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The Role of Boron-Doped Diamond and Platinum Anodes in the Three-Compartment Electrochemical Pretreatment of Stabilized Landfill Leachate – Response Surface Methodological Approach

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ABSTRACT

Stabilized landfill leachate contains high fractions of refractory organics that cannot be effectively degraded by simple biological or physicochemical treatment. Thus, primary treatment was required to improve biodegradability and enhance treatment efficiency. This study investigated the role of Boron-Doped Diamond (BDD) and platinum (Pt) anodes at a current density of 29.2 and 33.3 mA/cm^2 in the electrochemical processes for the pretreatment of stabilized leachate. A three-compartment electrochemical reactor was used in the research to enhance the removal of ionic pollutants. The pollutants were measured as total dissolved solids (TDS), chemical oxygen demand (COD), ammonium-nitrogen (NH_4–N), and nitrite (NO_2^-). The reactor performance was then analyzed using a regular two-level factorial design. The results showed that the electrochemical process effectively removed organic and inorganic pollutants. The highest removal was obtained at 33.3 mA/cm^2 using the BDD, measured around 48, 82, 60, and 79% for TDS, COD, NH_4–N, and NO_2^-, respectively. Meanwhile, the specific energy consumption for COD removal was estimated to reach 1.5 and 1.55 Wh/g for BDD and Pt, respectively. These results imply that the type of anodes and applied current densities significantly influence the treatment efficiency.

Keywords: boron-doped diamond, electrochemical process, leachate pretreatment, platinum, response surface methodology.

INTRODUCTION

Landfill leachate has created many problems due to its significant characteristic variations related to the type of waste, landfill age, landfilling technology, climate and hydrogeological conditions (Gómez et al., 2019; Kjeldsen et al., 2002; Mukherjee et al., 2015; Veli et al., 2021). Without proper treatments, the persistence and continuous flow of leachate from the active or closed landfill sites along with high loads of organic matter, ammonia-nitrogen, inorganic salt, heavy metals, and recalcitrant organics could pose some detrimental effects on human health and the environment (Deng et al., 2020; Hussein et al., 2019). The biodegradability of leachate will decrease along with the increase in landfill age. Young leachate tends to have a high biodegradability (BOD_5/COD ratio > 0.6), while old and stabilized leachate have a lower biodegradability (BOD_5/COD ratio < 0.3) and more complex structure of organic and inorganic compounds (Deng and Englehardt, 2007; Mukherjee et al., 2015). Simple biological and physicochemical treatments are typically adequate for young leachate but become insufficient for the stabilized leachate. In the past two decades, the integration of physicochemical and biological processes for stabilized leachate treatment has gained interest due to higher treatment efficacy and acceptable cost efficiency (Bagastyo et al., 2021a; Ukundimana et al., 2018; Zhao et al., 2010). One of the technologies that can be used as a pretreatment or post-treatment of landfill leachate is electrochemical oxidation (EO). This technology offers some advantages, e.g., high

versatility, selectivity, energy efficiency, cost-effectiveness, stability, no sludge production, and without chemical addition (Zambrano and Min, 2020; Zhao et al., 2010). The performance of EO may be affected by several factors, including the type of anode materials, current density, pH, reactor configuration, chloride concentration, applied voltage, flow rate, electrolysis time, and temperature (Deng and Englehardt, 2007; Mandal et al., 2017). The typical anode materials used for leachate treatment are BDD, IrO_2, RuO_2, Pt, SnO_2, PbO_2, and graphite (Mandal et al., 2017). However, BDD and Pt are mainly studied because they have good chemical stability, resistance to anodic corrosion, are cost-effective, and can accelerate the degradation of organic materials (Agustina et al., 2019; Fernandes et al., 2015; Oturan et al., 2015; Patel et al., 2013; Zambrano and Min, 2020; Zhou et al., 2016).

In the presence of ionic pollutants, such as dissolved salts and ionic nitrogen compounds in the leachate, a membrane-divided electrochemical reactor can be applied to eliminate the remaining ionic nitrogenous species or other ionic species from the treated leachate (Bagastyo et al., 2022). This reactor configuration is capable of concentrating the targeted pollutants into a dedicated compartment as recoverable compounds. In this study, a three-compartment reactor configuration was used by inserting ion exchange membranes between the compartments of EO system. However, there is a lack of knowledge regarding the role of electrodes and current density in the application of three-compartment electrochemical reactor as leachate pretreatment by means of response surface method (RSM) approach. Therefore, the objective of this study is to investigate the effect and interaction of anodes materials (i.e., BDD and Pt) and applied current density on the removal of pollutants, i.e., measured as TDS, COD, NH_4–N and NO_2^-. An RSM with a regular two-level factorial design was used to develop the model and analyze the effect of variables in the electrochemical processes. In addition, the required energy consumption was also calculated to determine the treatment efficiency.

