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Optimization of adsorption process by aluminum dross for treatment of cutting oil wastewater

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ABSTRACT: This research employed untreated aluminum dross obtained from auto parts manufacturing waste as an adsorbent to meet the sustainable goals of waste utilization in readily available, inexpensive, and environmentally friendly materials to treat cutting oil wastewater in auto parts manufacturing. Aluminum dross, categorized as white dross with 37.13% aluminum and 7.77% silicon, is a source of silicon- and aluminum-containing wastes. Process optimization was conducted using a central composite rotary design (CCRD) coupled with response surface methodology (RSM). The results indicated that the adsorption of aluminum dross at optimum conditions was 15 g adsorbed from 1% v/v cutting oil wastewater at pH 2 in 60 minutes, achieving the removal efficiency at 93.30% of chemical oxygen demand (COD), 88.33% of turbidity, and only 40.47% of total organic carbon (TOC). The second-order equation demonstrated coefficients of determination (R^2) and adjusted R^2 values of 0.9738 and 0.9345, respectively, for COD removal. The COD adsorbed on aluminum dross could be adequately explained using the pseudo-second-order kinetic and Langmuir models. Aluminum dross adsorption for cutting oil treatment has a low operating cost of 0.5398 USD/ m^3 .

KEYWORDS: chemical oxygen demand removal, wastewater treatment, waste utilization, response surface methodology

INTRODUCTION

A significant amount of waste, known as aluminum dross, is a by-product in the production of aluminum [1]; one of which is the result of melting aluminum when producing auto parts. Aluminum dross is categorized as hazardous waste, and its disposal in landfills is a worldwide problem [2], due to its negative impact on the ecosystem, surface water, and groundwater [3,4]. In general, aluminum dross is mainly composed of Al₂O₃, AlN, Al₄C₃, SiO₂, MgO, Al, and trace amount of Si. It is classified into three types: black dross, white dross, and salt cake. White dross contains 15%-70% recoverable metallic aluminum, black dross contains 12%-18% recoverable aluminum, and salt cake contains 3%-5% residual metallic aluminum [5]. In addition, aluminum dross is a waste product with practical applications in numerous devices and products, such as composite fillers, refractories, coagulants, deoxidizers in steel-making, and adsorbents [6, 7]

During machining, oil wastewater is generated by

cutting fluids, which improves efficiency by reducing power consumption, controlling rust, and extending the life of tools [8]. As most metalworking oils and fluids must be diluted before use, excessive cutting oil wastewater is produced [9, 10]. Metalworking fluids (MWFs) with high biochemical and chemical oxygen demands result from repeated use of cutting fluids contaminated with trash, small chips, bacteria, and metals [11]. Cutting oil wastewater can be treated via adsorption, which is a simple, eco-friendly, and economical process; through this process desirable adsorbent properties, such as excellent adsorption capacity, low cost, and abundance, should be obtained [12]. Several materials have been developed as adsorbents for treating oily wastewater, including agricultural waste, such as corncob [13] and straw [11]; synthesized materials, such as superhydrophobic MS/TiO₂/PDMS sponge [14] and modified sepiolite [15]; and waste materials, such as cement waste [16] and aluminum dross. Aluminum dross comprises SiO₂- and Al₂O₃based wastes that have been prepared for use as adsorbent materials [5]. Hence, it was used as adsorbent

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in this research without modification. As a result, the perspective of a sustainable goal with the use of solid waste-treated wastewater in terms of a circular economy perspective has a current trend and future opportunity for waste utilization [17].

Aluminum dross, a hazardous solid waste of the auto parts manufacturing process, was selected as an adsorbent for treating cutting oil wastewater in an auto parts manufacturing facility to achieve sustainable waste management. For improvement of performance in the experiments, a central composite rotary design (CCRD) coupled with response surface methodology (RSM) was used to optimize the operational parameters. This technique maximizes the benefits while requiring the fewest number of experiments and the shortest run period. The operation variables, namely, the initial pH, adsorbent dosage, and initial concentration, were optimized during the adsorption process.

