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Reduction of Iron (II) Ions in Synthetic Acidic Wastewater Containing Ferro Sulphate Using Calcium Carbide Residu

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Abstract. This paper discusses the use of calcium carbide residue to absorb iron (II) ions in acidic water. In this research, the acidic water was made synthetically using $F_eSO_4.7H_2O$ and processed by batch adsorption methodusing calcium carbide residue as an adsorbent. This study aims to determine the characteristics of adsorbent, absorption capacity and removal efficiency. The concentrations of Iron (II) ions was varied between 40 and 100 mg/L, at the pH of solution of 2.5, adsorbent mass of 5 g, and stirring speed of 200 RPM. Contact time was ranged from 5 to 60 min. The experimental results show that at the 60 min contact time, calcium carbide residue was able to increase the pH from 2.5 to 12.8 and decreased the iron concentration by 99.73%. The process of adsorption of iron (II) metal ions with calcium carbide residue was fitted well with Langmuir's isotherms adsorption model.

INTRODUCTION

One of the main problems related to water resources is the decreasing availability of clean water. This problem occurred because of high-level water pollution due to increased human activity in the industrial and mining sectors. Heavy metal from industrial and mining waste that can threaten the health of the population, source of life and ecological system is iron (II). The iron metal tends to accumulate in living organisms, which can trigger various diseases [1,2] and cause various health problems in living organisms [3].

There are a number of technologies that have been applied for neutralizing iron (II) ions, including adsorption, electrochemical technology [4], electrodialysis [5], and other technologies such as ultra-filtration, electrolysis, reverse osmosis and extraction [6]. Adsorption technology is most often used for industrial wastewater treatment because the design is simple, cheap, effective and efficient, and environmentally friendly. It is known that the good and efficient adsorbents in the adsorption process are activated carbon [7,8,9]. But the operating costs are relatively expensive. Hence, cheaper adsorbent materials need to be found [10,11]. Potential material that can be developed as an adsorbent material is solid waste from the welding industry that uses calcium carbide residue. Calcium carbide residue (CCR) has an alkaline pH (ranging from 12 to 13), a large surface area, fine grain, not soluble, easy to obtain, and the price is relatively cheap. Based on these characteristics, calcium carbide residue can be used as an adsorbent in removing iron (II) ions in acidic water.

The main objective of this study was to examine the ability of calcium carbide residue in removing iron (II) ions in acidic waste water. This process was done using batch adsorption process. Therefore, the equilibrium models Langmuir and Freundlich adsorption isotherms and also first-order and second-order pseudo kinetics models are studied in order to describe the adsorption mechanisms.

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MATERIALS AND METHOD

Equipment and Materials

The equipment used in the laboratory experiments included shakers, magnetic stirrer, pH meter, and beaker glass. An Atomic absorption spectrometer (AAS) (Shimadzu, type ASS-3600) was used for iron analysis. The Scanning Electron Microscopy (SEM-EDX) brand Zeiss type EVO MA10 was used to observed the adsorbent surface morphology. The chemicals were used in this study was synthetic acid water made from $FeSO_4.7H_2O$ (Merck, 99.5), calcium carbide residue, and H_2SO_4.

Adsorbent Preparation

CCR from a local welding in Palembang was dried in the sunlight and sieved into 80 mesh of particle size then soaked using distilled water for 24 h. The adsorbent was made into tablet form with a thickness of 2 mm and a diameter of 3 mm and heated using an oven at 150 ^{o}C for 120 minutes. The average weight of CCR adsorbent is 0.5 g, as shown in Fig. 1.

FIGURE 1. Calcium carbide residue adsorbent.

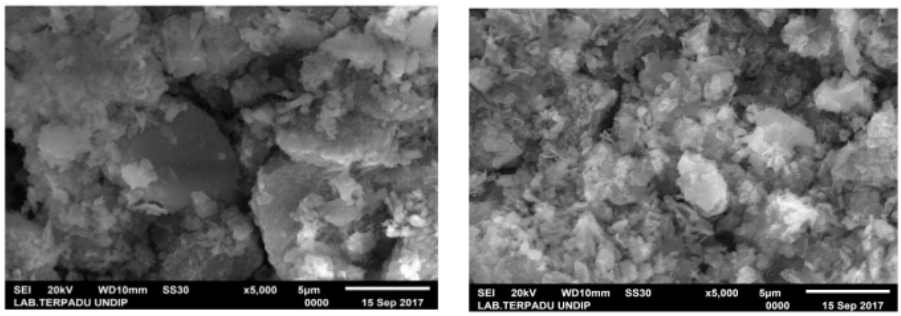
Batch Adsorption Method

In this experiment, the acidic water used was made using FeSO4. 7H2O. The process of adsorption was done in batch systems using a shaker glass containing 100 ml of synthetic acidic water with pH of 2.5 similar with the original acid mine drainage. The initial concentration of iron (II) ions were 40, 60, 80, 100 mg/L. The weight of calcium carbide residue adsorbent was 5 g, with a range of observations was 5 to 60 minutes, then stirred using a shaker with a speed of 200 RPM. The treated of synthetic acid water was obtained by filtration using filter paper in order to separate the filtrate from adsorbent. Finally, the pH values of the filtrate were measured using a pH meter and concentration of iron (II) ions was determined using AAS Shimadzu (ASS-3600)

