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Reverse micelle-based extraction technique for heavy metal ion removal from wastewater

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Abstract

Heavy metal contamination in industrial wastewater presents serious environmental and health risks due to the persistence and toxicity of metals such as chromium, nickel, copper and manganese. This study investigates the use of reverse micelle-based extraction for the removal of these heavy metal ions from both synthetic and CETP wastewater. CTAB and SLS/SDS were employed to form reverse micelles for the selective extraction of metal ions. Parameters such as pH, contact time (15-45 min), surfactant concentration (10,20,30 mM) and sample volume (10-50 mL) were systematically varied. Results demonstrated significantly higher removal efficiencies in CETP wastewater, with optimal conditions around 10-15 mM surfactant concentration and 30 minutes contact time. Maximum efficiencies recorded were 99.41% for copper (SLS), 95.69% for manganese (SLS), 79.73% for nickel (SLS) and 61.84% for chromium (CTAB). The study further analyzed the feasibility of applying a 1:2 micelle solution to wastewater ratio. The finding confirm that reverse micelle extraction is an effective and scalable technique for industrial wastewater treatment, ensuring high removal efficiency while optimizing process conditions for large-scale applications. Future research should focus on refining surfactant concentration to enhance cost-effectiveness and sustainability in real-world operations.

Keywords: Reverse micelle extraction, heavy metal ions, wastewater treatment, CTAB, SLS, surfactant system, phase separation, n-Hexane, liquid-liquid extraction, scalability

1. Introduction

The contamination of industrial wastewater with heavy metals such as chromium, nickel, copper and manganese pose significant environmental and public health challenges. These metals are persistent, non-biodegradable and toxic even at low concentrations, with a strong potential for bioaccumulation in aquatic ecosystems. Conventional treatment methods, including chemical precipitation and ion exchange, often suffer from limitations such as low efficiency at trace levels, high operational costs, and the generation of large volumes of secondary waste (Mungray *et al*., 2012) [6].

Reverse micelle extraction (RME) is an advanced liquid-liquid extraction technique where surfactants from nanoscale aggregates in a nonpolar organic solvent. These reverse micelles can selectively encapsulated metal ions from the aqueous phase, aided by electrostatic interactions, chelation or ion pairing (Chaturvedi *et al*., 2022; Stevens *et al*., 2004) [3, 8]. The specificity and adaptability of this technique make it a promising solution for removing metals from complex effluents (Caselli *et al*., 2004) [4]. The high surface-area interface and tunability of surfactants provide selective and efficient removal of target metals, making RME attractive for complex wastewater matrices (Chaturvedi *et al*., 2022) [3].

Surfactant selection plays a crucial role in the success of RME. Cationic surfactants such as cetyltrimethylammonium bromide (CTAB) and anionic types like sodium lauryl sulfate (SLS) or sodium dodecyl sulfate (SDS) are commonly used due to their stable micelle-forming properties and interaction with various metal ions.

Recent studies have shown that mixed surfactant systems and the use of biosurfactant can further enhance selectivity and environmental safety in RME systems. Moreover, green solvent-based reverse micellar systems have been proposed as eco-friendly alternatives to traditional organic solvents.

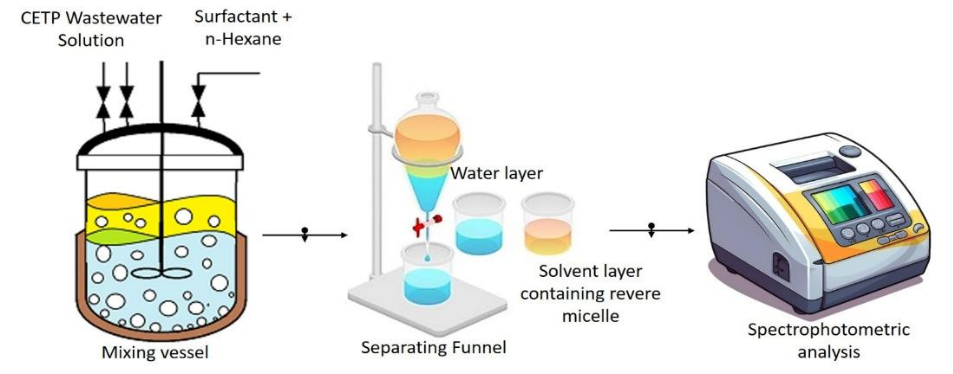
Despite its potential, RME still faces practical challenges, particularly in areas such as surfactant regeneration, mass transfer dynamics, and scale-up feasibility. To address these, the present study investigates the effectiveness of reverse micelle systems based on CTAB and SLS/SDS in extracting heavy metals from common effluent treatment plant (CETP) wastewater. The work focuses on process optimization, examining the role of pH and scalability.

2. Materials and Methods

Wastewater samples were collected from the primary screening outlet of the CETP in Surat, Gujarat. In addition to synthetic wastewater was prepared and analyzed to compare extraction behavior. Initial and final concentrations of Chromium (Cr), Nickel (Ni), Copper (Cu) and Manganese (Mn) were determined using a visible spectrophotometer (Model: EQ-826, Equiptronics). The procedures followed standard protocols prescribed by APHA (2012) [2] and Maiti (2004) [1]. The specific methods employed for each metal were: Diphenylcarbazide Method for Cr, Dimethylglyoxime Method for Cu, Neocuproine Method for Cu and Persulfate Oxidation Method for Mn.

