

# Improvement in removal of Pb(II) using surfactant emulsion membrane from PCB wastewater by addition of NaCl



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## ABSTRACT

The present manuscript discusses the removal of Pb(II) from printed circuit board (PCB) wastewater by using surfactant emulsion membrane (SEM) in the presence of NaCl. This SEM was prepared by stirring together the strip phase (sulphuric acid) and organic phase (surfactant: Span 80, extractant: D2EHPA, diluent: *n*-Hexane) using high speed homogenizer. The feed phase consisting of various metal ions (Na, Ca, Mg, Co, Zn, Mn, W and Pb) was treated with emulsion phase by maintaining a treat ratio 1:10. The parameters such as the extractant concentration, internal phase concentration and contact time were studied and optimized. Further, the effect of addition of NaCl salt to the feed phase was considered using the optimized parameters. A significant improvement of extraction efficiency of 99% was observed in the presence of NaCl with more than 2.5 times enrichment as compared to 82% extraction with 1 times enrichment in the absence of salt. Thus, the NaCl addition has a remarkable effect on the selective removal of Pb(II) from wastewater.

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## 1. Introduction

The emission of wastewater containing Pb(II) in to the environment by several industries such as steel industry, electroplating industry, fuel production, electrical industries and explosive manufacturers is abundantly increasing [1]. When the concentration of these heavy metals exceeds the limits set by WHO and USEPA (0.01 ppm) [2], it affects the surrounding environment adversely and it can lead to several health disorders such as anaemia, kidney damage and disorder in the nervous system [3]. Various methods for the treatment of effluents involving lead as major pollutant have being reported; including chemical precipitation [4], adsorption [5], ion exchange [6] and solvent extraction [7]. However these conventional separation processes, used for many years, bear certain limitations; for example, the solvent extraction process includes the need for dispersion and coalescence, problems of emulsifica-

tion, flooding and loading limits in continuous counter current devices, the need for density differences between the phases, phase disengagement difficulties, use of a scrubber, high solvent losses, use of toxic volatile solvents and large solvent inventories. The ion exchange process presents the problem of resin fouling, the requirement of excess complexing material, organic contamination from resin and selectivity. Chemical precipitation results in sludges that necessitate disposal in landfills. Moreover, these separation processes are equilibrium-limited. To overcome these disadvantages an emerging surfactant emulsion membrane (SEM) technique has been adopted for treatment of lead containing PCB effluent. SEMs offers ease of operation, low cost, high selectivity, large interfacial area for mass transfer, and high fluxes. It is an effective method for separation and concentration of solute when the target component being extracted is present at very low concentrations. On the contrary, solvent extraction isotherms are not feasible for very dilute solutions [8]. SEMs remove equilibrium limitations of solvent extraction by combining extraction and stripping in a single operation. SEMs have been successfully used to treat aqueous streams contaminated with heavy metal ions like cobalt, copper, zinc, cadmium, nickel, mercury, lead, and chromium [9,10]. Apart from it have successful application for instance, the separation of hydrocarbons [11], amino acid [12] and biological compound [13].

SEM has also been employed commercially on industrial scale for the removal of zinc at a textile plant in Austria; cyanide

*Abbreviation:* SEM, surfactant emulsion membrane; PCB, printed circuit board; DPP, direct plating process; WHO, World Health Organization; USEPA, United States Environmental Protection Agency; PPM, parts-per millions; SPAN 80, surfactant, sorbitan monooleate; TOC, total organic compound; dia, diameter; rpm, rotation per minutes; w/o/w, water-oil-water; D2EHPA, di-2-ethylhexyl phosphoric acid; MPa S, millipascal-second; min, minutes; M, molar.

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removal from waste liquors in gold processing plant in China and removal of phenol from wastewater [14]. The SEM process contains a three-phase dispersion system, which consists of a stripping phase (aqueous phase) encapsulated by a membrane phase (organic phase). The membrane phase contains the extractant (carrier) and surfactant in organic diluents. The role of surfactant is to stabilize the emulsion droplet. The dispersion of the three phases is achieved using high speed homogenizers. The metal ions present in aqueous solution (feed phase) forms a complex with the extractant (carrier) at the interface of the emulsion globule and the feed phase. The complex formed is then shuttled through the organic phase to the organic phase-stripping phase interface from where it is stripped into the bulk of the encapsulated stripping phase. The metal ion is recovered by breaking the emulsion [15].

In the present study, the feed phase was taken as wastewater from the printed circuit board (PCB) or e-waste recycling unit [16]. The effluent was characterized by using ion chromatography instrument. It was found to contain various metal ions such as Mn, Zn, Co, Ca, Na, Mg, W and Pb. Amongst them, the concentration of W(VI) and Pb(II) was significant viz. 600 and 150 ppm, respectively whereas the concentration of other co-metals in the effluent stream was about 5 ppm. SEM process was designed by taking suitable and appropriate amount of extractant, surfactant and stripping agent. Several important parameters of SEM were investigated to measure NaCl effect (salting out effect) on extraction and swelling. Efforts were made in achieving selective removal of Pb(II) from the wastewater without making any substantial changes to its composition.

