

Removal of Heavy Metal by Micellar Solubilization Followed by Coagulation Flocculation Process

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Abstract— Contamination of wastewater with heavy metals has become a crucial issue to the environment. Recently, micellar enhanced ultrafiltration (MEUF) is a common method that is widely used for the elimination of metals ion using membrane but unfortunately, this method is unfavourable due to the membrane fouling problem. In this research, micellar solubilization process followed by coagulation flocculation process was applied for the removal of cadmium ion from wastewater. Sodium lauryl sulfoacetate was utilized as plant based surfactant in micellar solubilization process while both of ferric chloride and polyacrylamide were used in coagulation flocculation process. The effect of pH and effect of surfactant concentration on the cadmium Cd^{2+} removal were studied to determine the efficiency of micellar solubilization process. Results obtained from this research study shows that the highest percentage removal for coagulation flocculation process was 99.76% while for the micellar solubilization was obtained at 99.78%. Both of these methods occurred at pH 11 for the best removal efficiency of Cd^{2+} . It was observed that highest removal efficiency of Cd^{2+} was achieved at 99.78% at surfactant concentration of 250 ppm. The regression analysis via Excel Software shows that R^2 and adjusted R^2 obtained for micellar solubilization were 0.85 and 0.82 respectively while R^2 and adjusted R^2 obtained for coagulation flocculation were 0.76 and 0.40 respectively. Regression statistics and ANOVA analysis justified that the mathematical expression can be used to forecast the removal efficiency of Cd^{2+} from wastewater.

Keywords— micellar solubilization, coagulation process, flocculation process, heavy metal, cadmium, plant-based surfactant, anionic surfactant, sodium lauryl sulfoacetate

I. INTRODUCTION

Heavy metals or known as trace metals are one of the persistent pollutants in wastewater. The existence of heavy metals in wastewater can lead to the several issues to the environments. According to the Abbasi, E. *et al.* (2011), the highly toxic, carcinogen and non-biodegradable are the main consequences of heavy metals in wastewater [1]. If these heavy metals are directly discharged without any sufficient treatment, it can originate to the toxicity of the environment. Therefore, the efficient and inexpensive preventive methods are needed to develop the existing techniques in order to comply with Department of Environment, (DOE) requirement. Synthetic wastewater contained cadmium ion is treated and the main reason of treating wastewater contained cadmium because this heavy metal is one of the metal that is difficult to be treated and need to remove at very low concentration

according to the DOE requirement. Moreover, cadmium has its significant risk to the environment and human health [2]. Cadmium is a trace metal that has significant detriment and the possible consequences to the critical exposure of cadmium are kidney dysfunction and death if the cadmium is exposed at higher levels [3],[4].

The variety of methods are available for the treatment and removal of metal ions such as chemical precipitation, adsorption, and membrane filtration but there are some limitation due to high operation cost and inefficient process [5]. But, unluckily these methods have no capability to reduce toxic level of each metals. Recent process that extensively used for the treatment and elimination of heavy metals from aqueous streams is metal precipitation method. Fundamentally, these method is based on pH control and it is difficult to control at certain pH. In addition, the sludge generation is higher [6] and it requires extra operational cost for the disposal of the sludge. Specifically, micellar enhanced ultrafiltration (MEUF) is a most common method that is widely used for the elimination of metals ion using membrane but unfortunately, this method is unfavorable due to the membrane fouling problem.

Furthermore, coagulation and flocculation process is performed along with metal precipitation for destabilization of small suspended colloids by neutralizing the repulsive force for the formation of large particles [7]. Hydroxide precipitation is a formation of metal hydroxide precipitates for the removal of soluble metal ions from solution. Metal hydroxide precipitate is achieved when metal ion binds or attracts to the hydroxide ion (OH^-). The coagulation process can be attained by adding chemical coagulant or destabilizing agent such as Ferric chloride, Poly Aluminium Chloride (PACL) and also Aluminium sulfate (Alum) [8],[9] that usually positive charged that help to neutralize the negative charged of the small suspended colloids. Meanwhile, flocculation is a process where the polymers which are high molecular weight molecules are added into the coagulated effluent in order for the agglomeration of suspended solids into larger particles [7] by formation of bridges between the flocs. The flocculating agents usually used in industry for bridging of flocs are polyacrylamide, Chitosan and sodium silicate. Besides, these chemicals helps to initiate the sedimentation of sludge containing heavy metals [10]. The restraint of coagulation flocculation method is production of sludge is higher compared with micellar solubilization due to higher consumption of chemicals. Thus, it requires extra operation cost for the sludge operation such as sludge treatment, transportation and final disposal [11].