To prepare the reverse micelle solution, surfactant solutions were first formulated by dissolving CTAB as a cationic surfactant and SLS/SDS as anionic surfactants in distilled water. These aqueous surfactant solutions were then mixed with n-Hexane to form a two-phase system. The solution was thoroughly stirred and allowed to settle until a clear phase separation was observed. The lower aqueous phase was discarded and the upper organic phase containing reverse micelles was used for further extraction.

For the extraction process, the micelle solution and wastewater (both CETP and synthetic) were mixed in a 1:2 ratio (one part micelle solution to two parts wastewater). The mixtures were stirred for varying time intervals 15,30 and 45 minutes to evaluate the effect of contact time on metal removal efficiency. After mixing, the solutions were transferred to separating funnels and allowed to settle until distinct phase separation was visible. The lower aqueous phases (treated wastewater) were collected for post-analysis, while the upper organic phases containing extracted metal ions were discarded. Post extraction metal concentrations were determined using the same spectrophotometric methods as in the initial analysis, enabling the calculation of removal efficiencies.



3. Results

This section presents the results of heavy metal ion extraction from synthetic and CETP wastewater using reverse micelle-based extraction with CTAB (cationic surfactants) and SLS (anionic surfactant). Parameters such as surfactant concentration (10,20,30 mM), contact time (15,30,45 minutes), pH were varied to evaluate their effect on extraction efficiency.

3.1 Chromium

Metals	CTAB Surfactant Concentration(mM)	Time (Minutes)	Concentration (mg/L)	Efficiency (%)
Chromium	-	0	29.48±0.17	-
	10	15	20.37±0.17	30.91
	10	30	21.81±0.19	26.00
	10	45	22.20±0.17	24.67
	20	15	18.84±0.10	36.09
	20	30	18.43±0.16	37.50
	20	45	19.63±0.14	33.42
	30	15	16.41±0.16	44.35
	30	30	19.43±0.23	34.07
	30	45	19.26±0.24	34.66

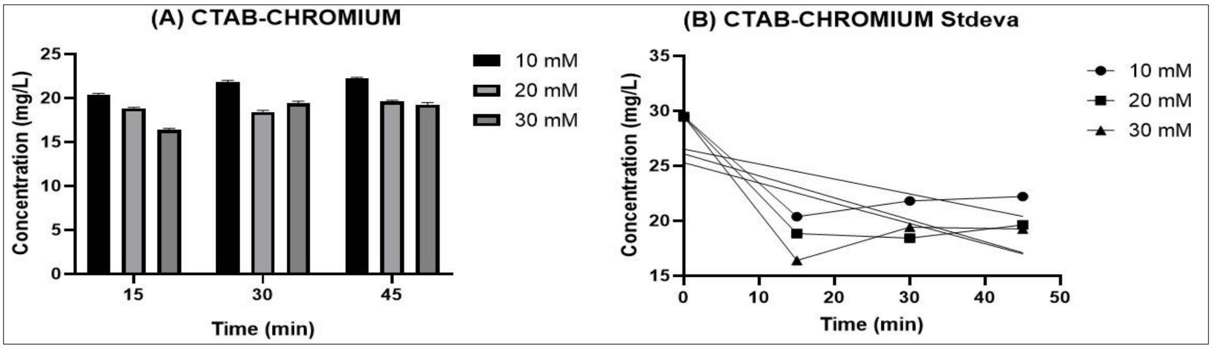


Fig 1: (A) Concentration of Chromium in wastewater pH=6.27, volume of sample 10 mL after extraction by CTAB surfactant different concentration reverse micelle solution, (B) Standard deviation graph of in Chromium concentration in different time extraction by CTAB surfactant different concentration reverse micelle solution.

Metal	SLS Surfactant Concentration(mM)	Time (Minutes)	Concentration (mg/L)	Efficiency (%)
Chromium	-	0	33.46±0.13	-
	10	15	27.06±0.16	19.16
	10	30	26.08±0.09	22.06
	10	45	29.88±0.10	10.71
	20	15	27.48±0.16	17.86
	20	30	27.68±0.09	17.29
	20	45	28.37±0.16	15.24
	30	15	17.75±0.17	46.95
	30	30	22.92±0.17	31.52
	30	45	19.69±0.10	41.14

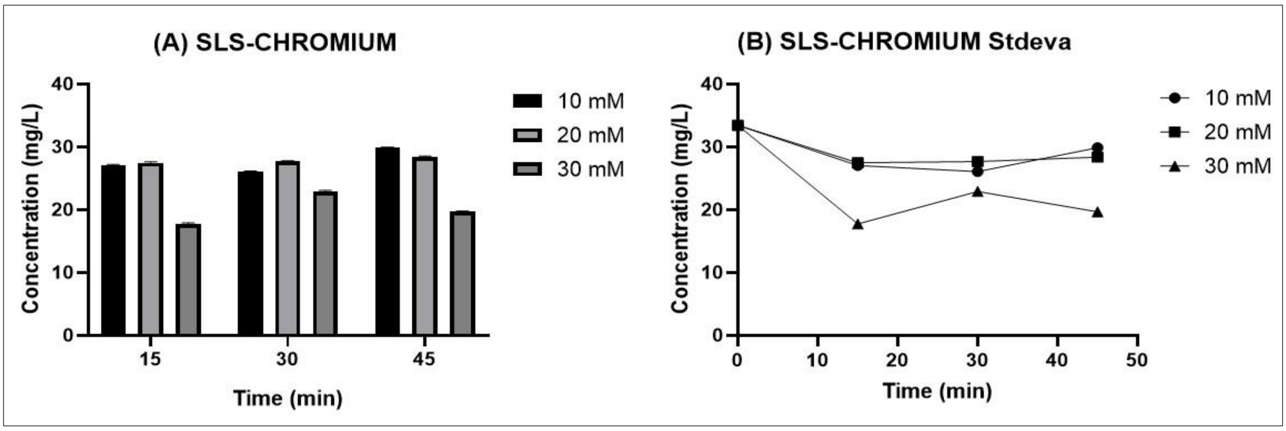


Fig 2: (A) Concentration of Chromium in Synthetic wastewater pH=6.30, volume of sample 10 mL after extraction by SLS surfactant different concentration reverse micelle solution, (B) Standard deviation graph of Chromium concentration in different time extraction by SLS surfactant different concentration reverse micelle solution.

