions are electrostatically bound in the structure of these micelles which have a molecular weight in the range of 2000. When this mixture is ultrafiltered through a compatible membrane, the resulting permeate will contain low concentrations of surfactant and heavy metal ions which may be reused in manufacturing. The retentate, whose volume is now significantly reduced, can be further treated by various chemical and physical methods (3).

This research involved the use of lecithin, a natural surfactant that has many sources including as a by-product recovered from soybean oil refining. Soybean lecithin is nontoxic, biodegradable, inexpensive ($0.08 per pound), and has good surfactant characteristics. It is used in a wide range of applications including uses in the medical, cosmetic, and food industries. A synthetic surfactant, sodium dodecyl sulfate, has been cited in the literature as an excellent binding and/or micelle-forming agent for some heavy metals; rejection ratios of 99+% have been observed for copper

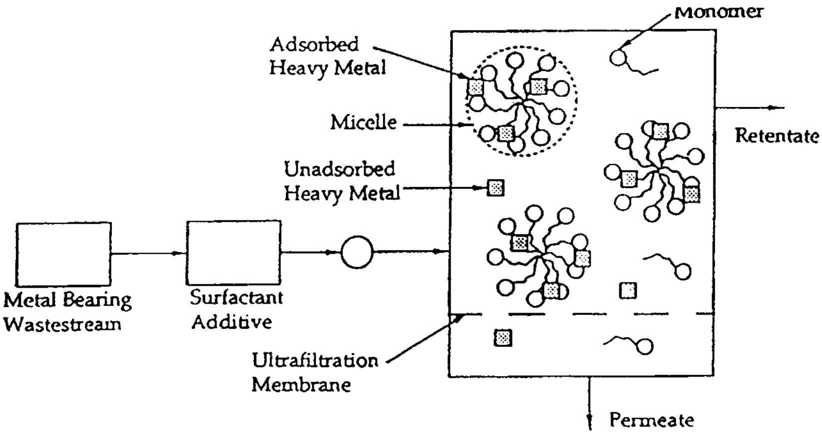

FIG. 1 Schematic of micellar enhanced ultrafiltration.

(3). However, the cost of this surfactant ranges from $30.00 to 35.00 per pound.

The goal of this project was to develop an efficient MEUF process using lecithin. This involved the study of the binding characteristics of different types of lecithins to five different heavy metals using a continuous diafiltration technique (4). This method is a relatively quick way for studying binding behavior. It also involves using a very small amount of toxic wastewaters to predict the performance of MEUF at the pilot-plant scale.

LITERATURE REVIEW

Micellar Enhanced Ultrafiltration

The idea of the addition of macromolecules or an emulsifying agent to an ultrafiltration process was first suggested by Michaels (5). The agent selectively binds to certain metal ions or groups of ions and provides for a more economical process since the size of the molecule to be removed has increased.

Strathman presented a process for the selective removal of heavy metal ions with macromolecular chelating agents or emulsified ion-exchange materials in combination with ultrafiltration (6). Rejections for cadmium, zinc, silver, copper, and mercury were reported to be above 98%.

Chaufer and Deratiani presented complexation–ultrafiltration as a means of removing metal ions with water-soluble macromolecules (7). Polymers which are known to bind to metals are added to a wastestream and then ultrafiltered. Rejection ratios of 96+% were observed for mercury and copper.

Christian and Scamehorn presented procedures for the treatment of heavy metals by micellar enhanced ultrafiltration (3, 8). Synthetic anionic surfactants were added to wastestreams containing heavy metals. In the removal of copper and chromium, rejection ratios of 99+% were observed.

Any surfactant will form macromolecules when its concentration is above a specific value, called its critical micelle concentration (cmc). In water, Fig. 2, these micelles will consist of molecules that have their hydrophobic groups bunched together internally, and their hydrophilic and cationic groups facing the outside.