An excellent treatment to hinder the negative impacts of heavy metals in wastewater is micellar solubilization process followed by coagulation flocculation process. Basically, micellar solubilization process implicated the use of surfactant for the rejection of contaminants from wastewater. Surfactant is added into the wastewater to a concentration above the critical micelle

concentration or known as CMC in order for the formation of micelle. In addition, surfactant is added as a special ingredient to enhance the removal of Cd^{2+} from wastewater. Surfactant will tend to assemble and accumulate to form spherical amphiphilic micelle and contaminants tend to solubilize into the micelle [12]. Currently, most of the researcher are performed Cd^{2+} removal using chemical surfactant such as sodium dodecyl sulfate (SDS) and linear alkylbenzene sulfonate (LAS) [13] because less found that use plant based surfactant to remove heavy metal. In this research, plant-based anionic surfactant such as sodium lauryl sulfoacetate can be an alternative to replace synthetic surfactant because it is an eco-friendly surfactant with variety of benefits such as organic, biodegradable material and also environmental friendly [14],[15]. Anionic surfactant is introduced into the wastewater for the removal of cadmium ion, Cd^{2+} because the hydrophilic head contained anionic or negative charge [16] will attract to the pollutant that carry cationic or positive charge such as heavy metal ion [12].

The ultimate goal of the research is to treat synthetic wastewater contained Cd^{2+} using micellar solubilization followed by coagulation flocculation process. The research study is investigated the effect of pH and surfactant concentration on the removal of Cd^{2+} with and without the presence of micellar. All the data obtained are predicted and verified using regression analysis via Excel software.

II. METHODOLOGY

A. Materials

Cadmium nitrate tetrahydrate, $[\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}]$ (purity of 98% and molecular weight of 308.48 g/mol) was supplied by Sigma-Aldrich Malaysia and used to prepare synthetic wastewater. Ferric chloride, (FeCl_3) (molecular weight of 162.20 g/mol) was supplied by Sigma-Aldrich Malaysia and used as coagulant. Polyacrylamide (molecular weight of 71.078 g/mol) was supplied by Sigma-Aldrich Malaysia and used as flocculant. Sodium hydroxide solution, (NaOH) (molecular weight of 39.997 g/mol) was obtained from Sigma-Aldrich Malaysia and used to adjust the pH of solution. An anionic plant based surfactant, sodium lauryl sulfoacetate (molecular weight of 330.42 g/mol and CMC of 200 ppm) was used in this experiment. The chemical structure of Sodium Lauryl Sulfoacetate is shown in Figure 1 below.

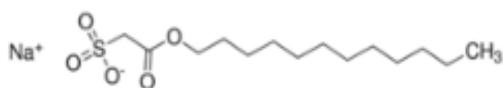


Fig.1: Sodium Lauryl Sulfoacetate

B. Preparation of synthetic wastewater and anionic surfactant

Synthetic wastewater was prepared at 1000 mg/L using cadmium (II) nitrate tetrahydrate, $[\text{Cd}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}]$ with distilled water. Anionic surfactant was prepared using sodium lauryl sulfoacetate and distilled water. The sodium lauryl sulfoacetate solution was prepared by mixing and stirring on the hot plate at speed of 150 rpm and at temperature 50°C for about 7 hours in order for the anionic surfactant to dissolve in the distilled water.

C. Coagulation and flocculation process

The synthetic wastewater was added into 6 different beakers at constant volume of 250 mL and mixed using jar tester at the speed of 100 rpm for 3 minutes for rapid mixing in order to homogenize the solution. The initial pH of synthetic wastewater in

each beakers was determined using pH meter. Next, 7.5 % of sodium hydroxide solution (NaOH) was added for hydroxide precipitation method and pH was adjusted to obtain the desired pH between pH 7 until pH 12. The selection of best pH for Cd^{2+} removal was obtained from minimum solubility of Cd^{2+} . The volume of NaOH used in each beaker was recorded. The pH was adjusted at pH 7.5 until pH 8 by adding 7.5% of ferric chloride solution (FeCl_3) and the volume of ferric chloride solution (FeCl_3) used was also recorded. Then, 0.4 mL of 4.0 % anionic flocculant, polyacrylamide was added into medium for the bridging of colloid particles. The solution was mixed by using jar tester at 50 rpm for 14 minutes at slow mixing in order to ensure the formation of flocs. Lastly, let the samples to settle down for the sedimentation process for 45 until 60 minutes. The samples were filtered using filter paper.