MATERIALS AND METHODS

Leachate characteristics

Leachate was obtained from a solid waste landfill site in Pasuruan, Indonesia. This landfill has been operated since 1996 and currently receives around 47,000 m^3 of municipal solid waste yearly. Leachate samples were collected from the leachate basin in the landfill site during the rainy season. It is then transported to the laboratory and immediately stored in the refrigerator at 4°C to minimize microbial activity. Table 1 illustrates the physicochemical characteristics of raw leachate.

Experimental setup

The electrochemical processes were carried out in a three-cell compartment and operated in a continuous recirculation system for 12 h. The dimensions of the anode and cathode compartments were 18 × 8 × 2 cm (effective volume 576 mL), respectively, whereas the dimension of the concentrate compartment was 18 × 8 × 1 cm. Ion exchange membranes, i.e., AMI-7001 and CEM-7000 (Membrane International Inc., USA), were used in the experiments. BDD and Pt were applied as the anode, while stainless steel (A-304) was employed as the cathode. Each electrode has a surface area of 24 cm^2 (8 × 3 cm) and a 2 cm inter-electrode gap. The experimental setup is described in Figure 1.

The electrochemical processes were operated galvanostatically using an adjustable 0–30 V DC power supply (LWK 305D, Cody, China) at an applied current of 700 and 800 mA (current density of 29.2 and 33.3 mA/cm^2, respectively). A peristaltic pump was used to recirculate 5 L of leachate and 5 L of buffer solution (i.e., 0.0133 M of $NaH_2PO_4.7H_2O$ and 0.0117 M of $Na_2HPO_4.H_2O$) in the separated feed and concentrated tank, respectively, at the same flow rate of 2 mL/s.

Analytical methods

A 0.45 μm PTFE membrane syringe filter was used to filter the sample before each analysis. All analyses were carried out according to the standard method (American Public Health Association, 2005). The pH, DO, and conductivity was measured using a YK-2005 pH meter (Lutron, Taiwan). The concentration of COD was measured using the colorimetric method and Spectroquant Nova–60 A analyzer (Merck). The concentrations of nitrogenous compounds (e.g., ammonium, nitrite, and nitrate) were determined by the Nessler method using a UV–Vis spectrophotometer (Genesys 30, Thermo Fisher Scientific, USA).

Table 1. Raw leachate characteristics

Parameter	Unit	Amount
BOD	mg/L	8,466
COD	mg/L	16,400
Ammonium	mg/L	390.8
Nitrite	mg/L	133.2
Nitrate	mg/L	82.1
TDS	mg/L	10,163
Salinity	ppt	12.12
pH	–	8.4

Design of experiment

The Design-Expert software (version 11.1.2.0, Stat-Ease, Inc., USA) was applied to develop the experimental design and perform data analysis. The factors included in the model were anode materials (i.e., BDD and Pt) and current density (29.2 and 33.3 mA/cm^2). A two-level factorial design was used to accommodate the small number of factors. This is the most efficient design because it could replicate the experiment through all combination levels of investigated factors (Montgomery, 2013). Moreover, the effect of a factor can be estimated at numerous levels of other factors, resulting in a valid conclusion over a range of experimental conditions. A total of 12 runs were carried out through 3 replicates on each combination factor. The 2FI polynomial model was selected to perform the statistical analysis. The regression can be described as follows (Montgomery, 2013):

$$y = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + \beta_{12} x_1 x_2 + \varepsilon \quad (1)$$

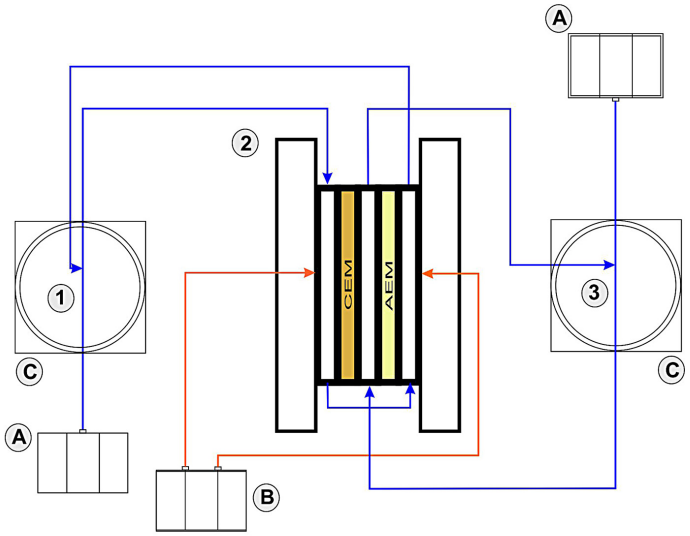
where: y – the response, β – the constant coefficient on each parameter, x_1 represents factor A, x_2 represents factor B, and ε is the error term.

The experimental matrix is shown in Table 2. The analysis of variance (ANOVA) was carried out to assess the developed model and evaluate the interaction of factors. The statistical significance is checked by the Fischer's F-test, where confidence levels of 95% (p-value <0.05) are considered significant for the model (Ahmad et al., 2021; Lessoued et al., 2017).