Metal	Surfactant Concentration(mM)	Time (Minutes)	Concentration (mg/L)	Efficiency (%)
Chromium	-	0	1.52±0.04	-
	CTAB 10	15	0.95±0.05	37.50
	CTAB 10	30	0.64±0.05	57.89
	CTAB 10	45	1.31±0.07	13.82
	CTAB 15	15	0.84±0.05	44.74
	CTAB 15	30	0.58±0.05	61.84
	CTAB 15	45	1.28±0.03	15.79
	SLS 10	15	1.46±0.02	3.95
	SLS 10	30	1.49±0.05	1.97
	SLS 10	45	1.49±0.02	1.97

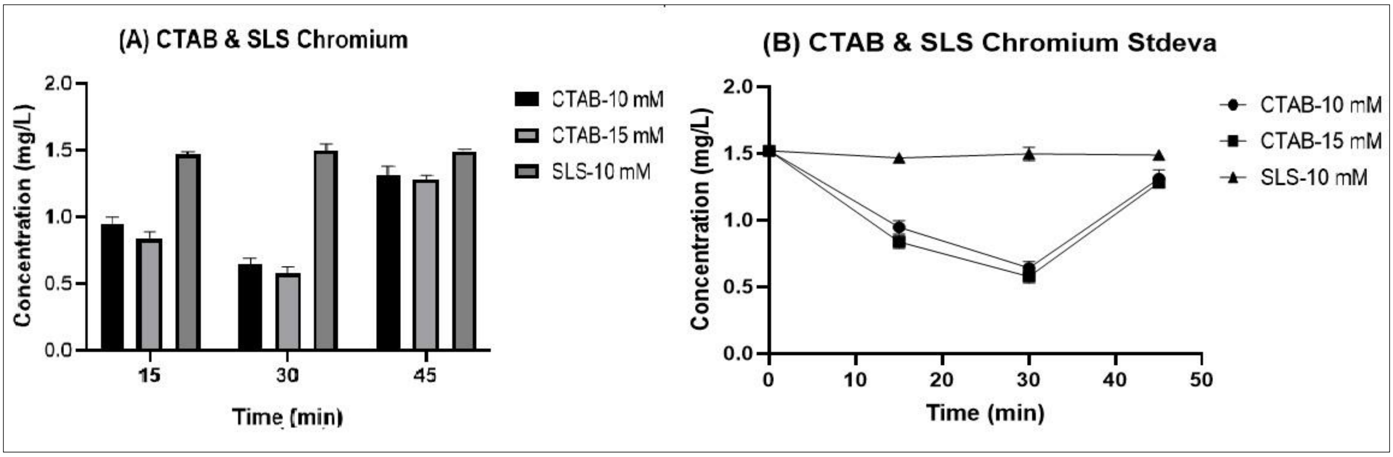


Fig 3: (A) Concentration of Chromium in CETP wastewater pH=7.22, volume of Sample 20 mL after extraction by CTAB & SLS surfactant different concentration reverse micelle solution, (B) Standard deviation graph of Chromium concentration in different time extraction by CTAB & SLS surfactant different concentration reverse micelle solution.

In synthetic wastewater, at pH 6.27 and sample volume 10 mL, the highest chromium removal efficiency using CTAB was 44.35% at 30mM and 15 minutes (Fig. 1). With SLS, at pH 6.30 the highest efficiency reached 46.95% at the same concentration and time (Fig. 2). However, in CETP wastewater, at pH 7.22 CTAB achieved up to 61.84% removal at 15 mM and 30 minutes, whereas SLS show poor removal efficiency, peaking at only 3.95% (Fig. 3).

3.2 Nickel

Metal	CTAB Surfactant Concentration(mM)	Time (Minutes)	Concentration (µg/L)	Efficiency
Nickel	-	0	83.64±0.44	-
	10	15	59.31±0.77	29.10
	10	30	69.35±0.44	17.09
	10	45	68.98±0.55	17.52
	20	15	58.36±0.66	30.24
	20	30	67.15±0.79	19.71
	20	45	69.86±0.99	16.46
	30	15	58.21±0.77	30.39
	30	30	69.93±0.77	16.38
	30	45	72.57± 0.45	13.28

