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## **Emulsion liquid membrane for lead removal: intensified low shear extraction**

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**Abstract:** The paper provides a technological innovation process to improve lead removal under low shear counter rotating Taylor-Couette column (TCC). Emulsion was prepared using TOA, Span 80, kerosene, and ammonia. Feed solution composed of lead nitrate in deionised water and HCl. The emulsion and feed solution were then contacted in TCC at various conditions including HCl concentration, initial feed concentration, volume ratio of emulsion to feed phase, extraction time, and ratio of angular frequency of outer and inner cylinders. The results showed that the system was effective for lead removal. The best extraction performance was obtained at HCl concentration of 0.1 M, initial feed concentration of 40 ppm, volume ratio of emulsion to feed phase of 1/5, extraction time of 7 min, and angular frequency ratio of 0.5. Overall, the use of low-shear TCC process intensified the mixing of emulsion and feed phases well that avoiding emulsion instability during the process.

**Keywords:** emulsion liquid membrane; lead removal; Taylor-Couette column; TCC; intensification; low shear extraction; novel extraction technology; innovation.

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## 1 Introduction

Textile industry is among the major industries in the developing country (Hussain et al., 2004; Kim et al., 2006; Tuzen et al., 2008). It was predicted that the export of textile and textile products in Indonesia would improve in about 11% per year. Based on data of Ministry of Industry of Indonesia, in 2017, the national number of small and medium sized enterprises were about 3.4 million in which the textile industries were 680,000. About 20% of textile industries or 136,000 were batik industry. The big number of batik industries was available in some areas such as Pekalongan, Yogyakarta, Solo, Cirebon, and Lasem. The batik industry has been successfully hired about 600,000 labours, nationally.

In spite of the economics contribution of batik industries, the environmental negative effects resulted by wastewater effluents must be very well considered. Majority of textile

home industries do not have their own wastewater treatment installation. Therefore they have to use shared wastewater treatment installation with limited capacity. Accordingly, many of them discharge textile industrial wastewater directly to the water body. Whereas, batik industry consumes large volume of water, in which 80–100 m<sup>3</sup> is needed per ton textile products. National production of batik in 2015 was 5,745 tonnes released 2,872 m<sup>3</sup> wastewater (Wang et al., 2011). Batik industry as the part of printing and dyeing processes contributes the main pollution in the textile industry. Batik production processes applied high ranges of chemicals, including dyestuffs (Savin and Butnaru, 2008). In Pekalongan, Indonesia, about 99.4% of home industries directly disposed the wastewater to the environment without any prior treatment due to the unavailability of wastewater treatment installation (Nindita et al., 2012).

It is therefore, textile effluents composed of textile dyes, solvents, total dissolved solids, chemical and biological oxygen demand and heavy metals. The major heavy metals available in textile wastewater are copper, zinc, lead, cadmium, chromium, and nickel (Imtiazuddin et al., 2012; Jaishree and Khan, 2014; Mekuyie, 2014). A research of Zille (2005) found that textile dyes such as acid dye, basic dye, direct dye, disperse dye, and vat dye contain lead in the components. Hussain et al. (2004) revealed that textile wastewater contained lead in the concentration of 11–61 mg/l. The presence of this metal might come from textile dyes, chemical agents, or the iron pipes used in the disposal. Agustina and Badewasta (2009) showed that Pb content in batik industrial wastewater was 0.2349 mg/l, higher than the maximum allowable limit, i.e., 0.03 mg/l.

The wastewater which is released in the water body without any prior treatment potentially pollutes groundwater. The disposed of textile wastewater composed of heavy metals could permeate into groundwater. Some researches on groundwater pollution by batik industrial wastewater have been done (Purwanti, 2004; Rizza, 2013; Yasser et al., 2016). The impact of industrial wastewater pollution on the quality of recipient water bodies variation depend on the nature and type of waste, volume and frequency of wastewater discharged. The presence of batik industries that grow in residential areas increases the amount of waste disposal which potentially contaminates groundwater. Well construction and water retrieval methods can also be a source of contamination. If the well construction does not meet the standard requirements, chances of contamination of the well water will increase. Dug wells used in a relatively long time have higher possibility of contamination due to the increase of pollution sources. Considering the harmful effects of heavy metals to environment and living organism, the removal is a great challenge.