MATERIALS AND METHODS

Adsorbent preparation and characterization

Aluminum dross was collected from casting and molding processes in an auto part manufacturing plant in Chonburi Province, Thailand, dried to a constant weight at 105 °C and sieved into size fractions of 0.21–0.50 mm using a mechanical sieve shaker, and placed in a desiccator. The aluminum dross was not modified in the study. The surface morphology was examined by scanning electron microscopy (SEM; Apreo, FEI, Netherlands). The specific surface area was measured using an ASAP 2020 BET analyzer (Micromeritics Instrument Corp., USA). The chemical state was investigated using X-ray diffraction (XRD; Empyrean, PANalytical, Netherlands), and the elemental contents were determined using X-ray fluorescence (XRF) spectroscopy (XGT-5200WR, HORIBA, Japan).

Synthesis of cutting oil wastewater

Cutting oil wastewater was synthesized using commercial cutting oil (Castrol Almeredge $^{\scriptscriptstyle \rm TM}$ BI) and mixed with deionized water before experimentation. The initial pH level of the solution was adjusted to meet the varying initial pH using sulphuric acid ($\rm H_2SO_4$) and sodium hydroxide (NaOH) purchased from Merck Millipore (Germany). The oil-in-water emulsion was continuously stirred at 500 rpm for 10 min to obtain a homogeneous solution. Deionized water (18.2 $\rm M\Omega.cm)$ was used to prepare solutions for all experimental operations.

Adsorption experiments

In all experiments, batch adsorption was conducted by mixing 0.2–0.5 mm aluminum dross with 100 ml of synthetic cutting oil in a 250-ml Erlenmeyer flask. The mixers were agitated in a rotating shaker, reaching equilibrium after 60 min at a mixing speed of 120 rpm. Every aliquot obtained at the end of the experiment

was filtered and evaluated for the response parameters COD and TOC (except turbidity). All adsorption experiments were performed in triplicate. The COD was determined using the dichromate closed reflux method (Method 5520C) (APHA, 2005). The TOC (Method 5310B; APHA, 2005) and turbidity levels were measured using a TOC analyzer (Shimadzu, Japan) with a range of 0-500 mg/l and a TN-100 waterproof turbidimeter (Thermo ScientificTM Eutech, USA) with a range of 0-1000 NTU. Moreover, heavy metals such as Fe, Mn, Cr, Cu, Al, and Si were monitored in aliquots under optimum conditions using inductively coupled plasma spectroscopy (Avio200, Perkin Elmer, USA). The adsorption efficiencies of COD, TOC, and turbidity were calculated using Eq. (1). The equilibrium adsorption capacity of COD adsorbed onto the adsorbent (Q, mg/g) was calculated using Eq. (2).

Adsorption efficiency (%) =
$$\frac{(C_0 - C_t) \times 100}{C_0}$$
, (1)

$$Q = \frac{(C_0 - C_e)V}{m},\tag{2}$$

where C_0 is the initial concentration of COD, TOC, and turbidity; C_t is the concentration of COD, TOC, and turbidity at time t; C_e is the equilibrium concentration (mg/l); V is the volume of solution (l); and m is the weight of the adsorbent (g).

Experimental design

In this experimentation, CCRD coupled with RSM was used to develop a second-order RSM model, and three optimized independent variables: initial pH (2–7), quantity of aluminum dross (5–15 g), and initial cutting oil concentration (1%–3% v/v) were employed. The three response functions; i.e., COD, TOC, and turbidity removal efficiency were analyzed to determine the optimum conditions in batch experiment. Sixteen CCRD experiments were required for each function with five levels (-1.68, -1, 0, +1, and +1.68), as listed in Table 1.