RESULTS AND DISCUSSIONS

RSM model fitting

Table 3 depicts the ANOVA results of the developed model. As seen in the Table, the model of each response significantly fit a p-value <0.05.



Notes:
1 : leachate feed tank (5 L)
2 : electrochemical reactor
3 : concentrate feed tank (5 L)
A : peristaltic pump
B : DC supply
C : magnetic stirrer

Figure 1. Experimental setup of electrochemical reactor

Table 2. Factor and response values used in the developed model

Runs	Electrode (x_1)	Current density (x_2)	Responses			
			TDS (%)	COD (%)	NH_4–N (%)	NO_2^- (%)
1	Pt	33.3	46.7	77.0	53.5	76.5
2	Pt	29.2	31.4	75.2	18.8	65.5
3	Pt	29.2	35.9	74.3	13.5	62.3
4	Pt	33.3	47.8	77.7	53.2	74.9
5	BDD	33.3	48.4	82.2	51.4	79.0
6	BDD	29.2	39.0	79.9	51.6	79.5
7	BDD	29.2	37.8	80.2	56.3	79.3
8	Pt	33.3	45.3	76.8	52.8	75.3
9	BDD	33.3	48.3	82.3	60.6	79.5
10	BDD	29.2	39.5	80.1	54.3	79.3
11	Pt	29.2	35.3	75.5	12.6	66.5
12	BDD	33.3	42.9	82.6	56.5	79.1

The p-value <0.0001 presented on COD, NH_4–N, and NO_2^- indicates that the model significantly suits the experimental data. R^2 value defines the efficiency of a model in predicting the response. It is said to be desirable when the value of R^2 is close to 1 (Ahmad et al., 2021). In this study, the R^2 values for TDS, COD, NH_4–N, and NO_2^- were 0.9039, 0.9862, 0.9787, and 0.9737, respectively. Meanwhile, the adjusted R^2 was 0.8678 (TDS), 0.9811 (COD), 0.9707 (NH_4–N), and 0.9639 (NO_2^-). Compared to R^2, the value of adjusted R^2 would diminish if nonsignificant factors are added to the model (Montgomery, 2013). On the other hand, the differences between adjusted R^2 and predicted R^2 for all responses were less than 0.2, indicating a close agreement and implying that the model could predict the response value. Although the pure error was detected in the model, no lack-of-fit can be analyzed in the TDS, NH_4–N, and NO_2^- removal. On the contrary, the lack-of-fit observed in COD removal was 0.0489 and 0.8305 for F-value and p-value, respectively. The COD lack-of-fit will be described further in the following subsection. The coefficient of variation (C.V.) obtained in each response was less than 10%. It means that all models significantly have good reproducibility (Ahmad et al., 2021). Additionally, the adequate precision for TDS, COD, NH_4–N, and NO_2^- were 10.1622, 31.5020, 23.0345, and 21.1635, respectively. Theoretically, an adequate precision greater than 4 is desirable to measure the signal-to-noise ratio (Montgomery, 2013). Thus, it can be concluded that the obtained adequate precision can be used to navigate the model. Furthermore, the normal probability plot (Figure 2) showed that most points in each response were close to the straight line. These results indicate that normal distribution adequately fits the data.

The effect of electrodes and current density on the EO process

The effects of factors on the responses are described through the Pareto chart (Figure 3). Each factor above the Bonferroni limit is very important to the response (Hu et al., 2016). At the same time, the factor above the t-value limit is possibly important and should be added to the model. In contrast,

Table 3. ANOVA of the developed model

Model response	Sum of squares	df	Mean square	F-value	Prob. > F-value	S.D	C.V. (%)	Adj. R^2	Pred. R^2	A.P
TDS	336.26	3	112.09	25.08	0.0002	2.11	5.09	0.8678	0.7837	10.1622
COD	94.41	3	31.47	191.02	< 0.0001	0.41	0.52	0.9811	0.9690	31.5020
NH_4-N	3521.98	3	113.99	122.37	< 0.0001	3.10	6.94	0.9707	0.9520	23.0345
NO_2^-	425.01	3	141.67	98.89	< 0.0001	1.20	1.60	0.9639	0.9409	21.1635

Note: df – degree of freedom; S.D – standard deviation; C.V. – coefficient of variation; Adj. R^2 – adjusted R^2; Pred. R^2 – predicted R^2; A.P. – adequate precision.