Many methods have been applied to remove lead from industrial liquid waste. Researches on lead removal by coagulation has been done by Mukherjee et al. (2018). Lead removals have also been studied by adsorption (Bai et al., 2017; Ho et al., 2017; Bhunia et al., 2018; Fang et al., 2018; Shi et al., 2018). Oke et al. (2017) studied lead removal using electrochemical method. Lead extractions from wastewater have been done by Shah et al. (1995) and Moon and Yoo (2017). An alternative method of solvent extraction which combines extraction and stripping processes in a single step could minimise both equipment and process time. Emulsion liquid membrane (ELM) involved the mixing of double emulsion, in which water in oil emulsion is poured into feed solution. The feed solution contains solute to be extracted; it will diffuse through oil membrane phase into stripping internal phase. ELM has been widely used in lead

extraction (Ahmad et al., 2013; Kassem et al., 2013; Ahmad et al., 2017; Zabat, 2017; Mesli and Belkhouche, 2018), however, this method is facing problem of emulsion instability. The formed emulsion tends to suffer from high shear during extraction process. As a result, the emulsion will break, leading to the release of the entrapped solutes. Overcoming the emulsion instability problem by decreasing the shear which commonly done by decreasing extraction speed resulted in the decrease of extraction efficiency. On the other hand, increasing emulsion stability by adding surfactant concentration will inhibit mass transfer process.

To optimise lead extraction process, a comprehensive developed ELM method has been used. In order to remove lead from liquid waste extensively, a counter rotating TCC was applied as the novel extraction technology for ELM application. Taylor-Couette column (TCC) intensively extracted lead under low shear mixing. TCC provides larger contact area since extraction occurs along the column (Ahmad et al., 2014). While low shear mixing almost nullify emulsion breakage. This technology is expected to be a solution to improve the performance of textile industry in addressing waste problems. In this work, effect of extraction conditions to extraction efficiency was studied.

## **2 Methodology**

### *2.1 Materials*

The aqueous lead solutions were prepared by dissolving lead nitrate (Merck) in deionised water. HCl (Merck) was added to the feed solution to adjust the pH. Lead and HCl concentrations were varied to see their effect to extraction performance. Trioctylamine (Merck) and span 80 (Merck) were used as extractant and surfactant, respectively. Low odour kerosene purchased from Sigma Aldrich was used as diluent. Ammonia (Merck) was used as internal phase solution.

### *2.2 Experiment procedure*

The emulsion was prepared using ultrasonic probe (Ahmad et al., 2012) then poured into the lead solution and rotated for lead extraction. Experiment under TCC was done as given in previous research (Ahmad et al., 2014) by following certain variable in extraction conditions. Volume ratio of emulsion to feed phase was varied to obtain the optimal yield. Rotation speed was also varied to reach the best yield with the minimum emulsion instability. Extraction was carried out for ½, 1, 3, 5, 7, 10, 15, 20, 25, and 35 min, sample was taken for every predetermined time. To investigate the effect of initial feed concentration, the concentration was varied in 10, 40, 70, and 100 mg/l. After extraction, the mixed solution was let for settling for 5 minutes. The lower layer solution was then taken sampled to check the lead concentration in the feed phase.

### *2.3 Analytical equipment*

pH of aqueous solutions was measured by using Fisher scientific accumet AB15 pH meter. The concentration of remaining lead in the raffinate phase and strip solution was determined spectrophotometrically using atomic absorption spectroscopy.

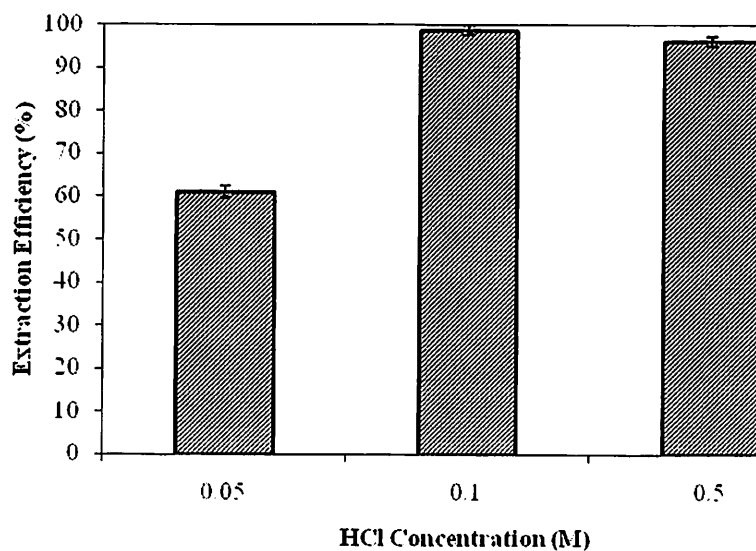
### 3 Results and discussion

It has been known that emulsion capability could be optimised in the proper solution pH thus the first parameter to be studied was HCl concentration. The optimum value was employed for subsequent experimental studies. This process was conducted to each parameter.