The correlation between the response and the independent variables is shown by the general second-order polynomial equation Eq. (3):

$$Y = \beta_0 + \sum_{i=1}^k \beta_i x_i + \sum_{i=1}^k \beta_{ii} x_i^2 + \sum_{i=1}^k \sum_{j \neq i=1}^k \beta_{ij} x_i x_j + \varepsilon,$$
 (3)

where Y is the predicted response (% removal); β_0 is a constant coefficient; β_i , β_{ii} , and β_{ij} are the coefficients for the linear, quadratic, and interaction effects, respectively; k is the number of independent variables; and ϵ is a random error.

The polynomial models were statistically validated using analysis of variance (ANOVA), and their statistical significance was confirmed using the F-test. The coefficient of determination (R^2) and adjusted

Factor	Coded level for factor						
	-1.68	-1	0	+1	+1.68		
Initial pH	0.3	2.0	4.5	7.0	8.7		
Quantity of aluminum dross (g)	1.59	5.00	10.00	15.00	18.41		
Initial cutting oil concentration (% v/v)	0.32	1.00	2.00	3.00	3.68		

Table 1 Factors and their values for the central composite rotatable design matrix.

 R^2 values were used to express the quality of the fitted polynomial model [18]. Furthermore, the adsorption kinetics and isotherms were modified to the optimum conditions for CCRD experimentation [19]. STATISTICA 10 (Statsoft, Tulsa, OK, USA) was used to analyze the data and design the statistics for the CCRD experiments. COD was considered a performance parameter for governing the kinetic study and isotherms presented herein [20]. The diagram of the experiment is illustrated in Fig. S1.

RESULTS AND DISCUSSION

Material characterization

Aluminum dross consists of small well-scattered particles of irregular shapes with no voids or cavities [21]. Investigation at higher magnification revealed low sharpness and unevenness in the images, possibly due to the interference of oxides located on the surface of the aluminum dross [6].

The chemical composition of aluminum dross (%wt) was determined using XRF. The aluminum dross was classified as white dross because it contained 15%–70% recoverable metallic aluminum. The XRD patterns of the 0.2–0.5 mm aluminum dross illustrated the primary Al 0 state forms characterized by the diffraction peak at 20 = 38.48° and elements such as Al $_2$ O $_3$, MgAl $_2$ O $_4$ [3], K $_{0.67}$ Al $_6$ O $_{9.33}$, SiO $_2$ [6], NaMgF $_3$, NaCl, KCl, and Si. Aluminum dross size of 0.2–0.5 mm was 6.4986 m 2 /g with a BET surface. Additionally, aluminum dross is a widely available and costeffective source of aluminum- and silicon-containing waste, which can be used to create porous materials for adsorbents [5].

Optimizing conditions

Regression model equation

Table 2 shows the influence of independent variables (initial pH, quantity of aluminum dross, and initial oil concentration) on the observed and the predicted response variables (COD, TOC, and turbidity removal efficiency). Among the three response variables, only the COD response model demonstrated adequate predictability. The coefficient of determination (R^2) for the COD removal response function was 0.9738, indicating that the fitted model could be described with an accuracy of 97.38% using the mathematical model of the COD removal response function. The adjusted

 R^2 value for the COD removal efficiency was 0.9345, indicating that the mathematical model of the COD response (4) could be accurately predicted for the independent variables. As listed in Table 3, the R^2 and the adjusted R^2 values were \sim 1.0, indicating a high correlation between the observed and the predicted values [18]. The mathematical model representing the response and the independent variable functions is as follows:

COD removal (%) =
$$94.9255 - 8.6186x_1 + 2.6908x_2$$

- $19.7276x_3 + 0.2601x_1^2 - 0.0575x_2^2 + 2.9198x_3^2$
+ $0.0804x_1x_2 + 1.3580x_1x_3 + 0.0690x_2x_3$, (4)

where x_1 is the initial pH, x_2 is the quantity of aluminum dross, and x_3 is the initial concentration (% v/v) of cutting oil.

