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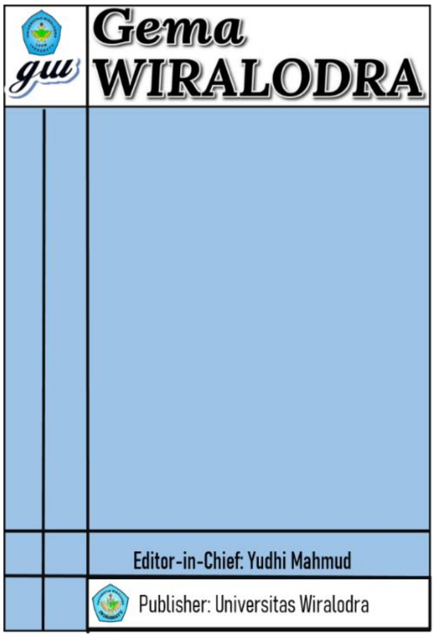

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Original Article

Removal of lithium, sodium, and phosphate from the Li-ion battery cathode recycling process using column adsorption

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Abstract

Lithium Ion Battery (LIB) is one type of battery whose demand is increasing every year. After the Li-ion battery usage cycle ends, more and more waste is generated. Wastewater from the LFP battery cathode recycling process still contains metals such as lithium, sodium, and phosphate. This experiment was carried out using the adsorption method. The adsorption method is used to remove metal ions in LFP battery waste. The adsorption process took place in a semi-continuous fixed-bed column made of glass cylinders with a height of 50 cm and a diameter of 2.5 cm. The artificial waste solution flowed into a column containing 135 g of activated carbon with variations in flow rates of 10, 15, and 20 mL/min for 2 hours. The concentration of the initial solution and the filtrate was analyzed using an Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES). The experimental results showed that the maximum percentage removal of lithium, sodium, and phosphate at a flow rate of 10 mL/min was 95.22%, 96.42%, and 68.02%, respectively. In addition, the results of the adsorption kinetic studies reviewed using the Thomas and Yoon-Nelson models showed that the column adsorption performance for lithium, sodium, and phosphate was most effective at a flow rate of 10 mL/min with an adsorption capacity of $8.75.10^5$ mg/g, $8.85.10^5$ mg/g, and $3.91.10^5$ mg/g, respectively. Meanwhile, the breakthrough times for lithium, sodium, and phosphate were 335 minutes, 441 minutes, and 126 minutes, respectively.

Keywords: Li-ion battery, column adsorption, activated carbon

1. Introduction

Lithium Ion Battery (LIB) is a secondary type of battery that can be recharged and is widely used as an energy source in electric cars, laptops, smartphones, and various other electronic equipment (Siqi et al., 2019). The global market for the LIB battery industry is growing rapidly due to the widespread use of electric vehicles (Bi et al., 2020). According to a forecast by the International Energy Agency, the number of electric vehicles on the world's roads will increase from 3 million to 125 million by 2030. Such an exponential increase in production volume and utilization of LIB poses recycling challenges of large volumes of end-of-life LIB e-waste generated (Yadav et al., 2020).

One type of LIB battery that is widely used is the LFP type battery. The lithium iron phosphate (LFP) battery was the first to be synthesized in 1996 and has gained many advantages in several applications such as energy storage, electronic equipment, and electric vehicles (Forte et al., 2021). Reporting from CNBC Indonesia, one of the electric car factories, Tesla, is reportedly developing an LFP-type battery. The reason for using these batteries is because the amount of raw materials iron and lithium is very abundant. The cathode material of the LFP battery has advantages including affordable price, non-toxic, easy to obtain, high thermal and chemical stability, stable electrochemical performance under fully charged conditions, and high energy density (170 mAh/g), as well as a long life cycle (Saleh and CNBC Indonesia, 2021).

LFP batteries are often referred to as environmentally friendly batteries because of their lower toxicity, however, they may still cause environmental problems (Yadav et al., 2020). Battery recycling is usually done only until the recovery of valuable metals (Li, Ni, Mn, Co). One of the most widely used recovery methods is the hydrometallurgical process. The hydrometallurgical process consists of several stages, namely pre-treatment, leaching, and purification. In general, the leaching process is carried out by adding an acid solution. Then the leaching solution is purified by precipitation (Elwert et al., 2019; Winslow et al., 2018). This leaching and refining process produces other waste which comes from the addition of chemicals used during the process.