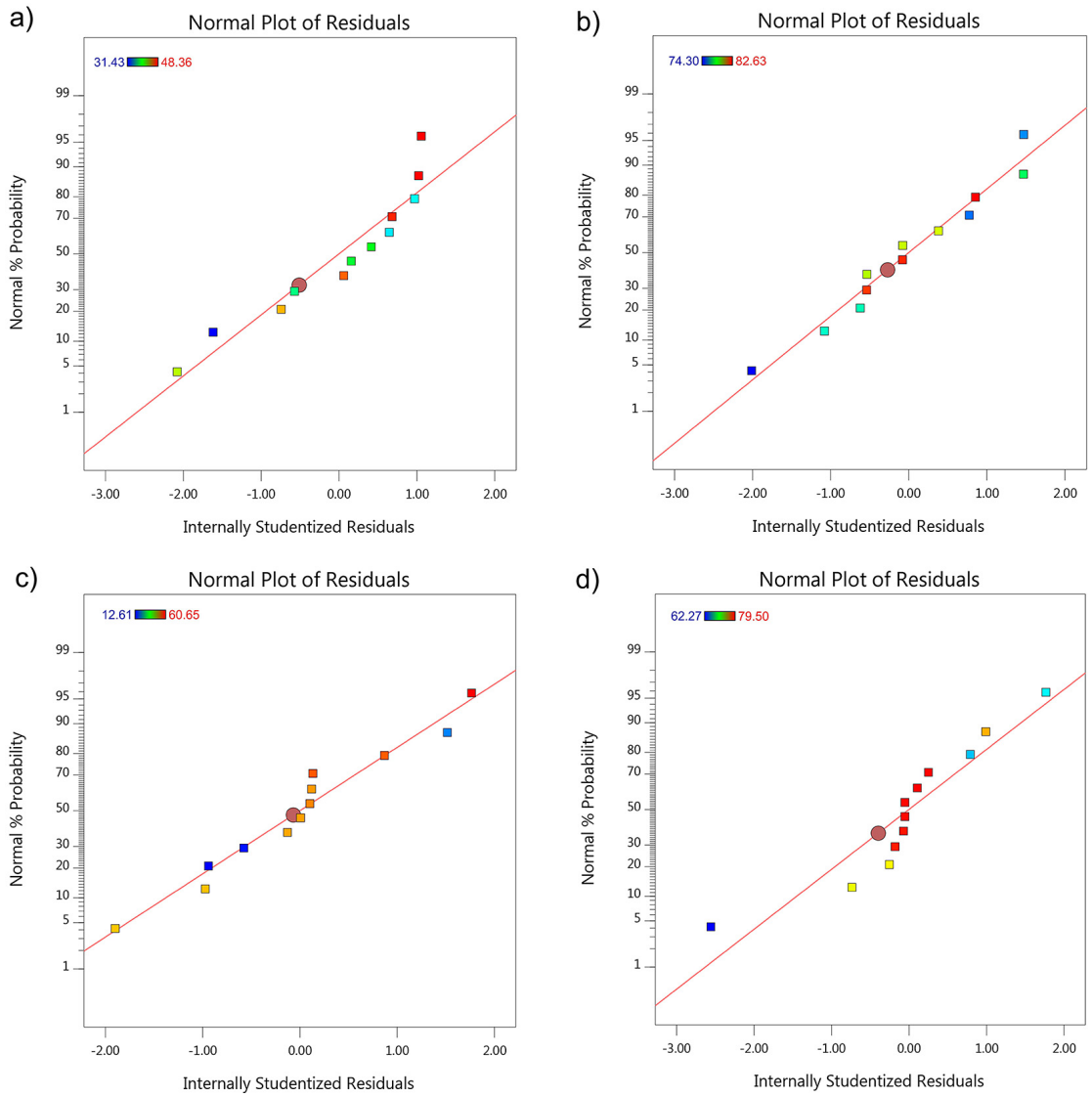


Figure 2. Normal probability plot for a) TDS, b) COD, c) NH_4–N and d) NO_2^- removal

any factor below the t-value limit may not be important to the response but could be forced into the model to support the model hierarchy.

In Figure 3a, the effect of current density was far above the Bonferroni limits, indicating a significant effect on the TDS removal. On the other hand, the effect of electrodes and the interaction factor of electrode/current density were below the t-value limit. These factors should be forced into the model to support the hierarchy. Otherwise, the model would not fit, indicated by a relatively low probability of lack-of-fit (p-value < 10%). Regarding COD removal (Figure 3b), the effect of the electrodes and current density was above the Bonferroni limit, while their interaction was below the t-value limit. Since the interaction effect was insignificant, it can be excluded from the statistical analysis. Regarding NH_4–N and NO_2^- removal, all bars in Figures 3c and 3d were above the Bonferroni limit, indicating a highly significant effect of all factors on the responses. A more detailed significant effect of each factor and their interaction with the response can be seen in Table 4–7. Among all results, higher current density appears to improve the oxidation efficiency regardless of electrode type. Higher current density allows the increase of anode potential and the change of the oxidation mechanism of organic pollutants. Higher generation of electrogenerated oxidant and hydroxyl radicals through the direct and indirect oxidation processes promotes the removal of organic matter and the whole efficiency process. Contrarily, the lower current density may not be conducive to the oxidation process, particularly when the electrode potential does not meet the requirement of a partial oxidation reaction (Fernandes et al., 2015).