#### 3.1 Effect of HCl concentration in feed solution

Lead removal using ELM system was governed by many parameters. Many factors govern metal removal using ELM system. In this research, lead ion was carried by TOA as basic extractant. The strong metal-carrier complex could be formed in proper solution pH. Suitable pH solution also contributes in accelerating diffusion process since it occurs by the pH difference of external and internal phase solution. In the present research, investigation was done at HCl concentration of 0.05 M, 0.1 M, and 0.5 M to give driving force for lead extraction while internal phase concentration was kept constant at 0.1 M ammonia solution. Figure 1 describes the performance of lead extraction under different HCl concentration. It could be seen that extraction efficiency increased as the increase of HCl concentration from 0.05 M to 0.1 M. However further increase of HCl concentration beyond 0.1 M resulted in significant decrease of extraction efficiency. This is may be due to beyond 0.1 M will form another species which could not ionise completely thus unable to form complex with TOA (Kumbasar, 2009). As a result, lower extraction rate was obtained.

Figure 1 Effect of HCl concentration on extraction efficiency



Low extraction efficiency of about 60% was found at HCl concentration of 0.05 M, due to the limited pH difference. When most of lead had been transferred into internal phase there was not enough driving force to continue the mass transfer process therefore the

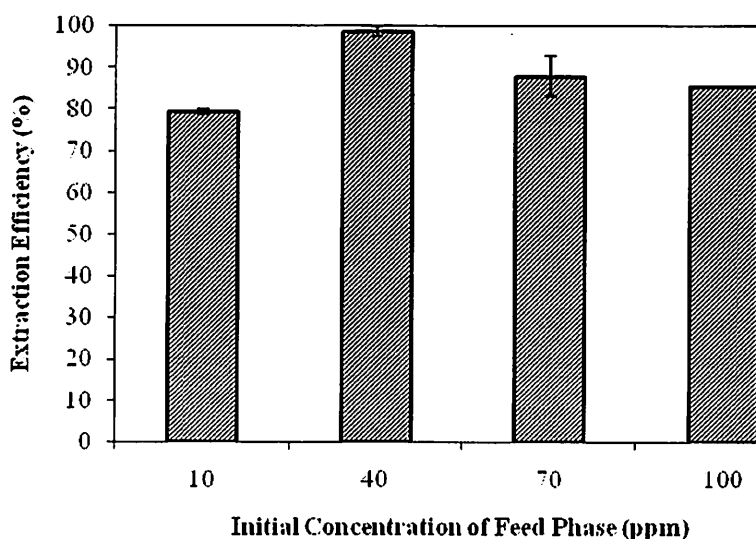
difference concentration of  $H^+$  in external and internal phase was inevitable. Further increase in HCl concentration to 0.5 M decreased extraction efficiency and it turn increasing emulsion swelling (Wan and Zhang, 2002) and enhanced the risk of lead back diffusion to external phase.

### 3.2 Effect of initial feed concentration

Chemical potential due to concentration difference between feed and internal phase impose a significant driving force for diffusion process. Extraction efficiency of lead was studied at initial feed concentrations of 10 ppm, 40 ppm, 70 ppm, and 100 ppm to make it similar with that of the real wastewater. Other parameters included concentration of carrier and internal phase were maintained constant. Both concentrations were enough to react with the highest lead concentration of 100 ppm. The effect of initial feed concentration on extraction efficiency is shown in Figure 2.

As shown in the figure, extraction efficiency increased with an increase of initial feed concentration due to higher chemical potential between feed and internal phase. The highest extraction efficiency of 99% was achieved by system with initial concentration of 40 ppm. Beyond that concentration, extraction efficiency slightly decreased to be about 88%. At the beginning of extraction process, most of lead has diffused into internal phase, occupying the outer portion of emulsion. Since the outer layer has been fulfilled, the rest of lead must diffuse more deeply inside the emulsion globule. It meant that the latter complex pursued longer diffusional path (Sengupta et al., 2006; Kumbasar, 2008) which in turn reduced the extraction rate. In the study done by Basualto et al. (2006) the amount of carrier was not enough to transport larger quantity of the metal hence decreased extraction efficiency. Mathematically, Datta et al. (2003) revealed that higher initial concentration is inversely proportional to removal percentage that might be detrimental to extraction process, especially at limited concentration of carrier.