In a study (Dyana et al., 2020) to extract lithium from the cathode of an LFP battery, leaching with phosphoric acid was used. Then to purify the leaching solution, it is carried out by multilevel precipitation using sodium hydroxide. The product obtained from the leaching and precipitation is a high-purity lithium compound in the form of 99% lithium phosphate. However, the leaching and refining process produces liquid waste containing various metals with varying levels. These metals come from the LFP battery cathode which contains not only lithium, but also other metals such as iron, phosphate, sodium, phosphate, and others. Therefore, further steps are needed to remove metals in wastewater from the LFP battery recycling process.

Wastewater from the battery recycling process contains compounds whose concentrations may be above acceptable environmental limits. This study aims to reduce the concentration of metals in wastewater from the leaching and purification of LFP battery cathodes by the adsorption process. Adsorption processes are widely used to remove contamination, including metals, from raw water (Patel, 2020). Adsorption using activated carbon is one of the oldest and most preferred techniques used for the removal of a wide variety of trace organics as well as nutrients from wastewater because of less cost, ease in operation, no by-products formation, no sludge generation, etc (Manjunath & Kumar, 2018). Activated carbon is used as an adsorbent due to its very high porosity, increased pore size and higher adsorption capacity (Guo et al., 2019). According to (Asuquo et al., 2017) the surface area of activated carbon ranges from 500 to 1500 m^2/g, making activated carbon very efficient for use in the adsorption process.

In this study, the adsorption feasibility of wastewater from LFP battery cathode recycling with activated carbon was analyzed using a fixed-bed adsorption column. Several process parameters such as flow rate and residence time were carried out to determine the percentage of contaminant removal and adsorption kinetics analysis.

2. Method

Material

Artificial waste is made based on data on the results of liquid waste resulting from the LFP battery recycling process. This artificial waste solution is made with a composition of lithium hydroxide, phosphoric acid, and sodium hydroxide. The three materials were prepared with concentrations similar to those of the liquid waste from the LFP battery recycling process as shown in table 1. The adsorbent used in the adsorption process was commercial activated carbon purchased from Pison Water Filter, Yogyakarta with a size of 8-30 mesh.

Table 1

LFP battery artificial liquid waste content

Ion content	Ion concentration [ppm]
Li	20
Na	900
P	

Table 1
BET characterization of activated carbon

Parameter	**Value**
Surface area, m^2/g	874.820
Total pore volume, cc/g	0.4458
Average pore diameter, nm	1.7976

Experiment detail

The adsorption column was made of a glass cylinder with a height of 50 cm and a diameter of 2.5 cm. A total of 135 g of activated carbon was put into the column. The artificial waste solution was pumped into the column at variations flow rates of 10, 15, and 20 mL/minute or residence times of 10.07 minutes, 6.71 minutes, and 5.03 minutes in a downflow direction by a peristaltic pump. Adsorption was carried out at room temperature. Adsorption samples were taken at various sampling points at intervals of 20 minutes for 2 hours. The filtrate was filtered using Whatman filter paper and then the concentration of the sample was measured using an Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES).

Data analysis

The concentration of metal ions contained in the adsorbed artificial waste solution was analyzed using an Inductively Coupled Plasma-Optical Emission Spectrometer (ICP-OES). The data analysis presented a calculation of the effectiveness of activated carbon in adsorbing metal ions contained in artificial waste. The determination of the percentage of metal ion removal can be calculated using equation (1)

$$\% \, Removal = \frac{C_o - C_e}{C_o} \times 100 \qquad (1)$$

Determination of adsorption kinetics

The experiment was carried out in an adsorption column by observing changes in the concentration of each ion in the filtrate every 20 minutes for 2 hours. To obtain data on the concentration of adsorbed ions at each time which is then plotted following the adsorption kinetic equation model.