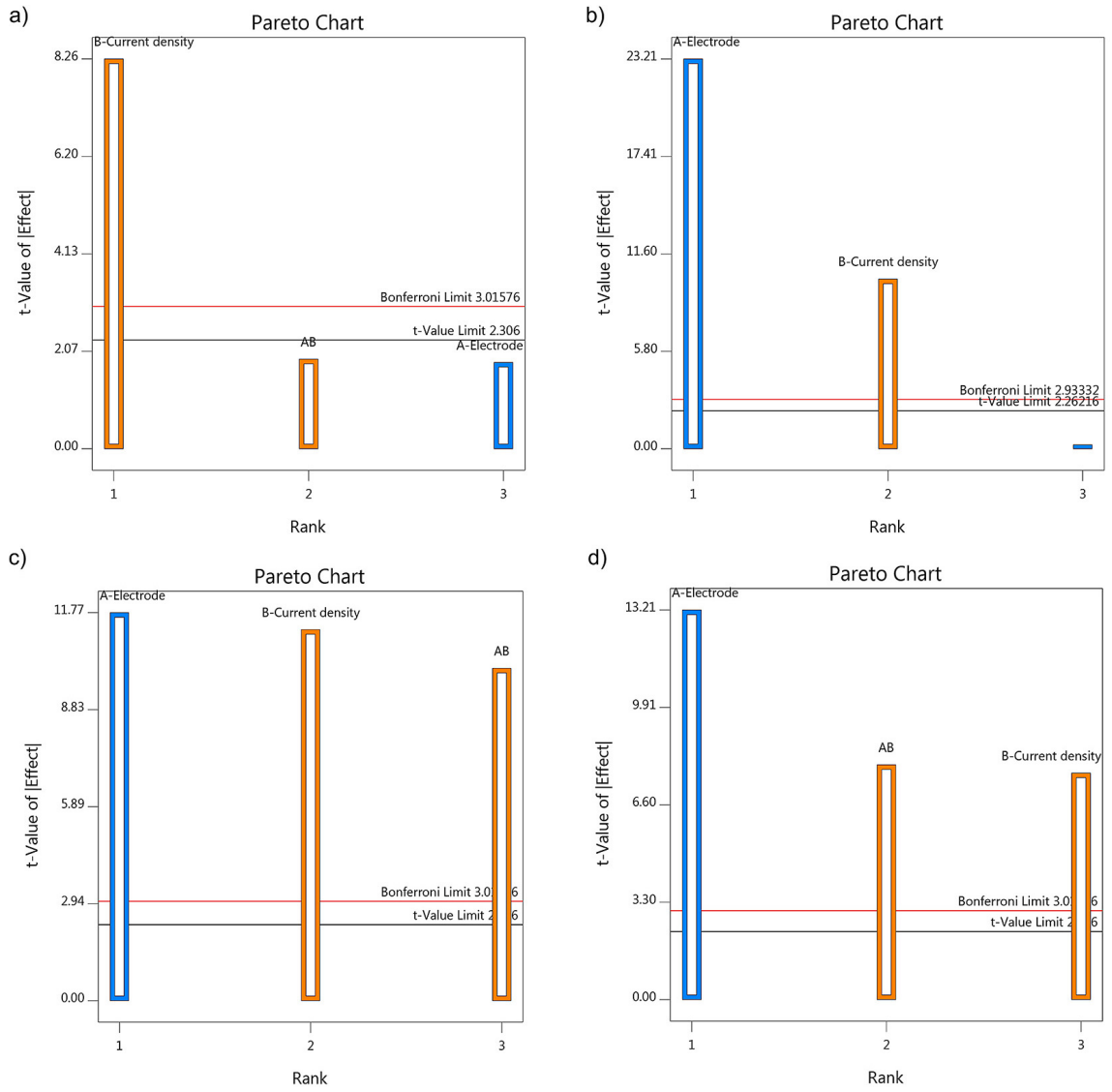


Figure 3. Pareto chart for a) TDS, b) COD, c) NH_4–N and d) NO_2^- removal. (Note: blue indicates negative effects, orange indicates positive effects)

Table 4. ANOVA for a factorial model of TDS removal

Source	Sum of squares	df	Mean square	F-value	p-value
Model	336.26	3	112.09	25.08	0.0002*
A-Electrode	14.97	1	14.97	3.35	0.1047
B-Current density	305.16	1	305.16	68.27	< 0.0001*
AB	16.13	1	16.13	3.61	0.0940
Pure Error	35.76	8	4.47		
Cor total	372.02	11			

Note: * significance.

Interaction effect of electrodes and current density to the responses

The interaction of electrodes and current densities on the pollutants removal is illustrated in Figure 4. As seen in Figure 4a, similar efficiency removal of TDS was observed at Pt and BDD electrodes at a current density of 33.3 mA/cm^2, i.e., ~46% on average. Meanwhile, at 29.2 mA/cm^2, the efficiency removals measured on both electrodes were down to 34% for Pt and 38% for BDD. These results indicate that the current

Table 5. ANOVA for a factorial model of COD removal

Source	Sum of squares	df	Mean square	F-value	p-value
Model	94.41	3	31.47	191.02	< 0.0001*
A-Electrode	79.36	1	79.36	481.72	< 0.0001*
B-Current density	15.04	1	15.04	91.28	< 0.0001*
AB	0.0081	1	0.0081	0.0489	0.8305
Pure Error	1.32	8	0.1647		
Cor Total	95.72	11			

Note: * significance.