RESULTS AND DISCUSSION

Characterization of Calcium Carbide Residue as Adsorbent

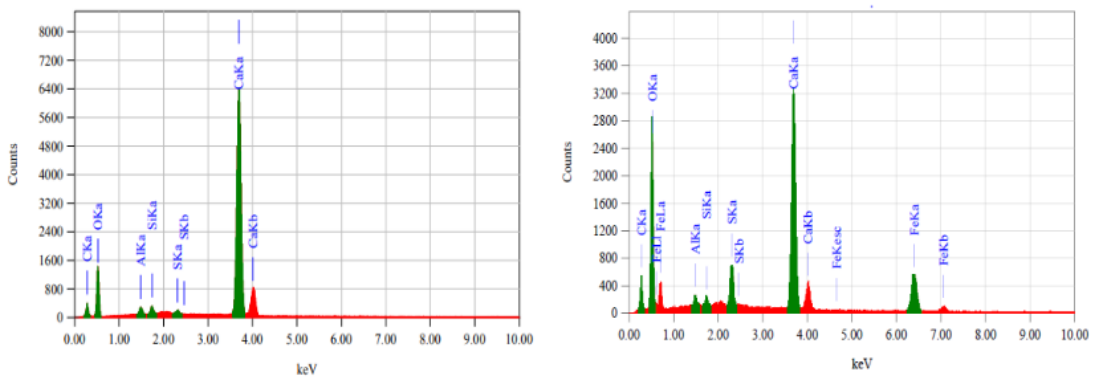
The adsorbents were observed its morphology using Scanning Electron Microscope with 5000 times magnification. The morphology of calcium carbide residue is shown in Figure 2.



(a) (b)

FIGURE 2. Morphology of the adsorbent particle surface: (a) Before adsorption and (b) After adsorption

Fig 3 shows the morphology of calcium carbide residue can be known after the adsorption process of iron (II) ions appears small particles of iron oxide. To detect the presence of iron (II) ions which appear on the surface of calcium carbide residue, the EDX analysis is performed. The results of the analysis using SEM-EDX on calcium carbide residue before and after use for the reduction of iron (II) ion concentrations are presented in Figure 3 and Table 1.



(a) (b)

FIGURE 3. EDX image of CCR: a) Before and b) After the adsorption process

TABLE 1. Composition of calcium carbide residue adsorbents before and after adsorption

No.	Component	Composition (% Mass)	
		Before Adsorption	After Adsorption
1	Carbon, C	13.84	27.53
2	Alumina, Al_2O_3	2.38	1.78
3	Silicon dioxide, SiO_2	2.61	1.62
4	Sulfite, SO_3	1.65	9.83
5	Calcium Oxide, CaO	79.53	38.58
6	Iron (II) Oxide, FeO	-	20.66
7	Total	100	100

From Table 1, it can be seen that after the adsorption process, iron (II) oxide (FeO) concentration increases to 20.66%. This indicates that the calcium carbide residue was able reduced iron (II) ions in the samples. The formation of iron oxide is influenced by several factors including: pH and concentration of iron (II) ions in solution. Iron oxide

was formed from the solution of iron (II) ions through the formation of the blackish green color of Fe $(OH)_2$ which occurs in alkaline conditions. The iron (II) hydroxide precipitates in an alkaline condition through the reactions as shown in Eqs. (1) and (2) [9, 12].

$$F_e^{2+} + 2\,OH \;\rightarrow\; F_e\,(OH)_{2\downarrow} \tag{1}$$

$$F_e\,(OH)_{2\downarrow} \;\rightarrow\; F_e O + H_2\,O \tag{2}$$

Table 1 shows that the amount of calcium oxide (CaO) decreased because it reacts with water to form calcium hydroxide. Calcium hydroxide has a high pH value (alkaline) that increases the pH of the solution, through the reaction of Eq. (3).

$$CaO\ (s) + H_2O\ (l) \;\rightarrow\; Ca(OH)_2\ (aq) \tag{3}$$

The percentage of calcium oxide (CaO) in calcium carbide residue as adsorbent decreased from 79.53% to 38.58%. The neutralization reaction between sulfuric acid (H_2SO_4) and calcium hydroxide $Ca(OH)_2$, is shown in the reaction as shown in Eqs. (4) and (5).