These macromolecules will consist of an often typical number of monomers, called an aggregation number. A micelle composed of ionic surfactants is highly charged. Due to electrostatic forces, these micelles and the counterions in the solution are brought close to each other. These counterions are present as a surface excess. Since adsorption results in a surface excess, these counterions may also be considered as adsorbed

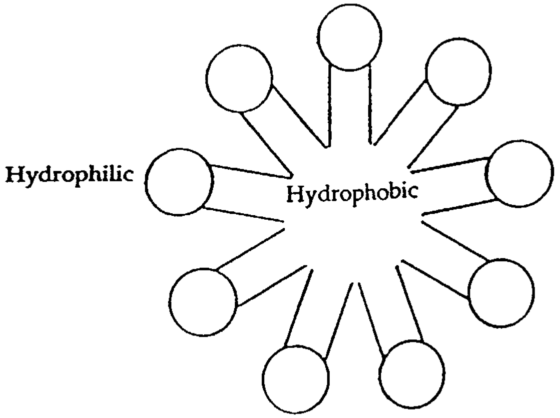

FIG. 2 Micelle structure in aqueous solution.

at the micelle–solution interface. These adsorbed counterions are either bound in the Stern layer of the micelle or present in excess in the electrical double layer surrounding the micelle. The higher the valence of a counterion, the more it will tend to be adsorbed on the micelle (3).

In the case of the ultrafiltration of a micellar solution containing an ionic surfactant and multivalent counterions, whenever the micelles are rejected, electroneutrality requires that the adsorbed counterions be rejected (3, 8).

Lecithin

Lecithin is a complex mixture of phosphatides, or phospholipids, produced from a variety of vegetable and animal sources, but primarily from plant seed. It is nearly insoluble in water—described as water dispersible (9). It is amphoteric—meaning that it can act as either a base or an acid (10).

Soybean lecithin is obtained as a by-product of oil processing and is mainly used because of its availability and excellent properties, especially its emulsifying behavior, color, and taste. Lecithin is used in a wide range of applications. In the baking industry it has been used for centuries because of its surface-active properties in the processing of foods. It is the principal emulsifying agent, in the form of egg yolk, used in the preparation of stable salad dressing emulsions such as mayonnaise (11).

Due to lecithin's emulsifying properties, it is believed to act as an acceptable surfactant in the application of MEUF for the treatment of heavy metals. The structure of lecithin can vary, but an average molecular weight of 750 was assumed for the types of lecithins used in these experiments. Even if the cmc is high and the aggregation number is low, the resulting lecithin macromolecule will be large enough to be used with membranes with a 2000–10,000 molecular weight cutoff (MWCO). Figure 3 illustrates the basic structure of a phospholipid.

EXPERIMENTAL

Experimental Plan

To study the binding characteristics of lecithin to heavy metals, a diafiltration procedure was used. This entails the addition of a reservoir to the

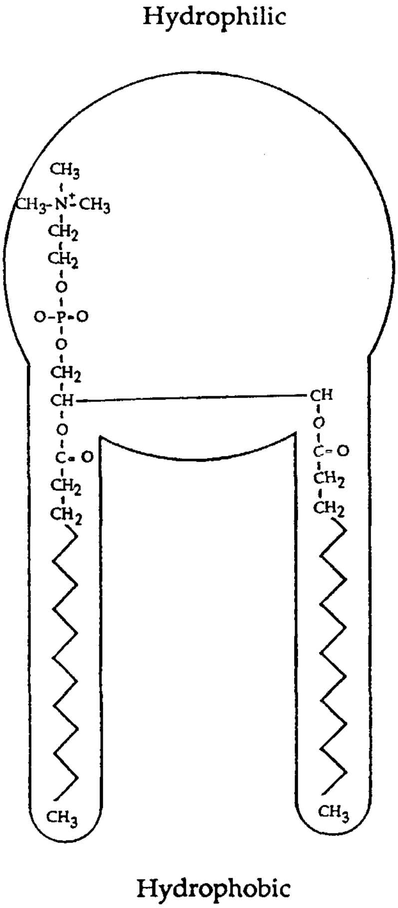

FIG. 3 Structure of a phospholipid.