**Figure 2** Effect of initial concentration on extraction efficiency

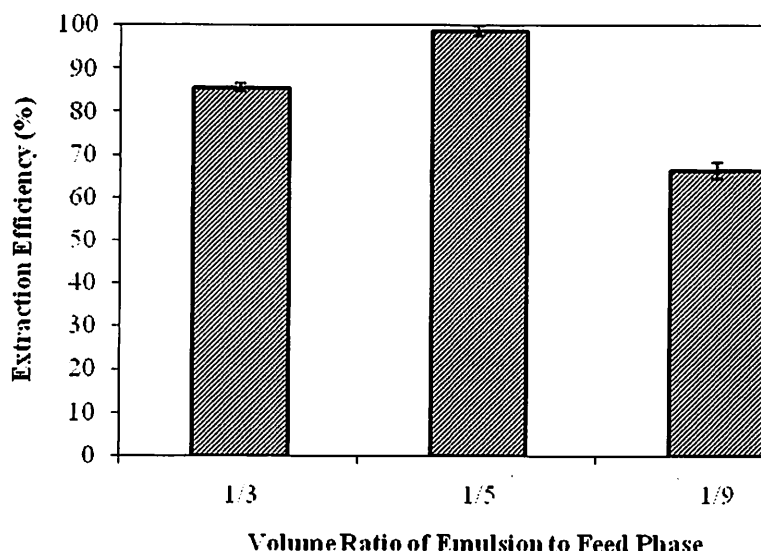


### 3.3 Effect of volume ratio of emulsion to feed phase

Another operating condition investigated in this study was the effect of volume ratio of emulsion to feed phase. Observation was done at ratio of 1/3, 1/5, and 1/9. Diffusion process and extraction rate are related to this treatment ratio. Figure 3 shows lead extraction efficiency as a function of volume ratio of emulsion to feed phase.

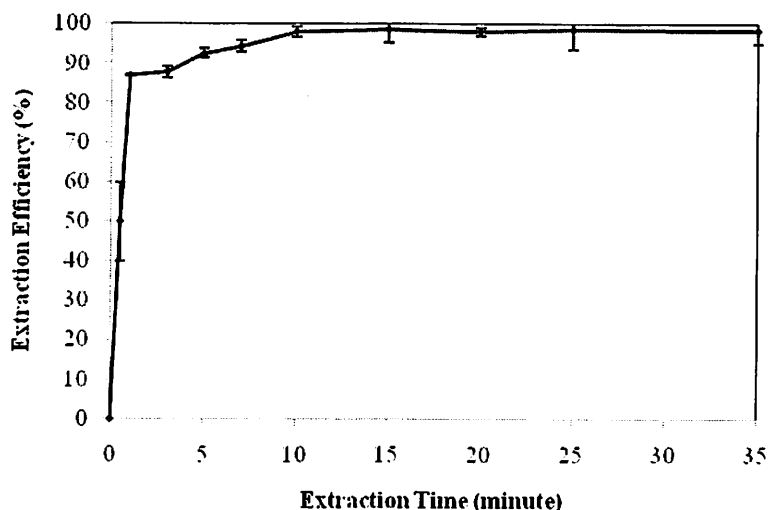
As observed from Figure 3, lead removal increased with the decrease of treatment ratio. The increase of extraction efficiency by decreasing treatment ratio from 1/3 to 1/5 could be explained that at treatment ratio of 1/3, the relative high amount of emulsion could increase the solution viscosity and therefore lowering the mixing intensity between the emulsion and the feed phases. By decreasing treatment ratio to 1/5, solution viscosity will decrease that can increase the amount of emulsion globules available for lead per unit volume of reaction mixture. Thus, the interfacial surface area for mass transfer increased, thereby increasing the rate of mass transfer from feed to emulsion globule. The increase of extraction rate was also driven by larger amount of carrier as well as internal phase solution in emulsion.

Figure 3 Effect of volume ratio of emulsion to feed phase on extraction efficiency



### 3.4 Effect of extraction time

Investigation on effect of extraction time to extraction efficiency was done at time variation of 1/2, 1, 3, 5, 7, 10, 15, 20, 25, and 35 min. Figure 4 depicts that extraction process occurred very fast, within 1 min almost 90% of lead could be extracted from external feed phase solution. After 1 min, extraction efficiency seems to be plateau. Slight increment was found at the end of extraction process of about 99%. The remaining complex of lead and TOA was too low, the concentration gradient was not enough to drive the complex diffused into internal phase.