$$H_2SO_4 + Ca(OH)_2 \rightarrow CaSO_4 + 2\,H_2O \tag{4}$$

$$CaSO_4 + 2\,H_2O \rightarrow SO_3 + Ca(OH)_2 \tag{5}$$

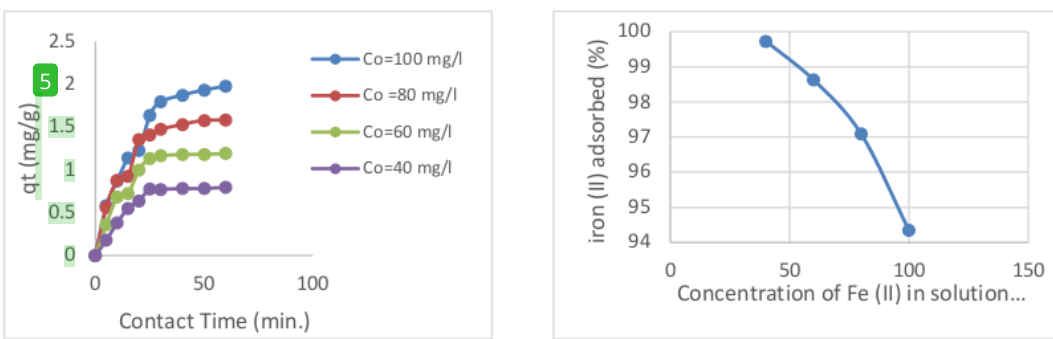
Effect of Initial Concentration and Contact Time on Iron (II) Reduction

The amount of iron (II) reduction per adsorbent mass at a certain time (q_t) was calculated based on Eq. (6) [9].

$$q_t = \frac{(C_0 - C_t) * V}{m} \tag{6}$$

Where, q_t is the adsorbent absorption capacity at time t; C_o is the initial concentration of metal in solution (mg/L); C_t is the concentration of metal in solution after time t (mg/L); V is the volume of the solution (L) and m is the mass of the adsorbent in the solution (g).

The amount of iron (II) adsorbed per mass of calcium carbide residue at equilibrium (q_e) of iron (II) content during the adsorption process for different initial concentrations shown in Fig. 4.



(a) (b)

FIGURE 4. (a) Effect of contact time of the removal concentration of iron (II) (b). The effect of initial iron (II) on adsorption percentage (%)

From Fig.4, it is known that the amount of solute which is absorbed per mass of adsorbent of calcium carbide residue (q_t) increases with increasing of contact time and the initial concentration of iron (II) ions in solution. Fig. 4. also suggested that the adsorbent can absorb iron (II) to a greater concentration than that used in this study. The ability of calcium carbide residue to absorb iron (II) ions has increased up to 60 minutes of contact time.

The percentage of adsorption (%) of iron (II) ions from the solution was calculated using Eq. (7) [13].

$$\text{Percent adsorption } (\%) = \frac{(C_o - C_t)}{C_o} \, x \, 100 \, \% \tag{7}$$

Where, C_0 is the initial concentration of iron (II) ions in solution (mg/L), C_t is the concentration of equilibrium iron (II) ion in solution (mg/L) after a certain time.

Effect of Initial Concentration of Iron (II) Ions in Solution on Adsorption of Iron (II) Ions

The effect of initial concentration of iron (II) ions in solution on percent adsorption of iron (II) ions using adsorbent of calcium carbide residue is presented in Fig. 5. From Figure 5, it is showed that percent adsorption of iron (II) ions in solution decreases with increasing initial concentration of iron (II) ions in solution. The initial concentration of iron (II) ions varies from 40 mg/L to 100 mg/L. The adsorption percentage decreased from 99.73% to 94.35%, due to the increase in the initial concentration of iron (II) ions in the solution with a contact time of 60 minutes. This was expected to provide information about the number of reactive sites in calcium carbide residue pores.

Adsorption Isotherms

There are two equations used in calculating adsorption isotherms, namely Langmuir and Freundlich equations. The Langmuir equation can be seen in Eqs. (8) and (9) [8, 14, 15].

$$q_e = \frac{Q_o K_L C_e}{1 + K_L C_e} \tag{8}$$

$$\frac{1}{q_e} = \frac{1}{K_L Q_o C_e} + \frac{1}{Q_O} \tag{9}$$

The constant values Q_o And K_L are determined by plotting a graph of $\left(\frac{1}{qe}\right)$ vs $\left(\frac{1}{Ce}\right)$

where, q_e: the number of particles adsorbed at (mg/g), Ce: equilibrium concentration of the substance in solution (mg/L), Q_o: maximum monolayer adsorption capacity (mg /g), K_L: Langmuir constant isotherm (L/mg).