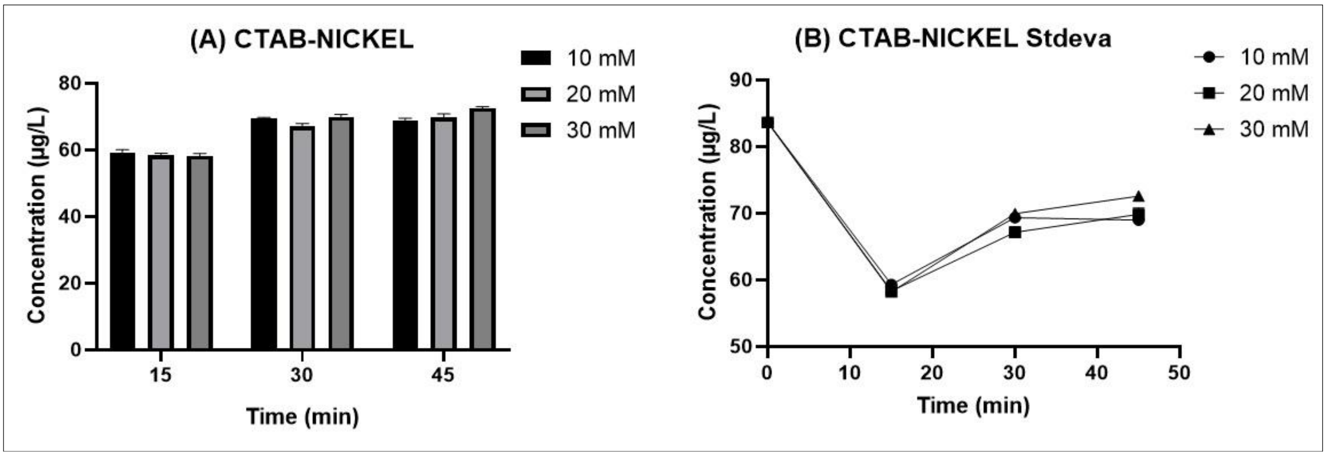


Fig 4: (A) Concentration of Nickel in wastewater pH=3.80 volume of sample 10 mL after extraction by CTAB surfactant different concentration reverse micelle solution, (B) Standard deviation graph of in Nickel concentration in different time extraction by CTAB surfactant different concentration reverse micelle solution.

Metal	SLS Surfactant Concentration(mM)	Time (Minutes)	Concentration (µg/L)	Efficiency (%)
Nickel	-	0	75.51±0.58	-
	10	15	58.73±0.55	22.20
	10	30	65.02±0.67	13.86
	10	45	59.97±0.55	20.58
	20	15	57.55±0.46	23.77
	20	30	58.94±0.55	21.92
	20	45	56.16±0.44	25.61
	30	15	59.17±0.55	21.65
	30	30	58.21±0.55	22.91
	30	45	56.82±0.79	24.78

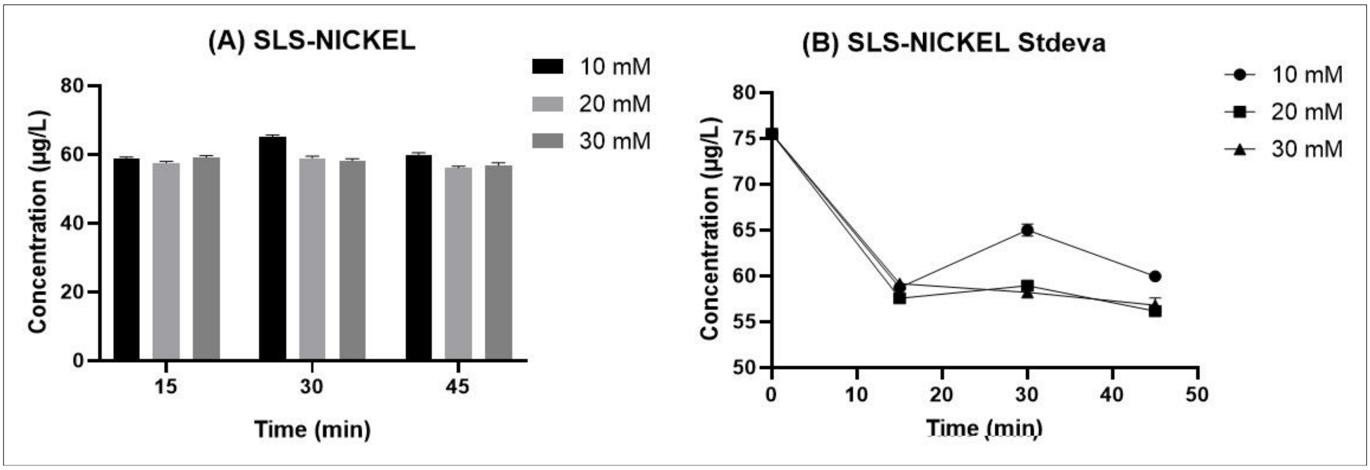


Fig 5: (A) Concentration of Nickel in wastewater pH=3.30 volume of sample 10 mL after extraction by SLS surfactant different concentration reverse micelle solution, (B) Standard deviation graph of in Nickel concentration in different time extraction by SLS surfactant different concentration reverse micelle solution.

Metal	Surfactant Concentration(mM)	Time (Minutes)	Concentration (µg/L)	Efficiency (%)
Nickel	-	0	12.05±0.46	-
	CTAB 15	15	9.41±0.13	21.90
	CTAB 15	30	2.81±0.13	76.68
	CTAB 15	45	5.45±0.55	54.77
	SLS 10	15	8.9±0.44	26.14
	SLS 10	30	2.44±0.34	79.73
	SLS 10	45	5.01±0.34	58.42