ultrafiltration cell. The surfactant was placed in the cell; the heavy metals were placed in the reservoir (Fig. 4). At first, pressure was applied to both units. Then a three-way valve was turned so it simultaneously cut off the pressure to the cell and opened the path for liquid to flow from the reservoir into the cell. The vacuum created between the units allows only the same amount of liquid *in* from the reservoir as it allows to permeate *out* from the cell. Therefore, the cell volume is kept constant while the heavy metal solution passes through. If the rejection is zero, then the concentration of the solute on either side of the membrane is equal. When performing binding studies with diafiltration, membranes are chosen which reject none of the solutes flowing in from the reservoir and which completely reject the macromolecules in the cell.

The following system characteristics were studied: 1) the flux rate, 2) the rejection of metal ions, 3) the rejection of lecithin, and 4) the pH of the feed, permeate, and retentate solutions.

Elmpur N-1 was examined for its heavy metal rejection and flux efficiency. Table 1 summarizes the characteristics of Elmpur N-1.

Cadmium, copper, lead, nickel, and zinc were used as their salts present in the metal finishing industry. Their concentrations were set higher than their typical effluent concentrations for metal finishing wastewaters in order to saturate the surfactant. Table 2 summarizes the concentration of the heavy metals used in this project.

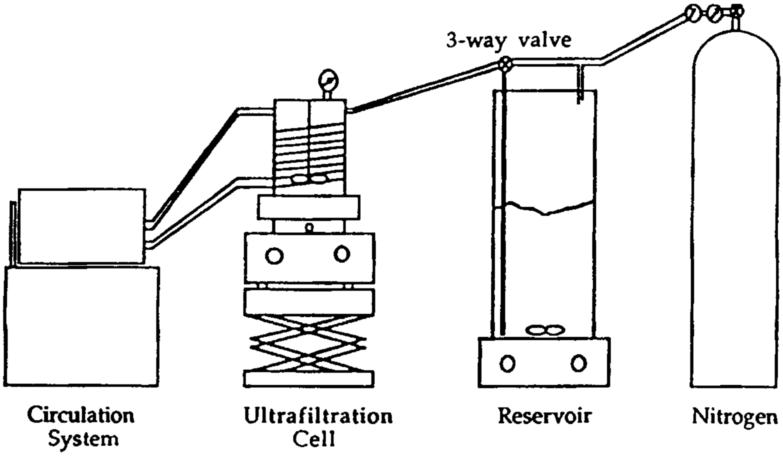

FIG. 4 Experimental apparatus.

TABLE 1
Approximate Composition of Elmpur N-1[a,b] (9)

Compound	Percent	Molecular Weight[c]	Formula
Phosphatidylcholine	22	758	$C_{42}H_{80}O_8NP$
Phosphatidlethanolamine	23	716	$C_{39}H_{74}O_8NP$
Phoshatidyl inositol	20	834	$C_{43}H_{78}O_{13}P$
Phosphatidic acid	5	671	$C_{37}H_{67}O_8P$
Phytoglycolipids	13		
Phosphatidylserine	2		
Other phospholipids	12		

[a] *Specifications*: acetone insolubles, 95–98%, color, yellow; moisture, 1.0%; physical state, powder.

[b] Consist of fatty acids: palmitic, stearic, oleic, linoleic, linolenic.

[c] Calculated as the 16:0, 18:2 form. Molecular weights vary approximately 24 daltons less than stated amount for 16:0, 16:0 form to 34 daltons more for the 18:0, 18:0 form.