The kinetics in the column was observed based on the breakthrough curve obtained from the experiment. The shape of the breakthrough curve in the column is affected by the velocity of the fluid flow and the concentration of the adsorbate in the influent. Many mathematical models have been developed to describe ion adsorption on adsorbents in column systems. This work used data from the transient experiment to evaluate the adsorption kinetics. Two kinetics models were proposed, namely the Thomas and the Yoon-Nelson models (de Franco et al., 2017).

The Thomas model equation has the following form:

$$\frac{C_t}{C_o} = \frac{1}{1+exp\left(\frac{k_{Th}}{Q}(q_o m - C_o t)\right)} \qquad (2)$$

where, k_{Th} = Thomas rate constant (mL/min mg), q_o = maximum adsorption capacity (mg/g), t = sampling time (min), C_o = ion concentration in the influent solution (mg/L), C_t = ion concentration in the effluent solution (mg/L), m = adsorbent mass (mg), Q = flow rate (mL/min). The constants in Eq. 2 can be evaluated by linearizing the equation as follow:

$$\ln\left[\left(\frac{C_o}{C_t}\right)-1\right]=\frac{k_{Th}q_o m}{Q}-k_{Th}C_o\,t \tag{3}$$

Kinetic constant (k_{Th}) dan adsorption capacity (q_0) can be determined from the plot $\ln(C_0/C_t-1)$ vs time (t) at a certain flow rate variation.

The Yoon-Nelson model is a simple theoretical assumption that does not concentrate on the properties of the adsorbate, the type of adsorbent, and the features of the adsorption bed. This model states that the decrease in adsorption rate is directly proportional to the adsorption of the adsorbate and the breakthrough in the adsorbent (Yagub et al., 2015). Evaluation of adsorption parameters includes adsorption rate constant and adsorption breakthrough time. The Yoon-Nelson model equation can be written as follows:

$$\frac{C_t}{C_0}=\frac{exp[k_{YN}(\tau-t)]}{1+exp[k_{YN}(\tau-t)]} \tag{4}$$

where, k_{YN} = Yoon-Nelson rate constant (min^{-1}), t = sampling time (min), τ = time required for adsorption breakthrough 50% (min).
The constants in Eq. 4 can be evaluated by linearizing the equation as follow:

$$\ln\left(\frac{C_t}{C_0-C_t}\right)=k_{YN}t-\tau k_{YN} \tag{5}$$

Plot $\ln[C_t/(C_0-C_t)]$ vs sampling time (t) according to Eq. 5 will produce a straight line with slope k_{YN} and intercept τk_{YN}.