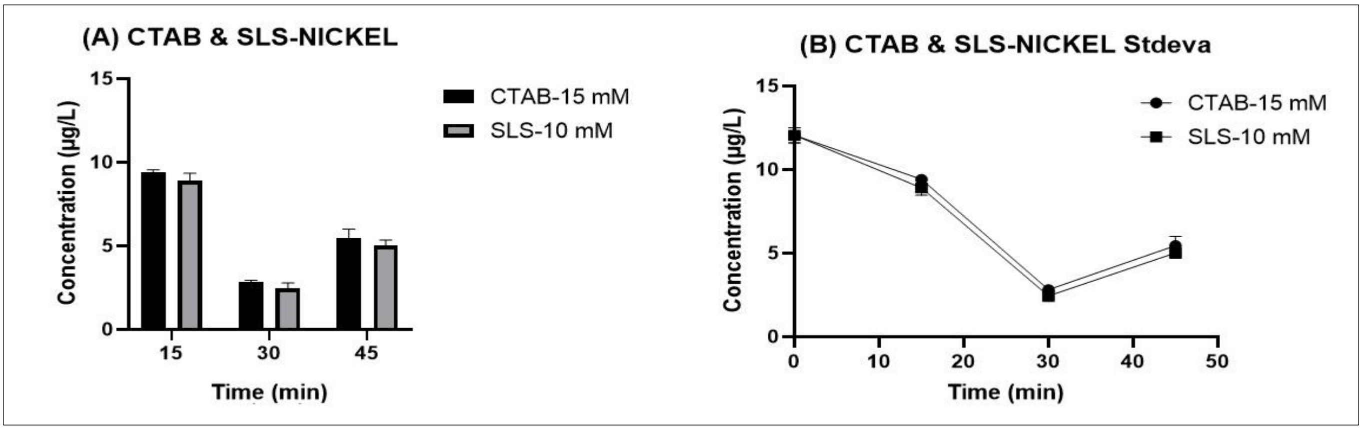


Fig 6: (A) Concentration of Nickel in CETP wastewater pH=7.22, volume of Sample 10 mL after extraction by CTAB & SLS surfactant different concentration reverse micelle solution, (B) Standard deviation graph of Nickel concentration in different time extraction by CTAB & SLS surfactant different concentration reverse micelle solution.

For synthetic wastewater, at pH 3.80, volume 10 mL, CTAB showed a maximum efficiency of 30.39% at 30 mM and 15 minutes (Fig. 4). At pH 3.30 SLS demonstrated similar results, reaching 25.61% at 20 mM and 45 minutes (Fig. 5). In CETP wastewater, at pH 7.22, volume 10mL CTAB reached 76.68% efficiency at 15 mM, 30 minutes, while SLS outperformed slightly with 79.73% efficiency at 10 mM, 30 minutes (Fig. 6).

3.3 Copper

Metal	CTAB Surfactant Concentration(mM)	Time (Minutes)	Concentration (µg/L)	Efficiency (%)
Copper	-	0	10210.3±93.94	-
	10	15	8815.33±81.77	13.68
	10	30	8256.67±96.88	19.13
	10	45	8140.67±58.35	20.26
	20	15	9458.67±104.71	7.36
	20	30	8993.33±58.35	11.91
	20	45	9210.33±71.25	9.81
	30	15	9373±35.68	8.21
	30	30	9179.33±48.44	10.10
	30	45	8971.33±48.95	12.12

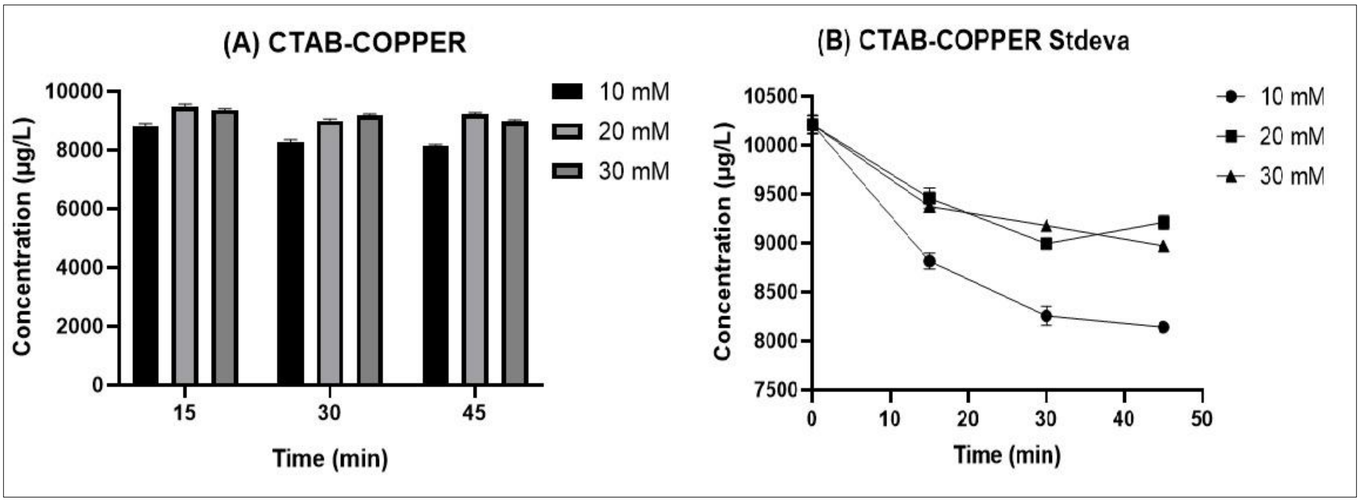


Fig 7: (A) Concentration of Copper in wastewater pH=2.05, volume of sample 10 mL after extraction by CTAB surfactant different concentration reverse micelle solution, (B) Standard deviation graph of in Copper concentration in different time extraction by CTAB surfactant different concentration reverse micelle solution.