Table 6. ANOVA for a factorial model of ammonium removal

Source	Sum of squares	df	Mean square	F-value	p-value
Model	3521.98	3	1173.99	122.37	< 0.0001*
A-Electrode	1330.14	1	1330.14	138.65	< 0.0001*
B-Current density	1216.28	1	1216.28	126.78	< 0.0001*
AB	975.56	1	975.56	101.69	< 0.0001*
Pure Error	76.75	8	9.59		
Cor Total	3598.73	11			

Note: * significance.

Table 7. ANOVA for a factorial model of nitrite removal

Source	Sum of squares	df	Mean square	F-value	p-value
Model	425.01	3	141.67	98.89	< 0.0001*
A-Electrode	249.89	1	249.89	174.43	< 0.0001*
B-Current density	84.44	1	84.44	58.94	< 0.0001*
AB	90.68	1	90.68	63.30	< 0.0001*
Pure Error	11.46	8	1.43		
Cor Total	436.47	11			

Note: * significance.

density and electrode's interaction effect were slightly significant for TDS removal. Compared to TDS removal, the interaction effect of current density and electrode on COD removal was more profound. According to Figure 4b, despite current density changes, the highest COD removal was shown on the BDD electrode. The removal of COD was recorded at over 80% using BDD and up to 77% with Pt. Other studies achieved similar results, i.e., 90 and 60% COD removal using BDD and Pt electrodes, respectively (Anglada et al., 2009; Feki et al., 2009; Ukundimana et al., 2018). The superiority of BDD over Pt to remove organic pollutants is attributed to inert surface and higher overpotential for oxygen evolution. The weakly adsorbed hydroxyl radicals on the inert surface of BDD promote more reaction of hydroxyl radicals with organic pollutants, consequently improving its oxidation efficiency (Deng et al., 2020; Panizza and Cerisola, 2009). The mechanisms are as follows:

$$BDD + H_2O \rightarrow BDD(\bullet OH) + H^+ + e^- \quad (2)$$

$$BDD(\bullet OH) + organics \rightarrow BDD + CO_2 + H_2O + others \quad (3)$$

$$BDD(\bullet OH) \rightarrow BDD + 0.5O_2 + H^+ + e^- \quad (4)$$

On the contrary, the Pt electrode has a nature of lower overpotential for oxygen evolution and strong interaction with the adsorbed hydroxyl radicals (Panizza and Cerisola, 2009; Panizza and Martinez-Huitle, 2013). These disadvantages induce the oxidation of hydroxyl radicals on the surface of the anode and lead to partial oxidation of organic pollutants. Regarding nitrogenous compounds, the differences in removal efficiency using BDD and Pt electrodes at 33.3 mA/cm^2 were minor. However,