D. Micellar solubilization process

The anionic surfactant, sodium lauryl sulfoacetate solution was prepared at different concentrations of 250 ppm, 300ppm, 350 ppm, 350 ppm, 400 ppm and 500 ppm. The solution was mixed adequately at speed of 300 rpm by using jar tester for 15 minutes to dissolve the surfactant into wastewater. Next, the speed of jar tester was slow down until 100 rpm and the initial pH of solution was recorded using pH meter. 7.5 % of sodium hydroxide solution (NaOH) was added and pH was adjusted to obtain the desired pH at pH 7 until pH 12 for each beaker. The volume of NaOH used was recorded. 7.5% of ferric chloride solution (FeCl_3) was added to adjust the pH at pH 7.5 until pH 8 and the volume of ferric chloride solution (FeCl_3) used was recorded. Then, 0.4 mL of 4.0% polyacrylamide was added into the medium for the flocculation process and the solution was mixed at speed of 50 rpm for 15 minutes to ensure flocs formation. The samples were allowed to settle down for the sedimentation process in 45 until 60 minutes and samples were filtered using filter paper.

E. Experimental analysis

The pH of medium was measured using Mettler Toledo pH Meter. The functional group of anionic surfactant, sodium lauryl sulfoacetate was analyzed using Spectrum One Fourier Transform Infrared Spectrometer (FTIR) from Perkin Elmer Sdn Bhd. Next, the coagulation and flocculation process was performed by using WiseStir Jar Tester. The concentration of the residual cadmium ions, (Cd^{2+}) was analysed using Hitachi Z2000 Atomic Absorption Spectroscopy (AAS) from Chemopharm Sdn Bhd. The cadmium ions, Cd^{2+} removal efficiency, $R(\%)$ was calculated using equation (1).

$$R(\%) = \frac{C_0 - C_f}{C_0} \times 100\% \quad (1)$$

Where C_0 is the initial concentration of cadmium ions and C_f is the final concentration of cadmium ions [2].

III. RESULTS AND DISCUSSION

A. Characterization of sodium lauryl sulfoacetate surfactant

Plant based surfactant such as sodium lauryl sulfoacetate was introduced in the micellar solubilization process and the type of surfactant used was anionic surfactant. The functional group of sodium lauryl sulfoacetate was determined using Spectrum One Fourier Transform Infrared Spectroscopy (FTIR) from Perkin Elmer Sdn Bhd.

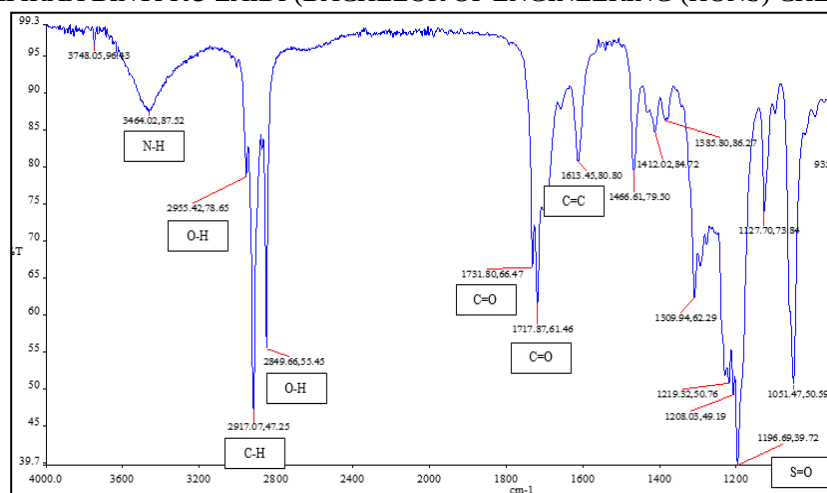


Fig. 2: FTIR spectra for sodium lauryl sulfoacetate

Figure 2 indicates the FTIR spectra for sodium lauryl sulfoacetate. From this figure, the strong absorption peak was observed at 1196.69 cm^{-1} that indicates sulfonate S=O stretch or known as sulfonate group. This peak contributed to the anionic part of surfactant that contained negative charge. This sulfonate group had ability to bind with positive charge of Cd^{2+} . Meanwhile, the peak visible at 1717.87 cm^{-1} that indicates ketone C=O stretch.