3. Results and Discussion

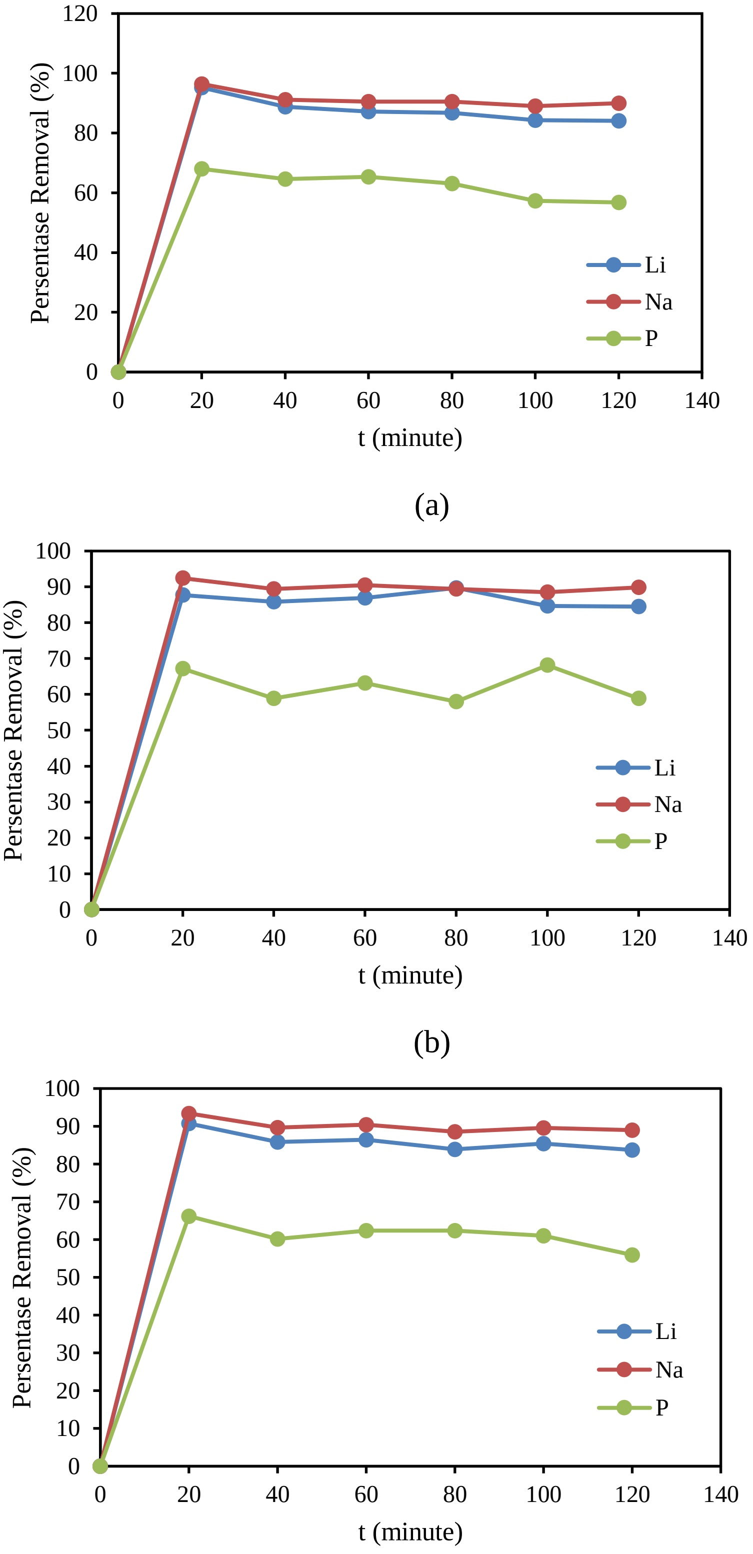
Effect of residence time on the percentage of removal

The flow rate plays an important role in determining the percentage of adsorbate removed in the liquid phase. The increase in flow rate in the downflow direction which is influenced by gravity causes the time required for the solution to exit the column to be shorter, because the time required for the solution to exit is short, the loading time will decrease (Kemala, 2018). Loading time is the time required for the solution to penetrate the pores of the adsorbent to exit the adsorption column (Haryanto & Chang, 2015).

The adsorption process at the initial time with a low flow rate takes place quickly due to the availability of an active site on the surface of the adsorbent which is still able to adsorb ions. At a certain time, the ion exchange process decreases because the active sites for ion exchange run out (exhausted) (Calero et al., 2009). In this study, variations in flow rates were carried out which could affect the percentage of lithium, sodium, and phosphate ion removal from LFP battery artificial recycle waste using activated carbon adsorbents in an adsorption column system. The residence time in adsorption at each flow rate was different, the residence times at flow rates of 10, 15, and 20 mL/minute were 10.07 minutes, 6.71 minutes, and 5.03 minutes, respectively. The following is a graph of the decrease in the percentage of removal which can be seen in Figure 1.

Figure 1

The percentage of lithium, sodium, and phosphate ion removal at residence time (a) 10.07 minutes; (b) 6.71 minutes; (c) 5.03 minutes (C_0 Li, Na, P= 20, 900, 2320 ppm; T= 25 °C; pH= 7)



(c)

Figure 1. shows that the longer the processing time, the smaller the percentage of removal. This is influenced by the conditions of the mass transfer zone. When the wastewater passes through the column containing the adsorbent with the same height as the mass transfer zone, the concentration of contaminants in the wastewater will decrease to a minimum. The first stage of adsorption is the stage that is quantitatively dominant and occurs at the beginning of the adsorption process. At this stage, the rate of the adsorption process is fast due to the availability of active adsorbent sites which are still many so the solution will be adsorbed to the adsorbent higher with increasing contact time (Fajrianti et al., 2016; Patel, 2020).