Equipment

Diafiltration runs were made using a Nucleopore 400 mL high-pressure stirred cell unit with an effective membrane diameter of 72 mm. Water at a constant temperature was circulated through a copper coil wrapped around the cell using a circulation system. Nitrogen was used as the pressure source. The mixture in the cell was agitated using a magnetic stirrer equipped with a tachometer. Permeate samples, 10 mL each, were taken throughout the run for metal ion and lecithin analysis. The time required to produce a 10-mL permeate was also measured so that flux rates could later be calculated.

New membranes were used in each experiment. Membranes were soaked in deionized water. This allowed the membrane to reach room temperature and also removed the protective glycerol layer applied before

TABLE 2
Concentration of Heavy Metals

	Concentration	
	mM	mg/L
Cadmium	4.6	517
Copper	4.6	425
Lead	0.13	27
Nickel	3.1	182
Zinc	6.5	292

shipment. All membranes were provided by Osmonics Inc., Minnetonka, Minnesota.

Deionized water was used in making all solutions and reagents. Glassware and all other equipment were washed with soap and water and rinsed with distilled water and, where not destructive, then rinsed with H_2SO_4 to minimize the effect of residual metal content between experiments.

Metal concentrations were analyzed with a Varian 975 Atomic Adsorption Spectrophotometer. Standard solutions were tested during all analyses to ensure the accuracy of the readings. Lecithin concentrations were measured by relating the phosphorus content of a sample to the concentration of the surfactant using the A.O.C.S. (American Oil Chemists Society) official method Ca 12-55.

Chemicals

Cadmium chloride, zinc chloride, nickel sulfate, copper sulfate, lead acetate, and sodium dodecyl sulfate (SDS) were obtained from Sigma, St. Louis, Missouri, and were used as received. Lecithin was provided by Lucas Meyer, Decatur, Illinois, and Central Soya, Fort Wayne, Indiana. Lecithin concentrations were set at 8.9, 17.8, 35.5, and 71.0 mM.

Procedures

The general differential mass balance in a diafiltration experiment is described by

$$\begin{aligned}\Delta \text{ moles in the cell} &= \text{moles in from reservoir} \\ &\quad - \text{moles out from the permeate}\end{aligned} \tag{1}$$

The metal ions in the cell can be divided into three sections; some are present as free ions in the solution, some are retained by the membrane due to rejection and/or sorption, and the rest are bound to the surfactant. A detailed discussion of this diafiltration method and procedures for qualifying its results are given by Ahmadi et al. (4). The second method discussed in that paper will be utilized to compare the binding behavior of the metals to lecithin; the moles retained due to the membrane–metal interaction are calculated at each sample interval.

Diafiltration studies require two experiments for each set of conditions: (a) in the presence of surfactant and (b) in the absence of surfactant. The binding isotherms are generated by "subtracting" the results of (a) from (b). In this manner the effect(s) of the membrane—whether rejection, absorption, or both—is(are) theoretically eliminated.

The differential material balance in the presence of surfactant can be expressed as

$$\frac{d(V_{\rm c}A_{\rm p} + M_{\rm m} + M_{\rm l})}{dt} = QA_{\rm r} - QA_{\rm p} \tag{2}$$

where $V_{\rm c}$ = volume of the cell

$$Q = \text{flow rate} = dV_{\rm p}/dt \tag{3}$$

and $V_{\rm p}$ = permeate volume
$A_{\rm r}$ = reservoir concentration
$M_{\rm m}$ = moles retained due to membrane–ion interaction
$M_{\rm l}$ = moles absorbed by the surfactant

Assuming a linear interaction between the membrane and the ions, and a linear interaction between the surfactant and the ions, $M_{\rm m}$ and $M_{\rm l}$ can be expressed as

$$M_{\rm m} = a_{\rm m}A_{\rm p} \tag{4}$$

$$M_{\rm l} = a_{\rm l}A_{\rm p} \tag{5}$$

where $a_{\rm m}$ and $a_{\rm l}$ are the interaction coefficients.