## 2. Experimental

### 2.1. Materials and methods

The effluent wastewater containing Pb(II) was obtained from PCB recycling unit industries. Surfactant, sorbitan monooleate (SPAN 80) and extractant, D2EHPA, were purchased from Sigma-Aldrich. All other chemicals used in the process were of AR grade and used without any further purification. The surfactant emulsion membrane was developed by a Wise Tis (HG-15D) homogenizer (2000–27,000 rpm), and the speed was adjusted with a digital stroboscope (Lutron DT-2249). The internal particle size of the emulsion was measured by particle size analyzer (Nanophox, 219F-2009, Germany). The pH of the feed phase samples was measured using pH meter (HI 2211 pH/ORP Meter). The recovery of metal from feed phase was performed using a Heidolph RZR 2012 overhead stirrer (200–2000 rpm). The concentration of Pb(II) and other co-ions in the sample were analyzed by Ion Chromatography (Metrohm, 883 Basic IC Plus, Switzerland). The viscosity of the emulsion was measured with Ostwald viscometer (Viscotech Hispania S.L., IP-20). The total organic content of wastewater was measured by using SHIMADZU, TOC-L series analyzers having range 4 µg/L–30,000 mg/L.

### 2.2. Wastewater characterization

The PCB wastewater samples were obtained from PCB recycling industry outlet. In this wastewater, the total organic content was found to be 4.73 mg/L. The wastewater was filtered and the filtrate was stored at 10 °C. The pH of the entire filtrate was found to be around 5. The samples were filtered prior to analysis performed using ion chromatograph. The PCB wastewater was found to contain anionic, cationic and transition metal ions including Pb(II) and the results are discussed in detail in Table 1. The dissolved solutes and their concentrations in wastewater (in ppm) are

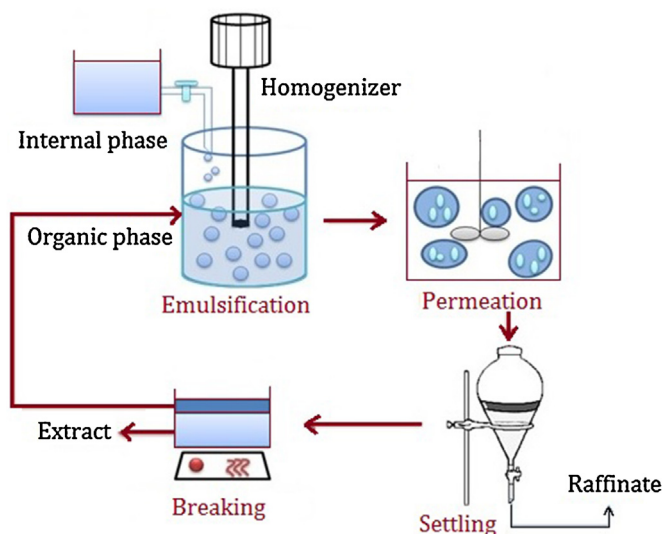


Fig. 1. A schematic of surfactant emulsion membrane (SEM) process.

Mn(II) = 1, Zn(II) = 1, Co(II) = 1, Ca(II) = 5, Mg(II) = 4, W(VI) = 600 and Pb(II) = 150.

### 2.3. Development of SEM

The SEM was developed by using organic diluents (*n*-Hexane) containing surfactant Span 80 (3% w/w) and extractant D2EHPA (0.06 M) along with an aqueous stripping agent (0.1–1 M H<sub>2</sub>SO<sub>4</sub>). The organic and the aqueous phase were maintained in 1:1 ratio. The mixture was stirred using high speed homogenizer at 6000 rpm for 15 min. This resulted in the formation of water-in-oil (w/o) emulsion with milky white appearance [17]. The internal globule size of the prepared emulsion was found to be in the range of 4–8 µm. The most stable emulsion was obtained when there was neither swelling nor breakage during the extraction run. A schematic of the ELM process constituting emulsion preparation, permeation of Pb(II) and breaking of the emulsion are depicted in Fig. 1. The emulsion swelling, extraction and enrichment were measured by using Formula (1)–(3) respectively.

$$\text{Swelling (\%)} = \frac{V_{i,t} - V_{i,0}}{V_{i,0}} \times 100 \quad (1)$$

$$\text{Extraction (\%)} = \frac{C_i - C_f}{C_i} \times 100 \quad (2)$$

$$\text{Enrichment} = \frac{C_{sp}}{C_{fp}} \quad (3)$$

where  $V_{i,0}$  and  $V_{i,t}$  are the initial and at time 't' internal phase volumes,

$C_i$  and  $C_f$  are initial and final concentration of solute contained in feed phase and

$C_{sp}$  and  $C_{fp}$  are concentrations of strip phase and feed respectively.

### 2.4. Removal of Pb(II)

The prepared emulsion was stirred with feed phase by maintaining a treat ratio of 1:10 for 6 min at 300 rpm [17]. In the feed phase, NaCl was added in different quantities (3–30 g/L). The analysis of the extracted metal ions was concluded by taking samples at different intervals from raffinate phase. Finally, the loaded emulsion was broken down by heating at 80 °C for 1 h in a closed vessel for the recovery of strip phase containing Pb(II) ions. The extraction

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