B. Characterization of synthetic wastewater

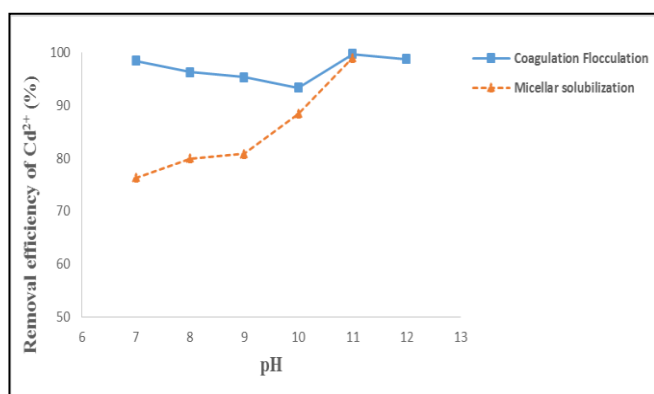
The characterization of synthetic wastewater is carried out in order to prepare wastewater according to Standard Methods for the Examination of Water and Wastewater [17]. Based on Table 1, concentration of Cd in synthetic wastewater was prepared at 1 mg/L while the initial concentration of Cd in synthetic wastewater measured by AAS was 0.8597 mg/L.

Table 1: Characteristic of Synthetic Wastewater

Type of Heavy Metal	Initial concentration of synthetic wastewater prepared (mg/L)	Initial concentration of synthetic wastewater measured by AAS (mg/L)
Cadmium	1.0	0.8597

C. Effect of pH

Removal efficiency (%) of the cadmium ion (Cd^{2+}) as a function of pH is presented in figure 2.

Fig. 3: Effect of pH on the removal efficiency of Cd^{2+}

Based on figure 2, the removal efficiency of Cd^{2+} for coagulation flocculation process increases sharply when it reaches pH 11. Therefore, the highest removal efficiency of cadmium ions (Cd^{2+}) is at pH 11 which is 99.76%. When pH of solution increases, the amount of H^+ in the solution decreased and the amount of OH^- increased that cause more Cd^{2+} attracted to the OH^- for the formation of cadmium hydroxide precipitation. Thus, the percentage removal increases sharply [18] at pH 11. This can be proved from the solubility curves where the pH at the optimum solubility for cadmium ions occurred at pH 11.2. So, pH 11.2 is the desired pH value to remove cadmium ions from wastewater. Caustic soda or sodium hydroxide solution (NaOH) was added at the beginning of experiment in order to adjust the desired pH at pH 7 until pH 12. The volume of caustic soda increasing when the pH increasing because the higher pH will generate more hydroxide ion (OH^-) and the hydroxide ion will react with cadmium ion (Cd^{2+}) to form cadmium hydroxide precipitate. Next, the optimum removal efficiency of cadmium ion (Cd^{2+}) is at pH 10 which is at 93.14% removal. The small amount of caustic soda used at pH 10 that lead to the least production of hydroxide ion (OH^-). So, the formation of cadmium hydroxide precipitate is reduce at pH 10. At lower pH, amount OH^- decreased that reduced the possibility of Cd^{2+} to attract to the OH^- . Hence, the quantity of cadmium precipitates becomes lower that minimized the removal of Cd^{2+} from wastewater.

Micellar solubilization process using plant based surfactant such as sodium lauryl sulfoacetate at concentration of 250 ppm was performed to investigate the effect of pH influences the removal efficiency of Cd^{2+} . It is observed that the highest removal of Cd^{2+} by using sodium lauryl sulfoacetate surfactant was obtained at 99.78% removal at pH 11. According to Xu, K. *et al* (2007), the Cd^{2+} rejection efficiency higher at pH 11 [18]. The removal efficiency of Cd^{2+} increases sharply with the increment of pH because of competition between H^+ and Cd^{2+} trapped on the surface of the micelles. H^+ can be bound on the surface of the sodium lauryl sulfoacetate micelles with positive charge and occupies the binding site. When the pH of solution increased, the amount of H^+ in the solution decreased and more Cd^{2+} bound on the surface of micelles and trap in the micelles. Thus, results in the increment of removal efficiency of Cd^{2+} [2]. Next, the lowest removal efficiency of Cd^{2+} was discovered at 76.29% removal at pH 7. This is due to the amount of H^+ at pH 7 is higher and the competition between Cd^{2+} and H^+ to trap on the surface of micelles [2]. More H^+ bound to the surface of micelles and occupied the binding site compared with Cd^{2+} because more H^+ in the solution corresponds to the lower removal efficiency [2]. At lower pH, the presence of surfactant caused hindrance to hydroxide precipitation and Cd^{2+} trapped in the micelles that reduced possibility to become precipitation.