The highest removal percentages for lithium, sodium, and phosphate ions were 95.22%, 96.42%, and 68.02% which were obtained at the lowest flow rate of 10 mL/minute or at a residence time of 10.07 minutes. This shows that the short contact time between the adsorbent and the adsorbate due to an increase in flow rate will result in a decrease in the percentage of removal in wastewater. In addition, at a faster flow rate, the movement of the adsorption zone along the column occurs faster and results in a decrease in the adsorption time of the adsorbate in liquid waste by activated carbon (Mizwar, 2013). Therefore, in this study, the best flow rate for the column adsorption process was 10 mL/minute.

Column Adsorption Kinetics

The adsorption kinetics of the column using activated carbon with variations in the flow rate of the adsorbate aims to determine the adsorption rate constant and adsorption capacity. These results will be observed with variations in flow rates of 10 mL/min, 15 mL/min, and 20 mL/min.

a. Thomas Model

The Thomas model is one of the most commonly used column performance theory models. The kinetic constant (K_{Th}) and column adsorption capacity (q_0) of the Thomas model in the ion adsorption experiment was obtained by graphing the relationship between t and ln (C_0/C_t-1). The adsorption rate constant (K_{Th}) and column adsorption capacity (q_0) follow the Thomas model equation for varying flow rates for lithium, sodium, and phosphate adsorption with activated carbon presented in Table 3.

Table 3
Parameter value data k and q_0 on adsorption of Ion Li, Na, and P

Ion	Q (mL/min)	K_{Th}	q_o	SSE
		(mL/min mg)	(mg/g)	
Li	10	$3.76.10^{-4}$	$8.75.10^{5}$	$1.00.10^{-2}$
	15	$4.44.10^{-4}$	$7.61.10^{5}$	$8.59.10^{-3}$
	20	$5.86.10^{-4}$	$3.76.10^{5}$	$5.50.10^{-3}$
Na	10	$3.63.10^{-4}$	$8.85.10^{5}$	$5.11.10^{-3}$
	15	$4.01.10^{-4}$	$4.89.10^{5}$	$4.71.10^{-3}$
	20	$4.88.10^{-4}$	$1.09.10^{5}$	$3.80.10^{-3}$
P	10	$4.49.10^{-4}$	$3.91.10^{5}$	$8.37.10^{-2}$
	15	$5.21.10^{-4}$	$3.27.10^{5}$	$6.97.10^{-2}$
	20	$5.82.10^{-4}$	$1.91.10^{5}$	$5.47.10^{-2}$

Table 3 shows the highest adsorption rate constant values for lithium, sodium, and phosphate ions at a flow rate of 20 mL/min, namely $5.86.10^{-4}$ mL/min mg, $4.88.10^{-4}$ mL/min mg, and $5.82.10^{-4}$ mL/min mg with an adsorption capacity of $3.76.10^{5}$ mg/g, $1.09.10^{5}$ mg/g and

$1.91.10^{5}$ mg/g. This shows that the higher the flow rate of the battery recycles synthesis waste solution, the greater the ion adsorption rate constant. However, the increase in the flow rate of the adsorbate results in a decrease in the adsorption capacity of activated carbon. This is by (Aksu & Gönen, 2004; Patel, 2020; Prasetya et al., 2013) who state that the higher the rate of adsorbate flowing into the column, the higher the adsorption rate constant. The flow rate also affects the ion adsorption capacity with the high flow rate of the adsorbate where the contact time of the adsorbate-adsorbent is faster, the adsorption capacity is getting smaller.

b. Yoon-Nelson Model

The Yoon-Nelson model is an equation that can be used to determine the optimum time for lithium, sodium, and phosphate ions to be adsorbed. Kinetic constant (K_{YN}) and time required for the breakthrough of 50% adsorbate (τ). The Yoon-Nelson model in the ion adsorption experiment was obtained by graphing the relationship between t and ln $(C_t/(C_0-C_t))$. The adsorption rate constant (K_{YN}) and time required for breakthrough 50 adsorbate (τ) follow the Yoon-Nelson model equation for varying flow rates for lithium, sodium, and phosphate adsorption with activated carbon presented in Table 4.