In the absence of surfactant, $M_{\rm l}$ is equal to zero and the material balance equation becomes

$$\frac{d(V_{\rm c}A_{\rm p} + M_{\rm m})}{dt} = QA_{\rm r} - QA_{\rm p} \tag{6}$$

After substitution for dt and further simplification, Eqs. (2) and (6) are solved for $M_{\rm m} + M_{\rm l}$ and $M_{\rm m}$, respectively:

$$M_{\rm m} + M_{\rm l} = \int_0^{V_{\rm p}} (A_{\rm r} - A_{\rm p})dV_{\rm p} - V_{\rm c}A_{\rm p} \tag{7}$$

$$M_{\rm m} = \int_0^{V_{\rm p}} (A_{\rm r} - A_{\rm p}^{s'})dV_{\rm p} - V_{\rm c}A_{\rm p}' \tag{8}$$

The prime on $A_{\rm p}'$ indicates the absence of surfactant.

The first term on the right-hand side of Eqs. (7) and (8) can be numerically integrated. Each equation is then summed from zero (or the lower limit of the integration and summation) to the final value of $V_{\rm p}$. The value for the amount of ions absorbed by the surfactant, $M_{\rm l}$, is then obtained by subtraction.

A plot of $M_{\rm l}$ versus concentration reveals the nature of the relationship between the ions and surfactant. If the behavior is linear, the interaction coefficient is equal to the slope of the line. If the y-intercept is above zero, it points to some initial irreversible absorption by the system. If the relationship is not linear, a plot of r, [(moles sorbed)/(moles of absorbent)], versus concentration passed, called a sorption isotherm, can be used to evaluate the binding behavior. A similar procedure is followed to determine the relationship between the membrane and ions.

Numerous experiments were carried out with various lecithins and varying concentrations using different membrane types with varying pore sizes. The following section discusses the results of experiments with the best lecithin (Elmpur N-1; 35.5 mM) and the most suitable membranes: RGO3, acrylanitryl (3000 MWCO) for experiments with mixture of metals; and 0-VF, vinylidine flouride (3000–5000 MWCO) for experiments with individual metals. The operating parameters were optimized with a pressure of 690 kPa (100 psi), a temperature of 30°C, the pH of the reservoir and cell (unadjusted) at approximately 6, and a rate of cell agitation of 200 ± 50 rpm.

RESULTS AND DISCUSSION

Diafiltration

In our diafiltration experiments, a reservoir was attached to the stirred cell (Fig. 4). The volume of the solution in the cell was set at 0.050 L; the procedure called for the collected volume to be at least three times that of the cell volume because this translates into a 95% solute exchange. If five times the cell volume was collected, the solute exchange became 99%.

Experiments were performed to study the binding of each metal individually and in a mixture with other metals. All experiments were carried out until 0.150 L permeate was collected. The feed concentration for each metal was set at the same level for all of the experiments. The sorption isotherms are plots of r, the amount of metal sorbed divided by the total amount of lecithin, versus the permeate concentration (concentration passed). The smoothness of the curves is due to the use of polynomial regressions in the calculations. The terms used here, such as weak and strong binding, are relative qualifications of the sorption behaviors that were investigated.

Lead exhibited no detectable binding to the lecithin used here, whether individually or in a mixture of metals.

Figure 5 compares the isotherms for *nickel* (denotes only nickel in the feed) and *nickel 5* (denotes five metals in the feed). *Nickel* has a constant sorption from about 0.70 to 3.0 mM. *Nickel 5* exhibits a weak binding from 0.15 mM up to about 0.70 mM. From this point until about 1.25 mM there is no binding. Beyond this point the curve suggests more binding. However, this is most likely due to precipitation and the interaction with