**Figure 4** Effect of extraction time on extraction efficiency

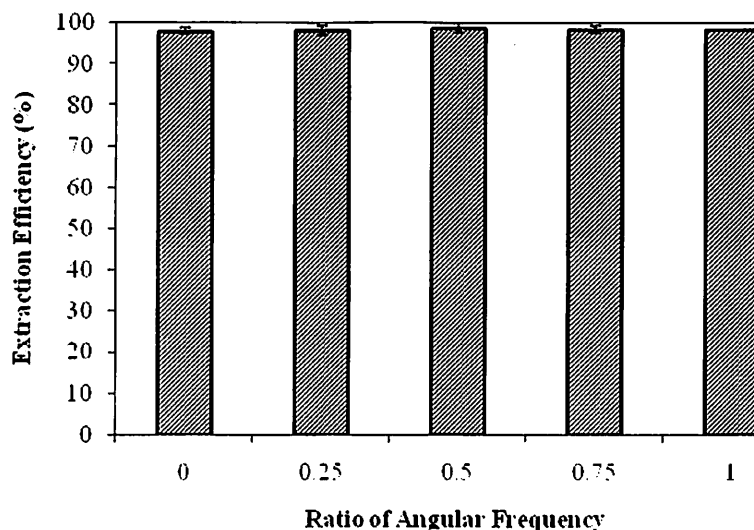
### 3.5 Effect of ratio of angular frequency

Effect of ratio of angular frequency of outer and inner cylinders on extraction efficiency was investigated at 0, 0.25, 0.5, 0.75, and 1. As shown in Figure 5, the extraction efficiency was almost the same at any ratio of angular frequency. This is due to in TCC, mass transfer process occurs along the column, unlike that in stirred vessel in which extraction only conducts around the stirrer. The difference performance of rotational speed is in extraction time. System done in rotational speed ratio of 0, i.e., system in which the outer cylinder rotation was zero, reached maximum extraction efficiency in 35 minutes. While at ratio 0.25 and 0.5, maximum extraction efficiencies were obtained in 25 and 10 minutes, respectively. Maximum extraction efficiency of ratio 0.75 and 1 were reached within similar time of 7 minutes.

**Table 1** Comparison of current study with similar works in ELM application

<i>Method</i>	<i>Extraction process</i>	<i>Result</i>	<i>Ref</i>
Emulsion liquid membrane	Counter rotating TCC	>99%	Current study
Emulsion liquid membrane	Stirred vessel using ionic liquid membrane	>97%	Lende et al. (2014)
Emulsion liquid membrane	Stirred vessel using ionic liquid membrane	~82.61%	Mesli and Belkhouche (2018)
Emulsion liquid membrane	Stirred vessel using emulsion liquid membrane	~96.67%	Fouad et al. (2017)
Bulk liquid membrane	Electrodialysis and electrodeposition	93%	Sadyrbaeva (2017)
Emulsion liquid membrane	Stirred vessel	~98%	Gürel et al. (2005)
Emulsion liquid membrane	Stirred vessel using complexation of lead with monovacant heteropolyanion of Dawson type	90%	Zabat (2017)



**Figure 5** Effect of stirring speed on extraction efficiency

Based on the above results, in general, the use of novel extraction technology of TCC in this study helped in improving the performance of waste extraction process. This finding is very beneficial in achieving environmental-friendly industrial process of textile liquid waste treatment. Table 1 shows comparison of the findings of current study with similar works in the removal of lead under liquid membrane process. As shown, compared to stirred vessel, TCC extraction provided more promising lead extraction efficiency. The counter rotating TCC processes allow well-mixing intensity of emulsion and feed phases along the column by avoiding emulsion instability problems. This is possible because the low shear stress of counter rotating TCC could maintain emulsion stability during extraction process. The lower shear stress, the higher emulsion stability thus leading to better extraction process (Ahmad et al., 2014).

#### 4 Conclusions

Intensified low shear extraction of lead by using a counter rotating TCC was successfully investigated. The TCC processes allow well-mixing intensity of emulsion and feed phases without worrying about emulsion instability problems. The system provided high extraction efficiency of lead that is one of the most harmful heavy metals found in contaminated-groundwater. The results showed that more than 99% of lead can be removed in a very short extraction time. The optimum extraction parameters were obtained at HCl concentration of 0.1 M, volume ratio of emulsion to feed phase of 1/5, angular frequency ratio of 0.5, and initial feed concentration of 40 ppm.

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