For the ANOVA results, the F-value for the model was 24.77 for the COD, which is higher than the $F_{\rm tabulated}$ of 4.10. The F-value for the lack of fit for the model was 66.18 for the COD, which is lower than the $F_{\rm tabulated}$ of 230.16 for a 95% confidence level. As expected, only the mathematical model for the COD fitted the experimental data well, indicating that this was a significant model, and that a non-significant lack of fit was exhibited [22].

The predicted equation of the COD removal efficiency (4) was used to generate its response surface plots (Fig. 1). The increased initial pH level resulted in a lower COD removal efficiency for all quantities of aluminum dross (Fig. 1a) and initial oil concentrations (Fig. 1b). The results indicated that increasing the initial pH level from 2 to 7, under an initial cutting oil concentration of 1% v/v and 15 g of aluminum dross decreased the COD removal efficiency from 95.51% to 76.94%. The results of aluminum dross, as an adsorbent, were identical to those obtained by the unmodified lingo-cellulosic straw, which achieved the maximum removal efficiency of oil adsorption under strongly acidic conditions [11, 23].

High removal efficiencies were achieved by treatment with a high quantity of aluminum dross (Fig. 1c). An increase in the quantity of aluminum dross from 10 to 15 g at initial pH 2 with a cutting oil concentration of 1% resulted in an increase in the COD removal efficiency from 88.09% to 95.51%. A critical factor was the amount of adsorbent used – the higher the amount, the higher the removal efficiencies; due to

Table 2 Observed and predicted responses of COD, TOC, and turbidity removal using aluminum dross as an adsorbent for 60 min.

Run		Coded levels		COD (%)		TOC (%)		Turbidity (%)	
	$\overline{x_1}$	x_2	x_3	Y ^a	Y ^b	Y ^a	Y ^b	Ya	Y ^b
1	2.00	5.00	1.00	79.43	77.80	76.56	47.40	42.43	46.03
2	2.00	5.00	3.00	69.32	67.83	60.16	62.10	7.13	7.09
3	2.00	15.00	1.00	93.30	95.51	40.47	52.18	88.33	74.84
4	2.00	15.00	3.00	86.46	86.91	74.59	50.45	19.70	23.85
5	7.00	5.00	1.00	57.14	55.22	28.32	47.18	11.83	13.90
6	7.00	5.00	3.00	62.50	58.82	78.87	61.88	9.13	28.85
7	7.00	15.00	1.00	76.92	76.94	48.75	41.53	49.21	55.48
8	7.00	15.00	3.00	81.77	81.93	15.91	39.80	55.77	58.39
9	0.30	10.00	2.00	88.64	88.20	0.75	21.78	-1.30	5.14
10	8.70	10.00	2.00	62.50	65.02	26.20	12.63	22.41	7.17
11	4.50	1.59	2.00	46.31	50.79	64.00	76.53	26.68	14.61
12	4.50	18.41	2.00	87.50	85.10	67.06	61.99	60.40	63.67
13	4.50	10.00	0.32	82.29	82.37	58.78	59.69	82.18	86.10
14	4.50	10.00	3.68	76.17	78.17	64.04	70.59	68.53	55.80
15	4.50	10.00	2.00	71.88	72.01	57.88	54.18	40.41	41.94
16	4.50	10.00	2.00	72.50	72.01	51.77	54.18	41.96	41.94

 x_1 , initial pH; x_2 , quantity of aluminum dross (g); x_3 , initial oil concentration (% v/v); Y^a , observed value; Y^b , predicted value; COD, chemical oxygen demand; TOC, total organic carbon.

Table 3 ANOVA test results for COD levels.