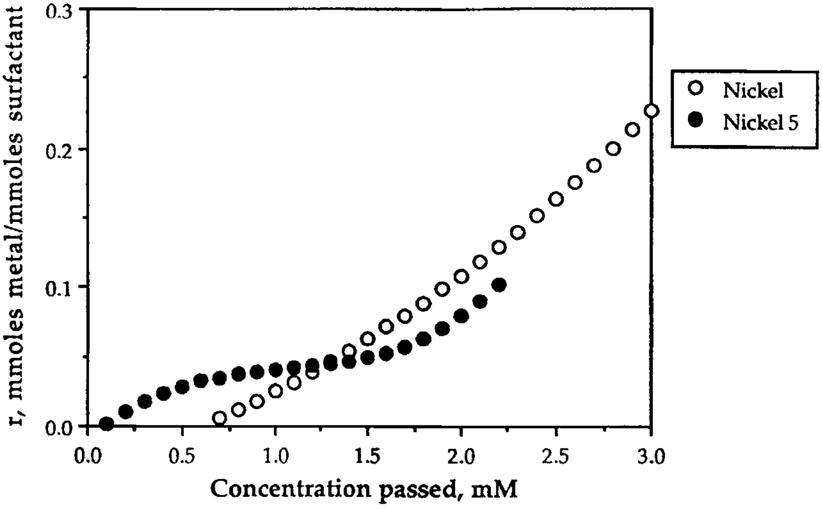

FIG. 5 Sorption isotherms: nickel and nickel 5.

the other metals, for there was some precipitate on the membrane at the end of the experiment.

Figure 6 compares the sorption of cadmium. Both curves follow the same general trend; strong binding at the beginning which slowly tapers off. *Cadmium 5* exhibits stronger binding than *Cadmium* and indicates some desorption after the passage of 2.0 mM.

The sorption isotherms for copper are compared in Fig. 7. *Copper 5*, which has a stronger sorption than *copper*, exhibits some binding from 0.0 mM up to about 0.60 mM. From this point until approximately 1.8 mM there is no binding. Beyond this point the curve indicates more sorption which is, as in the case with nickel, most likely due to precipitation and interaction between the metals. *Copper* shows a stronger binding from about 0.40 mM up to 1.1 mM than that of the rest of its curve.

Figure 8 compares the isotherms for zinc. *Zinc 5*, which has the stronger sorption of the two curves, exhibits a strong binding from 0.0 mM up to about 1.5 mM. Beyond this point the curve suggests a desorption behavior which continues until the passage of 4.5 mM. From this point until 5.0 mM there is no more binding. *Zinc* shows a continuous binding which becomes weaker after the passage of about 2.0 mM.

Figure 9 compares the sorption isotherms for the metals when present in a mixture. It is interesting to note that the curves for zinc and cadmium are very close to each other, and that the ones for copper and nickel, although shifted, also resemble one another closely.

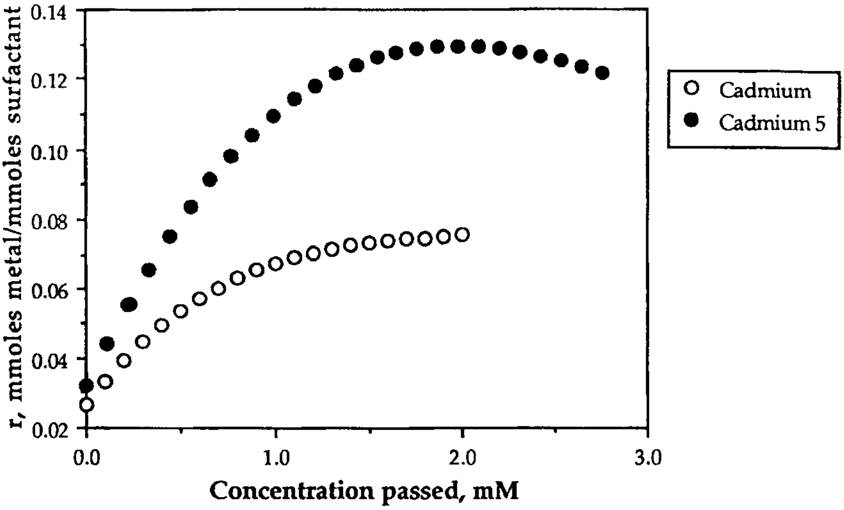

FIG. 6 Sorption isotherms: cadmium and cadmium 5.