Metal	SLS Surfactant Concentration(mM)	Time (Minutes)	Concentration (µg/L)	Efficiency (%)
Copper	-	0	9877±35.68	-
	10	15	8745±35.68	11.47
	10	30	8349.67±70.81	15.48
	10	45	8644.33±48.43	12.47
	20	15	8365.33±58.35	15.27
	20	30	7923.67±81.77	19.77
	20	45	8970.33±70.81	9.17
	30	15	8040±58.41	18.59
	30	30	7660±83.89	22.42
	30	45	8295.67±35.13	16.02

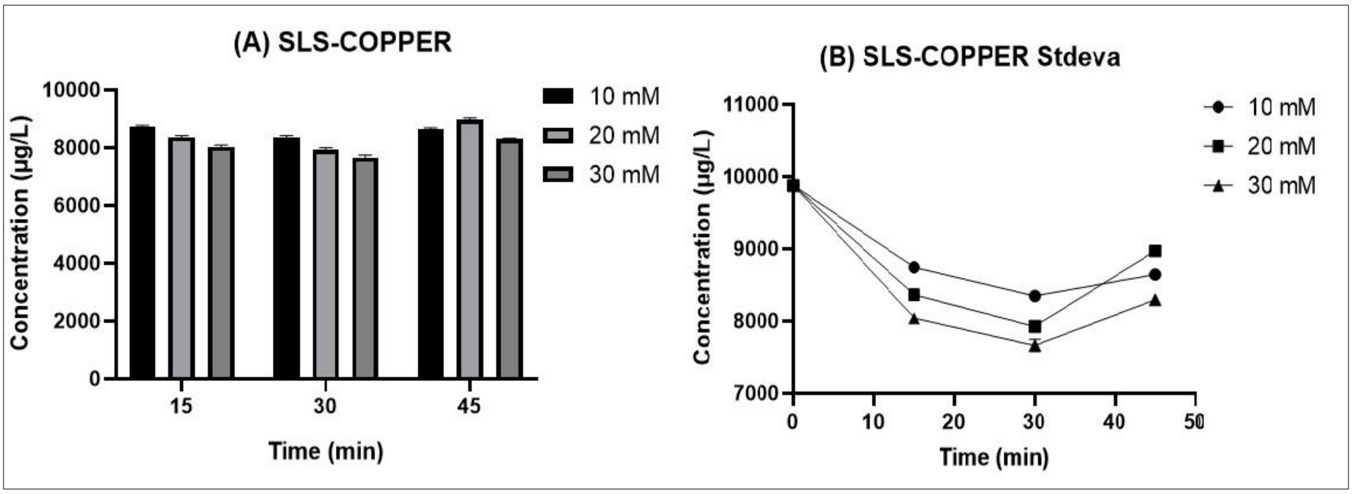


Fig 8: (A) Concentration of Copper in wastewater pH=2.03, volume of sample 10 mL after extraction by SLS surfactant different concentration reverse micelle solution, (B) Standard deviation graph of in Copper concentration in different time extraction by SLS surfactant different concentration reverse micelle solution.

Metal	Surfactant Concentration(mM)	Time (Minutes)	Concentration (µg/L)	Efficiency (%)
Copper	-	0	5360.67±17.89	-
	CTAB 10	15	3569.67±46.97	33.42
	CTAB 10	30	47.28±24.20	99.12
	CTAB 10	45	496.89±35.53	90.73
	SLS 10	15	89.92±17.76	98.32
	SLS 10	30	31.77±17.76	99.41
	SLS 10	45	287.59±46.99	94.63

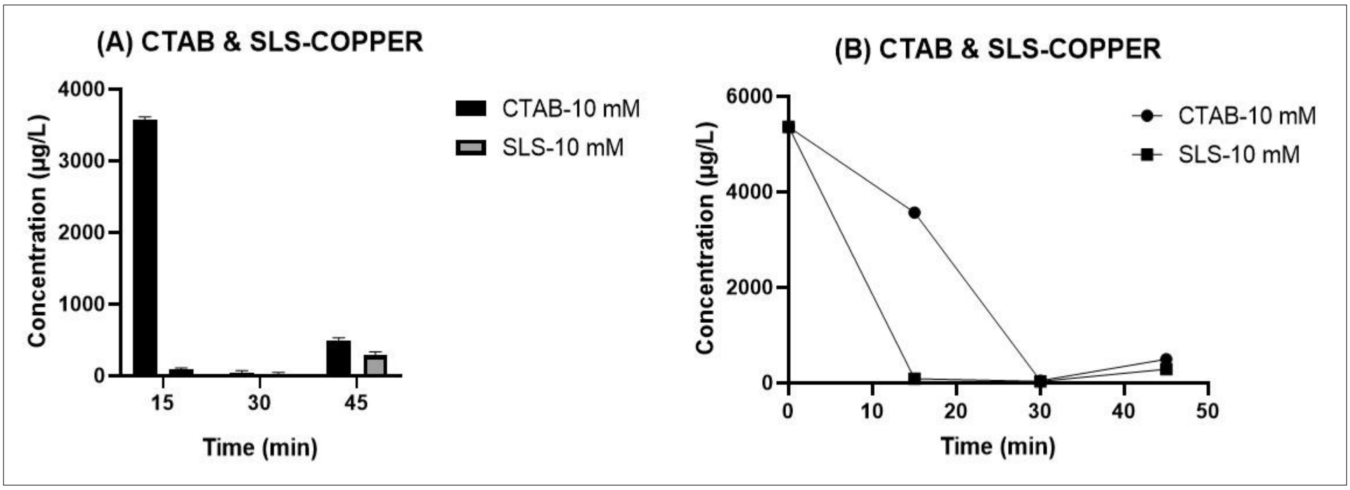


Fig 9: (A) Concentration of Copper in CETP wastewater pH=7.22, volume of Sample 20 mL after extraction by CTAB & SLS surfactant different concentration reverse micelle solution, (B) Standard deviation graph of Copper concentration in different time extraction by CTAB & SLS surfactant different concentration reverse micelle solution.