Table 4

Parameter value data k and $\boldsymbol{\tau}$ *on adsorption of ion Li, Na, and P*

Ion	**Q (mL/min)**	**K_{YN} (1/min)**	**τ (min)**	**SSE**
Li	10	$7.53.10^{-3}$	335	$1.00.10^{-2}$
	15	$8.87.10^{-3}$	289	$8.59.10^{-3}$
	20	$1.17.10^{-2}$	248	$5.50.10^{-3}$
Na	10	$6.18.10^{-3}$	441	$5.11.10^{-3}$
	15	$7.26.10^{-3}$	390	$4.99.10^{-3}$
	20	$9.80.10^{-3}$	321	$3.80.10^{-3}$
P	10	$8.98.10^{-3}$	144	$8.37.10^{-2}$
	15	$1.04.10^{-2}$	129	$6.97.10^{-2}$
	20	$1.16.10^{-2}$	126	$5.47.10^{-2}$

Table 4 shows the highest adsorption rate constant values for lithium, sodium, and phosphate ions at a flow rate of 20 mL/minute, namely $1,17.10^{-2}$ minute^{-1}; $9,80.10^{-3}$ minute^{-1}; and $1,16.10^{-2}$ minute^{-1} with breakthrough times of 248 minute, 321 minute, and 126 minute. This shows that the adsorption rate constant value of the Yoon-Nelson model increases with the increasing flow rate used. This is because a higher flow rate reduces the adsorption process time and speeds up the adsorption equilibrium (Zhang et al., 2011). It can be seen the difference in adsorption capacity between lithium, sodium, and phosphate ions using activated carbon at a flow rate of 10 mL/minute, where sodium ions produce a much longer breakthrough time than lithium ions and phosphate ions, the breakthrough time for sodium ions is 441 minutes. while lithium ions for 335 minutes and phosphate ions for 144 minutes. This shows that activated carbon can adsorb sodium ions longer and more effectively at relatively low flow rates, but results in relatively short purification of lithium and phosphate. So it can be said that the greater the flow rate, the lower the breakthrough time due to the effect of contact time on ion adsorption capacity. The breakthrough time is assumed to be the time in which 50% of the concentration before adsorption can be purified without any other ion impurities (Yahya et al., 2020; Yoon & Nelson, 1984).

Among the studied Thomas and Yoon-Nelson models, the error values (SSE) for the two models are almost the same. Thus, it was concluded that in comparison of SSE values from predictions and experimental data, both the Yoon-Nelson and Thomas models can be used to describe the behavior of the adsorption process. In addition, it was found that more sodium ions were adsorbed, followed by lithium ions, while phosphate ions were less adsorbed. This is due to competitive adsorption for multi-component systems assuming that all adsorption sites are equivalent and there is no interaction between adjacent adsorption sites of the adsorbent. It also assumes that each site on the adsorbent can accommodate one particular adsorbate molecule, but not a different molecule (Al-Mahbashi et al., 2022; Manjunath & Kumar, 2018). Therefore, it is very important to measure the performance of an adsorbent in a multi-component adsorption system.

4. Conclusion

Adsorption experiments at various flow rates were carried out to investigate the removal of lithium, sodium, and phosphate from synthetic solutions to activated carbon. adsorption processes were carried out in fixed-bed columns with flow rates of 10, 15, and 20 mL/min or at residence times of 10.07, 6.71, and 5.03 minutes. Lithium, sodium, and phosphate removal reached the maximum percentage when adsorption was carried out at a flow rate of 10 mL/min with a residence time of 10.07 minutes, namely 95.22%, 96.42%, and 68.02%, respectively. Two kinetic models are applied to study adsorption kinetics, namely Yoon–Nelson and Thomas, both models can be used to describe the behavior of the adsorption process. Among them, the Yoon–Nelson model was used to determine the breakthrough time, while the Thomas model was used to determine the adsorption capacity. The adsorption kinetics study showed that the adsorption performance of the column for lithium, sodium, and phosphate was found to be most effective at a flow rate of 10 mL/min with an adsorption capacity of $8.75.10^5$ mg/g, $8.85.10^5$ mg/g, and $3.91.10^5$ mg/g, respectively. While the breakthrough times for lithium, sodium, and phosphate were 335 minutes, 441 minutes, and 126 minutes, respectively.

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