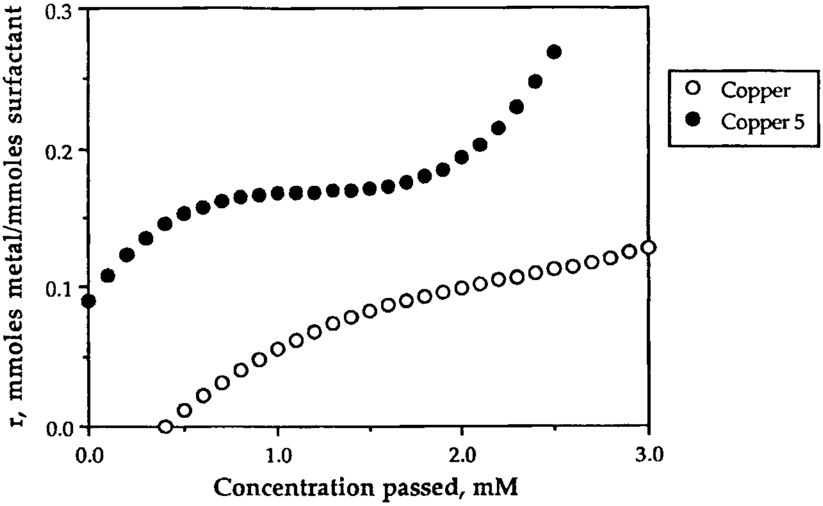

FIG. 7 Sorption isotherms: copper and copper 5.

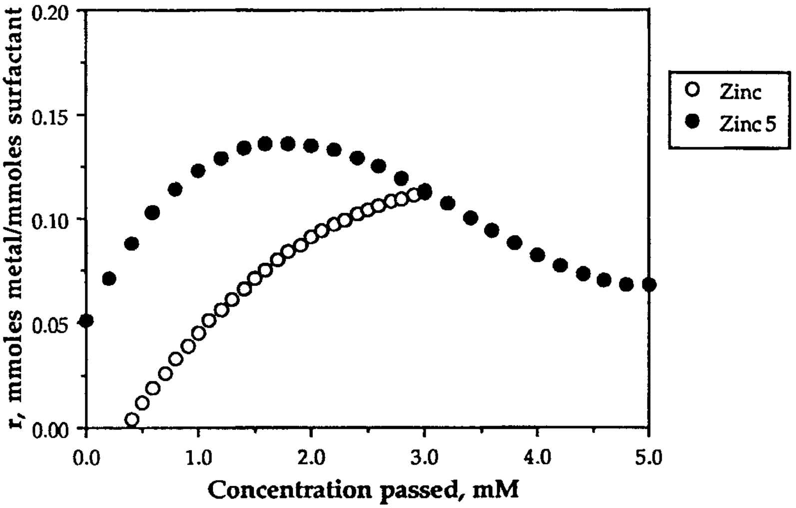

FIG. 8 Sorption isotherms: zinc and zinc 5.