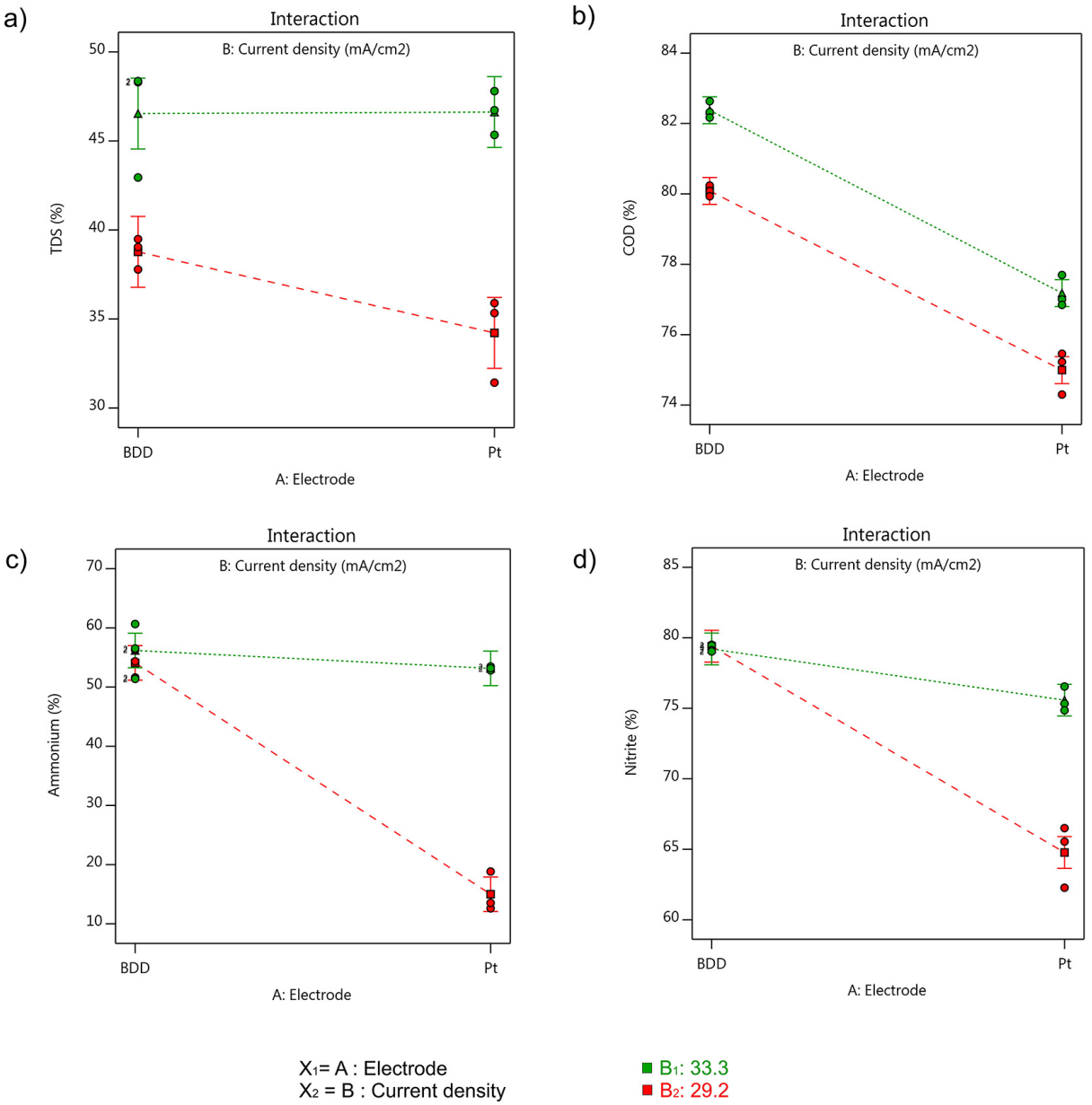


Figure 4. Interaction between factors on a) TDS, b) COD, c) NH_4–N and d) NO_2^- removal

when the current density decreases, significant differences in removal efficiency were observed; see Figures 4c and 4d. These results somewhat contradict other studies which revealed Pt could outperform BDD in terms of ammonium removal. In some studies, the removal of ammonium was between 80–100% with Pt (Feki et al., 2009; Zambrano and Min, 2020) and 34–74% with BDD electrodes (Anglada et al., 2011; Fernandes et al., 2014; Zhou et al., 2016). Typically, metal oxide electrodes such as Pt have higher total nitrogen removal efficiency than BDD. Under the Pt electrode, the main product of ammonium-N oxidation was nitrogen. While under the BDD electrode, the oxidation process resulted in an increased ratio of ammonium to nitrate (Deborde and von Gunten, 2008; Deng et al., 2020). Nonetheless, the 3D surface plots of the pollutant removals are shown in Figure 5.

Energy consumption

The role of anode material and current density on the leachate treatment efficiency was also evaluated using the specific energy consumption (E_{sp}), as seen in Eq. 5 (Bagastyo et al., 2021b).

$$E_{sp}(Wh/g) = \frac{V\ i\ \Delta t}{\Delta m} \quad (5)$$

where: V is the applied voltage (v); I is the applied current (A); Δt is the duration of electrolysis (h); Δm is the removed pollutant mass (g).