In synthetic wastewater, at pH 2.05, volume 10 mL, CTAB and SLS systems showed limited copper extraction, with CTAB reaching 20.26% at 10 mM, 45 minutes and at pH 2.03, volume, volume 10 mL SLS up to 22.42% at 30 mM, 30 minutes (Figs. 7 and 8). In CETP wastewater, at pH 7.22, volume 20 mL however, removal was highly effective, with CTAB achieving 99.12% at 10 mM, 30 minutes and SLS showing a slightly higher efficiency value of 99.41% under the same conditions (Fig. 9).

3.4 Manganese

Metal	CTAB Surfactant Concentration(mM)	Time (Minutes)	Concentration (mg/L)	Efficiency (%)
Manganese	-	0	12.2±0.07	-
	10	15	10.82±0.05	11.31
	10	30	8.6±0.08	29.51
	10	45	8.93±0.05	26.80
	20	15	6.27±0.08	48.61
	20	30	1.66±0.10	86.39
	20	45	8.43±0.05	30.90
	30	15	4.78±0.05	60.82
	30	30	1.15±0.09	90.57
	30	45	9.73±0.05	20.252

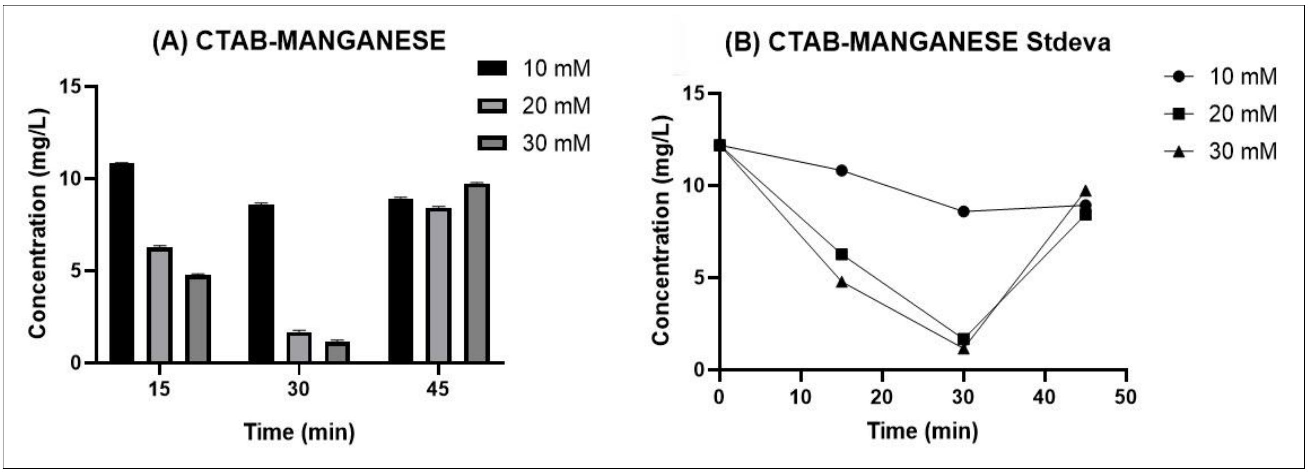


Fig 10: (A) Concentration of Manganese in wastewater pH=1.39, volume of sample 10 mL after extraction by CTAB surfactant different concentration reverse micelle solution, (B) Standard deviation graph of in Manganese concentration in different time extraction by CTAB surfactant different concentration reverse micelle solution.

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Metal	SLS Surfactant Concentration(mM)	Time (Minutes)	Concentration (mg/L)	Efficiency (%)
Manganese	-	0	12.39±0.05	-
	10	15	9.82±0.07	20.75
	10	30	10.12±0.05	18.33
	10	45	9.49±0.05	23.44
	20	15	9.39±0.07	24.24
	20	30	9.75±0.05	21.32
	20	45	10.42±0.04	15.88
	30	15	8.69±0.05	29.87
	30	30	9.17±0.07	26.00
	30	45	9.42±0.07	23.96