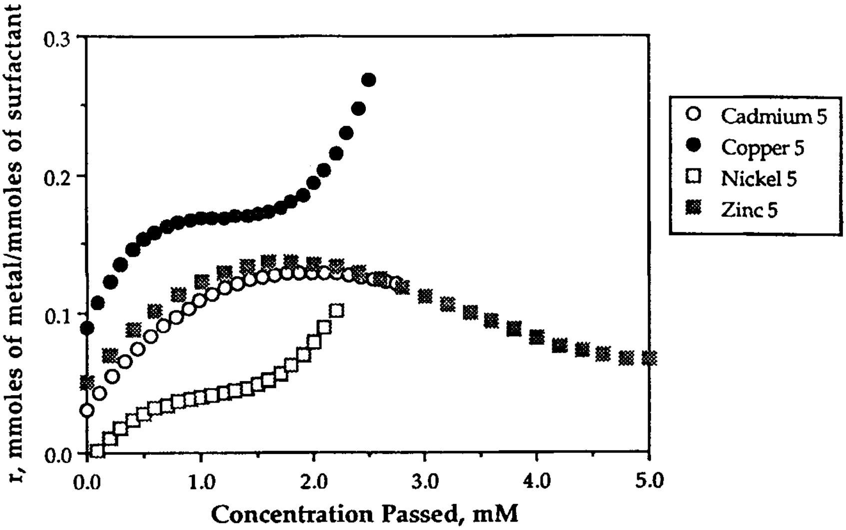

FIG. 9 Sorption isotherms: cadmium 5, copper 5, nickel 5, and zinc 5.

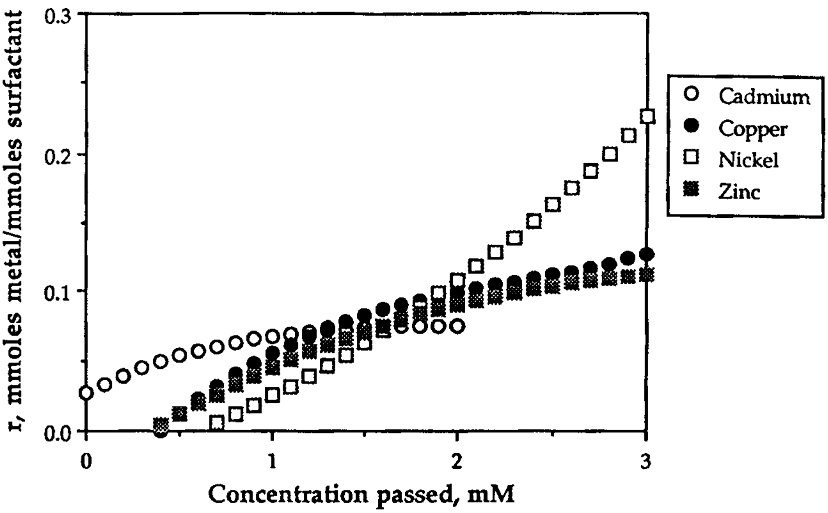

FIG. 10 Sorption isotherms: cadmium, copper, nickel, and zinc.

The isotherms for the metals when present individually are compared in Fig. 10. The isotherms are generally bunched together, with cadmium being the weakest and nickel being the strongest, and suggesting the potential for more binding beyond 3.0 mM.

SUMMARY

Micellar-enhanced ultrafiltration is a promising method for the treatment of heavy metals. It does not involve a phase change and is generally less expensive than conventional treatment techniques. With the use of a natural, inexpensive surfactant, the cost can be further reduced and no secondary toxic compound is added to the wastestream.

Continuous diafiltration is an excellent method for qualifying the binding behavior of surfactants to metal ions; an entire isotherm can be generated from a single experiment.

Lecithin exhibits some binding behavior in relation to cadmium, copper, nickel, and zinc. The binding is affected by the nature of the wastestream; competitive binding and precipitation may occur in the presence of more than one metal. Lecithin can be used as a relatively cheap pretreatment MEUF step prior to the use of more expensive and effective synthetic surfactants.

FUTURE WORK

Synthetic surfactants, such as SDS, will be studied using the diafiltration method in order to provide for an accurate comparison. Pilot-plant trials will be performed using synthetic and natural surfactants to test the predicted binding behavior shown in this work.

LIST OF VARIABLES

A_p	permeate concentration
A_r	reservoir concentration
a_l	surfactant–metal interaction coefficient
a_m	membrane–metal interaction coefficient
M_l	amount sorbed by lecithin
M_m	amount sorbed and/or rejected by membrane
Q	flow rate
r	(moles sorbed)/(moles of absorbed)
V_c	ultrafiltration cell volume
V_p	permeate volume

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