Source	Sum of squares	Degrees of freedom	Mean square	F-value	$F_{ m tabulated}$	<i>p</i> -value
Model	2369.43	9	263.27	24.77	4.10	0.0005
pН	648.96	1	648.96	61.04		0.0002
Al dross	1421.54	1	1421.54	133.70		0.0000
Oil	21.24	1	21.24	2.00		0.2072
pH^2	24.29	1	24.49	2.30		0.1799
Al dross ²	19.15	1	19.15	1.80		0.2281
Oil^2	78.98	1	78.98	7.43		0.0344
pH*Al dross	8.08	1	8.08	0.76		0.4168
pH*Oil	92.21	1	92.21	8.67		0.0258
Al dross*Oil	0.95	1	0.95	0.09		0.7748
Residual	63.79	6	10.63	_		_
Lack of fit	63.60	5	12.72	66.18	230.16	0.0930
Pure error	0.19	1	0.19	_	-	_
Total	2433.22	15				

 $R^2 = 0.9738$ R^2 adj = 0.9345

ANOVA, analysis of variance.

more accessible binding sites for adsorption and an increase in the likelihood of collisions between adsorbent particles and oil droplets. Although the number of binding sites accessible for oil adsorption increased, the number of binding sites available for oil adsorption remained constant. Therefore, the adsorption capacity of aluminum dross increased from 15 to 20 g. This might be attributable to the adsorbent particles in the emulsion being overcrowded, causing the adsorption sites to overlap [11]. In addition, Fig. 1c shows the relationship between the quantity of aluminum dross and the cutting oil concentration at pH 2. The treatment of 15 g of aluminum dross at pH 2 with initial

concentrations ranging from 1% to 3% v/v resulted in a decrease in removal efficiency due to the high initial oil concentration.

Optimization of adsorption

Cutting oil was diluted with water and turned into oil in a water emulsifier. As a result, surface tension between the water and the oil phases was reduced, which improved oil adsorption [12]. COD, TOC, and turbidity reductions were observed in the oil wastewater. The optimum conditions were successfully achieved at run 3 with conditions of pH 2, 15 g of aluminum dross with a size in the range of 0.2–0.5 mm, and an initial cutting

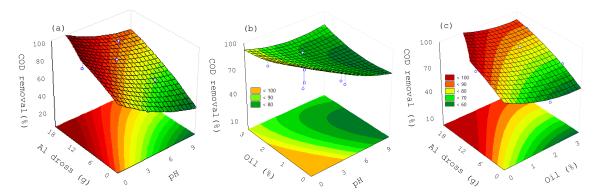


Fig. 1 Response surfaces determined using the CCRD method with Eq. (4) for the COD adsorption efficiency (%) after treatment with aluminum dross as the adsorbent for the combined effect of (a) the quantity of aluminum dross and initial pH, (b) initial cutting oil concentration (%) and initial pH, and (c) the quantity of aluminum dross and initial cutting oil concentration ($\frac{4}{3}$ v/v).

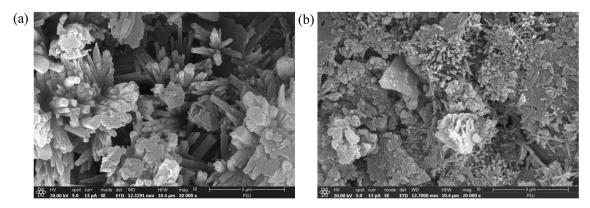


Fig. 2 SEM image of aluminum dross with size in the range of 0.2–0.5 mm (a) before and (b) after treatment. The magnification in both images is $20000 \times$.

oil concentration of 1% within 60 min. Consequently, the highest removal efficiencies of 93.30%, 88.33%, and 40.47% were achieved for COD, turbidity, and TOC, respectively. The comparison of COD removal efficiency under different conditions was shown in Fig. S2.

pH is a critical factor in oil adsorption, particularly for a higher degree of de-emulsification and electrostatic interactions under acidic conditions; the emulsion pH influences emulsion breaking and the surface of the adsorbent. Our results confirmed that the highest COD removal was achieved under acidic conditions. This could be explained by the influence of de-emulsification on the cutting oil concentration to obtain free oil molecules in an oil-in-water emulsion. Considering that the final pH at the optimum condition was >7 after adsorption process, this was higher than the pH_{zpc} of aluminum dross of \sim 6.42. The surface of aluminum dross might have had negative charges, and oil adsorption was not favored because of the leading repulsive force between the negative surface of the adsorbent and the oil molecules. When the surface charge of the adsorbent became increasingly negative with increasing pH, the adsorption could not be favored because of deprotonation [12]. Whereas COD removal in produced water with pH < pH_{zpc}, the COD was efficiently removed because of the protonated oxygenated functional groups [24]. However, previous studies reported that COD removal from wastewater had a practical pH in the range of 7–8. The type of adsorbent and initial wastewater pH did not significantly affect the adsorption process [23].