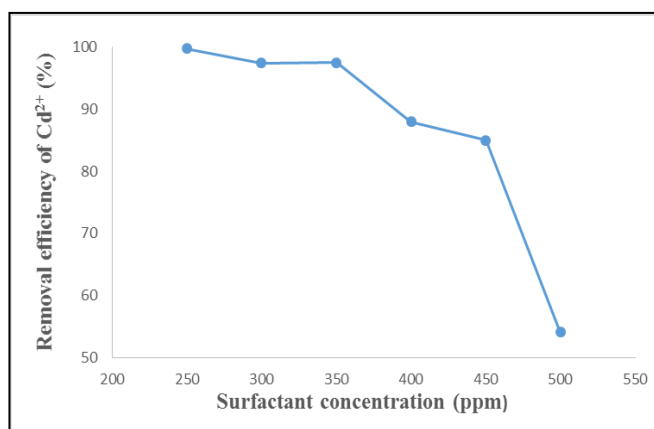
Table 2: Comparison between Coagulation Flocculation and Micellar Solubilization Method

Methods	Coagulation Flocculation		Micellar Solubilization	
Desired pH	11	12	10	11
Dosing of NaOH (mL)	0.65	8.37	0.10	0.64
Dosing of FeCl ₃ (mL)	0.8	11.25	0.05	0.74
Final Concentration of Cd ²⁺ (mg/L)	0.0021	0.0107	0.0995	0.0019
Percentage Removal (%)	99.76	98.76	88.43	99.78

Dosing of coagulant such as ferric chloride (FeCl₃) increases as well as volume of sodium hydroxide solution (NaOH) with the increment of pH for both methods. The consumptions of NaOH and FeCl₃ for coagulation flocculation method are higher compared with micellar solubilization method. Thus, micellar solubilization method is more efficient and economical as the dosage of chemicals are lowered compared with coagulation flocculation method. Greater utilization of chemicals are not economic and environmental friendly as it requires extra operating cost. The formation of sludge in coagulation flocculation method is higher due to the higher consumption of NaOH and FeCl₃. The concentration of residue cadmium ion (Cd²⁺) at pH 11 is lowest compared with others which is 0.0021 mg/L for coagulation flocculation method while 0.0019 mg/L for micellar solubilization. Both concentrations of residue Cd²⁺ are compatible and comply with Department of Environment (DOE) requirement because the allowable concentration of Cd²⁺ must be 0.02 mg/L and below in order to discharge the wastewater [19].

D. Effect of Surfactant Concentration

Figure 3 shows the effect of surfactant concentration on the removal efficiency of Cd²⁺. The micellar solubilization using sodium lauryl sulfoacetate surfactant at different concentration (250 ppm, 300 ppm, 350 ppm, 400 ppm, 450 ppm, 500 ppm) were conducted at pH 11 in order to investigate the effect of surfactant concentration on the removal efficiency of Cd²⁺.

Fig. 4: Effect of surfactant concentration on the removal efficiency of Cd²⁺

According to figure 3, the removal efficiency of Cd²⁺ decreases with the increases of the surfactant concentration. It is observed that the highest removal of Cd²⁺ by using sodium lauryl sulfoacetate surfactant was obtained at 99.78% removal at surfactant concentration of 250 ppm while the lowest removal efficiency of Cd²⁺ was found at 54.06% at 500 ppm surfactant concentration. From the previous studies, according to Mungray, S.

et al. (2011) the highest rejection of Cd²⁺ was 99.4% for the surfactant concentration as parameter studied [20]. The critical micelle concentration (CMC) of sodium lauryl sulfoacetate surfactant was 200 ppm and the highest rejection of Cd²⁺ was at 250 ppm which was above the CMC value. However, when the surfactant concentration is higher than CMC value, with the increment of surfactant concentration, the removal of Cd²⁺ is decreased which also further increase in the surfactant concentration would make the removal of Cd²⁺ decreased [12]. This phenomenon was discovered by Xu, K *et al.*, it was analyzed that the shape change with the increased of surfactant concentration, but the number of effective binding site do not increase correspondingly [18].