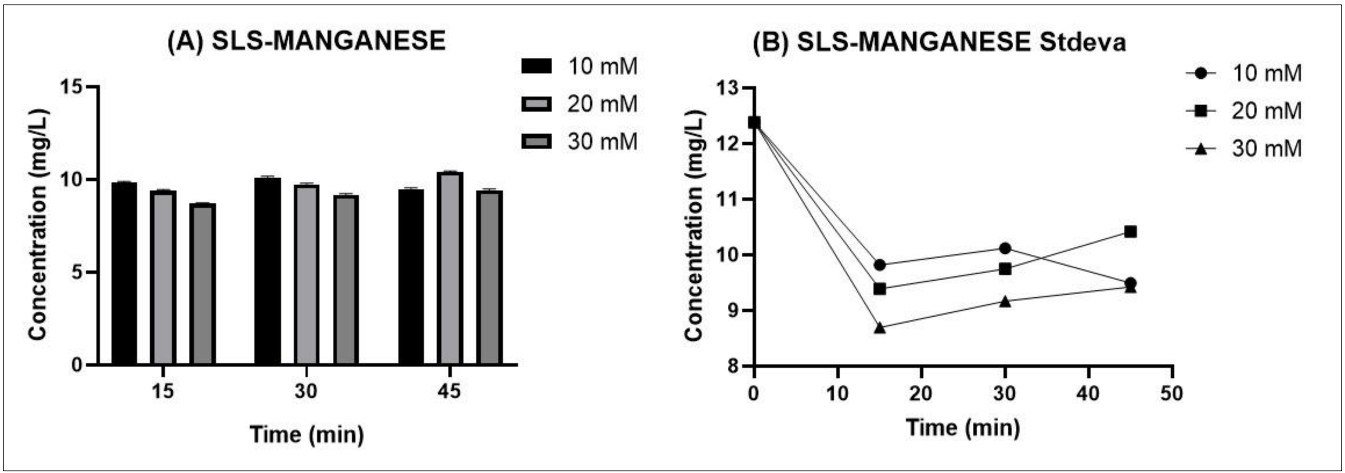

Fig 11: (A) Concentration of Manganese in wastewater pH=1.38, volume of sample 10 mL after extraction by SLS surfactant different concentration reverse micelle solution, (B) Standard deviation graph of in Manganese concentration in different time extraction by SLS surfactant different concentration reverse micelle solution.

Metal	Surfactant Concentration(mM)	Time (Minutes)	Concentration (mg/L)	Efficiency (%)
Manganese	-	0	8.36±0.09	-
	CTAB 10	15	1.58±0.01	81.10
	CTAB 10	30	1.46±0.01	82.54
	CTAB 10	45	1.51±0.01	81.93
	SLS 10	15	1.24±0.01	85.17
	SLS 10	30	0.36±0.01	95.69
	SLS 10	45	1.28±0.01	84.68

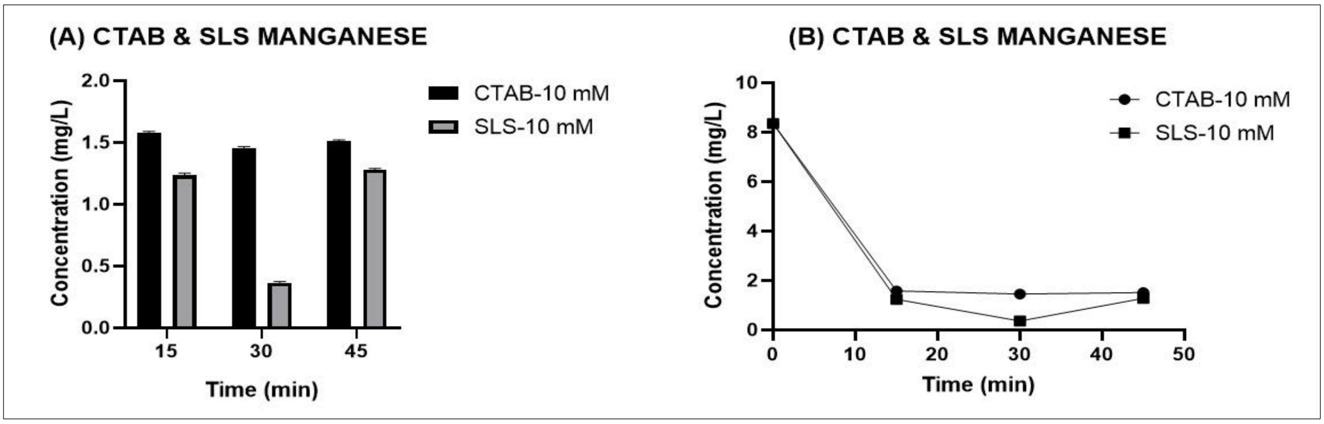

Fig 12: (A) Concentration of Manganese in CETP wastewater pH=7.22, volume of Sample 50 mL after extraction by CTAB & SLS surfactant different concentration reverse micelle solution, (B) Standard deviation graph of Manganese concentration in different time extraction by CTAB & SLS surfactant different concentration reverse micelle solution.

For synthetic wastewater, at pH 1.39, volume 10 mL, CTAB demonstrated strong performance, reaching 90.57% removal at 30 mM, 30 minutes, while SLS at pH 1.38 removal was lower at 29.87% 30 mM, 15 minutes (Figs. 10 and 11). In CETP wastewater, at pH 7.22, volume 50 mL, CTAB achieved 82.54% at 10 mM, 30 minutes and SLS achieved the highest efficiency among all metals at 95.69% at 10 mM, 30 minutes (Fig. 12).

4. Conclusion

The experimental evaluation of reverse micelle-based extraction reveals that both CTAB and SLS surfactants are effective in removing heavy metals from synthetic and CETP wastewater under optimized conditions of pH, surfactant dose and contact time. Effect of pH and Sample Volume, removal efficiency was influenced by solution pH, with acidic conditions favoring some metal extractions. CETP wastewater (pH ~7.22) consistently showed higher removal efficiencies across all metals compared to synthetic samples, likely due to its complex iconic composition. Sample volumes ranged from 10 mL to 50 mL depending on metal type and wastewater sources. CTAB vs. SLS Performance, CTAB showed better performance for chromium and copper, particularly in CETP wastewater. SLS outperformed CTAB in removing nickel and manganese from CETP samples. Optimal Extraction Conditions, Highest efficiencies were generally achieved at 10-15 mM surfactant concentration and 30 minutes extraction time. Research has affirmed the potential of reverse micelle extraction for environmental remediation, further developments are needed to adopt the process for large-scale operations. This includes selecting environmentally compatible surfactant solvent systems, optimizing process parameters for flow through treatment systems and ensuring economic viability.

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