Two heavy metals, Al (43.08 mg/l) and Si (0.385 mg/l), were detected in the treated aliquots. The SEM image (Fig. 2) depicted the changes in the aluminum dross surface layer following the adsorption process; the surface was completely adhered to the adsorbent surface and covered with a collection of oil cavities. The XRF results revealed that the Al and Si contents increased slightly from 37.13% to 55.87% and 13.01% to 7.77%, respectively. Aluminum dross demonstrated an Al 0 state in the form of Al, aluminum oxide (Al $_{2}O_{3}$), magnesium aluminum oxide (MgAl $_{2}O_{4}$), and aluminum hydroxide (Al(OH) $_{3}$) and

includes Si, silicon oxide (SiO_2), sodium magnesium fluoride ($NaMgF_3$), and manganese sulfite hydrate ($MnSO_3 \cdot 2H_2O$).

Adsorption kinetics

Under optimum conditions, rapid adsorption with aluminum dross was completed within 10 min and slowed down until equilibrium was reached at 60 min, which was a shorter time compared with the adsorbed quantity of oil increased with the contact time and reached equilibrium after 2 h with corncob and modified sepiolite [13, 15]. The linearized pseudo-second-order model had a higher correlation coefficient (R^2) than the pseudo-first-order model. Table S1 summarizes the parameters calculated for each model. The adsorption kinetics fitted the pseudo-second-order reaction well, with an R^2 of 0.976 and a pseudo-second-order decay rate constant of 0.002 g/mg.min (Fig. S3). The adsorption of COD on the aluminum dross surface was maintained to best fit the pseudo-second-order kinetic model. This indicateed the chemisorption of the COD on the aluminum dross surface, and valence forces and ion exchange between the COD and adsorbents could have occurred [13].

Adsorption isotherm

The adsorption isotherm is a plot illustrating the relationship between the adsorbate concentration in the aqueous solution and the quantity adsorbed in the adsorbent when the adsorbent and adsorbate are in equilibrium [13]. Langmuir and Freundlich isotherms were used to examine the equilibrium data in this study, and the results were listed in Table S2.

The linear correlation coefficient (R^2) of the Langmuir model was higher than that of the Freundlich model, implying that COD adsorption on aluminum dross followed the monolayer process. Previous research showed that the Langmuir isotherm model could explain the link between the adsorption of oil molecules with metal ions in biopolymers [12]. Then, the adsorption mechanism was monolayer and chemisorption, similar to that of COD in oil with chitosan beads [25] and the physical monolayer adsorption mechanism of oil on wheat and rice straw [11].

In this study, the maximum adsorption capacity for COD removal in cutting oil wastewater was 3.33 g/g, which was greater than wheat straw, rice straw [11], and modified sepiolite [15]. The operation cost was evaluated using a cost-effectiveness method based on chemical costs such as those of the pH reagents and commercial-grade chemicals and operating costs such as that of electricity. The operating cost of the adsorption with aluminum dross at 0.5398 USD/m³ for cutting oil wastewater treatment was lower than those for other treatment processes; for example, electrocoagulation using Al and Fe electrodes [26, 27]. Consequently, low-cost wastewater technologies that employ

waste as an adsorbent can be used to treat wastewater in auto parts manufacturing processes. Then, granular materials with particulate sizes greater than 0.2 mm can be utilized in column adsorption for practical purposes [28].