E. Regression Analysis

A linear regression model via Excel Software was applied to analyze and verify the relationship between removal efficiency of Cd²⁺ as the response (y) and two parameters involved such as initial pH (x₁) varying from pH 7 until pH 12 and surfactant concentration (x₂) in the range of 250 ppm until 500 ppm using micellar solubilization process. The mathematical expression can be expressed in equation (2):

$$y = -1.6 \times 10^{-5} x_2^2 x_1 + 111.7568 \quad (2)$$

The regression statistics and ANOVA for the expression above is tabulated in Table 3 below.

Table 3: Regression statistics and ANOVA for removal efficiency of Cd²⁺ for micellar solubilization.

Regression statistics and ANOVA	
Multiple R	0.922979
R Square	0.85189
Adjusted R Square	0.814862
Standard Error	7.385688
Observations	6
Significance F	0.00867
P-value for intercept	4.84×10^{-5}
P-value for $x_2^2 x_1$	0.00867

From Table 3, it can be seen that the significance F and P-value for $x_2^2 x_1$ and intercept are less than 0.05 and it shows that the results obtained are quite reliable and proved that the correlation is acceptable [21]. The value R² and adjusted R² attained were 0.85189 and 0.814862 respectively. The value of adjusted R² signified that about 80% of the variations of Cd²⁺ removal in the model can be forecasted by predictor parameters such as pH and surfactant concentration.

For coagulation flocculation method, a linear regression model via Excel Software was applied to analyze and verify the relationship between removal efficiency of Cd²⁺ as the response (y) and two parameters involved such as dosage of NaOH as (x₁) and dosage of FeCl₃ as (x₂) The mathematical expression can be expressed in equation (3):

$$y = 105.4125 x_1 - 81.2176 x_2 + 0.363368 x_1 x_2 + 95.94044 \quad (3)$$

The regression statistics and ANOVA for the expression above is tabulated in Table 4.

Table 4: Regression statistics and ANOVA for removal efficiency of Cd^{2+} for coagulation flocculation

Regression statistics and ANOVA	
Multiple R	0.872239
R Square	0.7608
Adjusted R Square	0.402001
Standard Error	1.9259
Observations	6
Significance F	0.33640
P-value for intercept	0.000117
P-value for x_1	0.217318
P-value for x_2	0.234557
P-value for x_1x_2	0.609411

From Table 4, it shows that only P-value for intercept is less than 0.05 while significance F, P-value for x_1 , x_2 and x_1x_2 are more than 0.05 and it shows that the results obtained are not reliable. The value of R^2 and adjusted R^2 acquired were 0.7608 and 0.4020 respectively and it indicates that the data are not well fitted.

IV. CONCLUSION

Cadmium, Cd^{2+} removal from wastewater was investigated in this research study by micellar solubilization followed by coagulation flocculation process using anionic plant-based surfactant such as sodium lauryl sulfoacetate. The capability of sodium lauryl sulfoacetate surfactant to increase the rejection of Cd^{2+} from wastewater near to 100% removal. The highest removal efficiency of Cd^{2+} for micellar solubilization was obtained at 99.78% while for coagulation flocculation process was attained at 99.76%. Both of these methods achieved the best removal efficiency of Cd^{2+} at pH 11. From the previous studies, pH 11 was chosen as the best pH for the maximum removal of Cd^{2+} from wastewater. Surfactant concentration at 250 ppm achieved the highest removal efficiency of Cd^{2+} which was at 99.78% removal. This shows the sodium lauryl sulfoacetate was the effective plant-based surfactant for the treatment of wastewater. The data of process treatment was predicted and verified using regression analysis via excel software. It is proved that ANOVA analysis of the mathematical expression can be applied to forecast the removal efficiency of Cd^{2+} for micellar solubilization varies from pH 7 until pH 12 for initial pH and also surfactant concentration in the range of 250 ppm until 500 ppm while ANOVA analysis of the mathematical expression for coagulation flocculation method is not well fitted as the values obtained were not reliable.

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