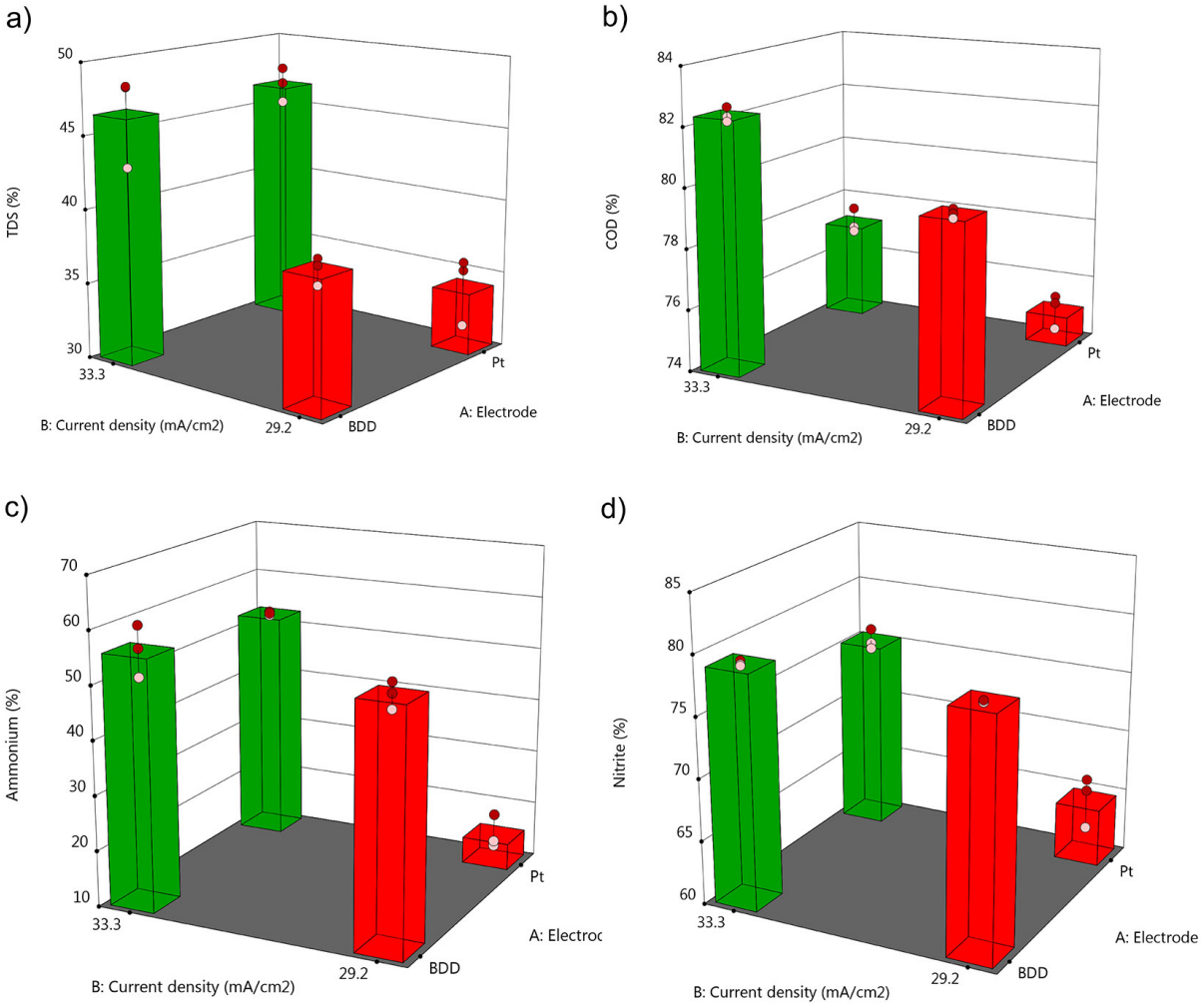


Figure 5. 3D surface plot for a) TDS, b) COD, c) NH_4–N and d) NO_2^- removal

As seen in Table 8, increasing current density significantly enhances the specific energy consumption on both electrodes. In the BDD electrode, the specific energy consumption for COD removal increases by 32% when the current density increases to 33.3 mA/cm^2. Likewise, a 50% energy increase was observed in the Pt electrode. The removal percentages of COD in both electrodes were slightly similar; thus, the effect of electrodes on the energy consumption was insignificant. On average, the specific energy consumption for COD removal obtained in this study was lower than other studies that achieved 9.4–62.9 Wh/g COD using BDD (Agustina et al., 2019; Bagastyo et al., 2020). However, the results are understandable since those studies were performed in a higher current density, i.e., 50–78 mA/cm^2. In addition, the effect of the electrode and current density on the energy consumption was more pronounced in the NH_4-N removal. At 29.2 mA/cm^2, the energy requirement was 82.45 and 252.13 Wh/g NH_4-N for BDD and Pt

Table 8. Specific energy consumption

Anode	I (A)	J (mA/cm^2)	Voltage (V)	Average COD removed		Average NH_4-N removed		Specific energy consumption	
				(%)	(g)	(%)	(g)	Wh/g COD	Wh/g NH_4-N
BDD	0.7	29.2	8.93	80.09	65.67	54.08	0.91	1.14	82.45
	0.8	33.3	10.57	82.38	67.55	56.18	0.95	1.50	107.36
Pt	0.7	29.2	7.57	74.99	61.50	14.99	0.25	1.03	252.13
	0.8	33.3	10.23	77.18	63.29	53.16	0.89	1.55	109.82

electrodes, respectively. When the current density increased to 33.3 mA/cm^2, the energy consumption in the BDD electrode was enhanced by 30%, while the energy consumption in the Pt electrode was decreased by 56%. These results justified the previous statistical analysis, which indicates that the electrodes and current density and their integration significantly influenced NH_4–N removal.

CONCLUSION

A significant amount of pollutants (e.g., measured as TDS, COD, NH_4–N, and NO_2^-) has been successfully removed from the stabilized landfill leachate by using a three-compartment electrochemical reactor. The evaluation of both anode materials and current density by using a two-level factorial design yields an R^2 value of 90.39% (TDS), 98.62% (COD), 97.87% (NH_4–N), and 97.37% (NO_2^-). Furthermore, the results suggested that the influence of current density was higher for TDS removal compared to the anode materials. Regarding the COD removal, although the individual factor of electrodes and current density have significant influences, the integration of both factors was identified as insignificant. In contrast to the TDS and COD removals, the individual factor of electrodes and current density and their integration significantly affected to the removal of NH_4–N and NO_2^- compounds. Among the anode materials and current densities that have been tested, BDD and current density of 33.3 mA/cm^2 attained the highest removal efficiency of pollutants, which were approximately 48% (TDS), 82% (COD), 60% (NH_4–N), and 79% (NO_2^-).

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