CONCLUSION

Non-modified aluminum dross from auto parts manufacture was utilized as an adsorbent to eliminate pollutants from cutting oil wastewater. The experimental design, with combined CCRD and RSM, was effectively employed in this research to optimize critical variables, including initial cutting oil concentration, initial pH, and quantity of aluminum dross. A COD response model was developed to ensure appropriate predictability. The optimum conditions at pH 2, 15 g of aluminum dross, and 1% v/v of initial oil concentration were achieved at the maximum removal efficiencies within 60 min. For further study, column treatment and operation parameters will be examined for industrially practical adsorption methods to remove highpollutant wastewater loading. Moreover, there are many different types of waste generated during the production of auto parts, such as shot blast, salt cake, and black dross; hence, the potential of using single or multiple adsorbents for industrial applications should be examined. In addition, the recovery of white aluminum dross can recover zero-valent aluminum (ZVAl), which can be used in a single or bimetallic system in advanced oxidation processes (AOP) like the Fenton reaction to remove pollutants. The treatment process in this study was cost-effective with respect to chemical costs; and, in particular, it reduced the costs of waste transportation and disposal, subsequently contributing to the circular economy.

Appendix A. Supplementary data

Supplementary data associated with this article can be found at http://dx.doi.org/10.2306/scienceasia1513-1874. 2024.053.

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Appendix A. Supplementary data

 Table S1
 Parameters of the adsorption kinetics model for COD adsorption on aluminum dross.

Model	Pseudo-First-Order			Pseudo-Second-Order			
Parameters	$k_1 \text{ (min}^{-1}\text{)}$	q _e (mg/g)	R^2	k_2 (g/mg.min)	q _{e,cal} (mg/g)	q _{e,exp} (mg/g)	R^2
Aluminum dross	0.128	65.536	0.893	0.002	90.090	87.893	0.976

Table S2 Isotherm model parameters for the adsorption of COD on aluminum dross.

Adsorbents		Langmuir			Freundlich			
	q ₀ (g/g)	K _L (L/mg)	R^2	n	K _F (L/g)	R^2		
Aluminum dross, 15 g	3.33	0.0272	0.9953	1.0794	0.1493	0.9903		

COD, chemical oxygen demand

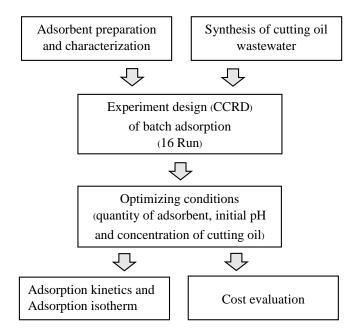


Fig. S1 The diagram of the experiment.

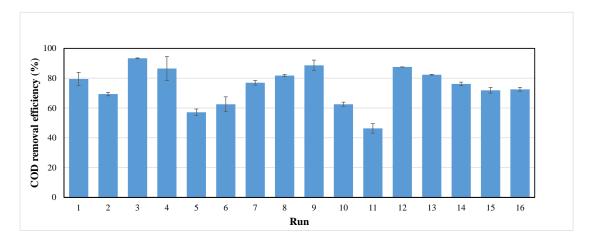
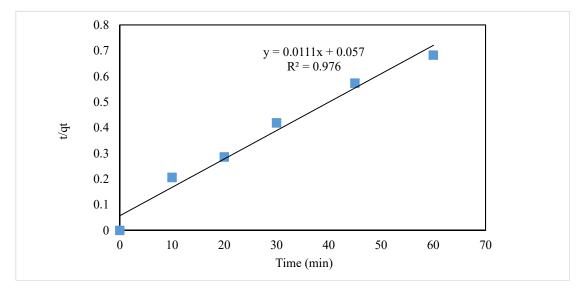


Fig. S2 Removal efficiency of COD under different conditions using CCRD design.



 $\textbf{Fig. S3} \ \ \textbf{The plot of pseudo-second order kinetic at optimum condition}.$