According to the Freundlich isotherm adsorption model, the amount of adsorbent mass absorbed per mass of adsorbent can be expressed as a function of solute concentration, Ce according to Eqs. (10) and (11) [16].

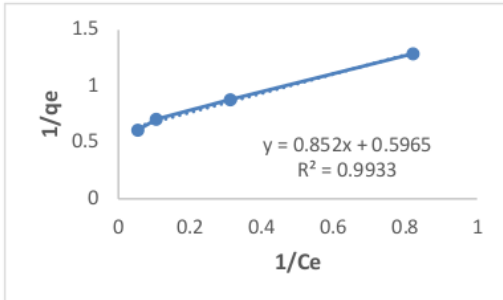
$$q_e = K_f . C_e^{\frac{1}{n}} \tag{10}$$

$$ln \, q_e = lnK_f + \frac{1}{n}(lnC_e) \tag{11}$$

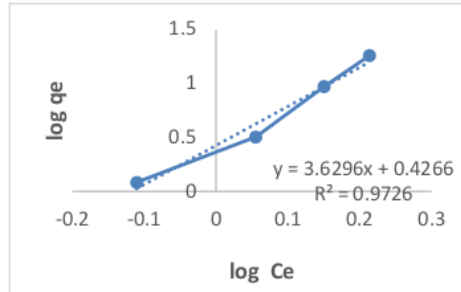
Where, K_f: Freundlich constant adsorption capacity, q_e: the amount of solute adsorbed at equilibrium (mg/g), Ce: equilibrium concentration the solute in solution (mg/L), n: is the intensity of Freundlich constant adsorption.

Figure 5(a) shows the plot of 1/qe versus 1/Ce (Langmuir isotherm adsorption method) from the experimental data at 25 minutes of contact time. From Fig., it can be seen that the plot of 1/qe vs 1/Ce produce a straight line with the equation, y = 0.852x + 0.5965 and the linear regression value R^2 is 0.9933.

Figure 5(b) shows the log qe plot against log Ce (Freundlich isotherm adsorption method) from the experimental data at 25 minutes contact time. From Figure 5(b), it can be seen that the log qe plot against log Ce obtained a linear line with the equation, y = 3.6296x + 0.4266 and the R^2 price is 0.9726 from the value 1/n (heterogeneity factor) greater than 1, namely 3, 6296 and K_F value of 2.6705. The full parameters are presented in Table 2.



(a)



(b)

FIGURE 5. (a) Langmuir Isotherm (b) Freundlich Isotherm

TABLE 2. Parameters of Fe adsorption from linear distribution of the adsorption model of Langmuir and Freundlich isotherms

Time	Langmuir					Freundlich			
0	Linear Equation	q_o	R^2	b	K_L	Linear Equation	R^2	K_F	1/n
5	Y= 222.79 X–2.019	-0495	0.981	222.79	9.06X10-3	Y=0.5901X+1.9171	0.8618	82.623	0.5901
10	Y= 46.311X +0.063	15.87	0.7447	46.311	1.36X10-3	Y=0.9067X+1.6684	0.6759	4..601	0.9067
15	Y=15.541X+0.6083	1.64	0.9486	15.54	3.9X102	Y=1.6902X+1.5675	0.9716	36.940	1.6902
20	Y=6.4644X+0.4885	2.047	0.5348	6.464	7.5X1O2	Y=1.2444X+1.1338	0.3506	13.608	1.2444
25	Y=0.852X+0.5965	1.676	0.9933	0.852	0.7019	Y=3.6296X+0.4266	0.9726	2.6705	3.6296
30	Y=0.884X+0.4807	2.08	0.8723	0.884	0.544	Y=2.6151X+0.2851	0.8665	1.9279	2.6151
40	Y=0.596X+0.4451	2.247	0.7725	0.596	0.6862	Y=2.22829X+0.077	0.829	1.1945	2.2829
50	Y=0.6321X+0.2660	3.843	0.5568	0.632	0.4116	Y= 1.2341X-0.0331	0.5862	0.9266	1.2341
60	Y=0.0794X+0.5445	1.8365	0.9193	0.079	6.8587	Y=2.7005X-0.6313	0.956	0.2336	2.7005

From Table 2, was known that the maximum adsorption capacity, according to the Langmuir model at 15 minutes contact time is 1.640 mg / g adsorbent with an R^2 = 0.9486 and at 25 minutes contact time only 1.676 mg / g with an R^2 of 0.9933 and at 60 minutes contact time 1.837 mg / g and with an R^2 = 0.9193. Whereas for Freundlich isotherm models R^2 for contact times 0f 15, 25 and 60 minutes, the values of R^2 0.9716, 0.9726, and 0.9560 respectively.

The application of the isotherm adsorption model shows a linear relationship between the amount of adsorbed substance per gram of adsorbent and the Langmuir equation parameter and the Freundlich equation. So that the two isotherm adsorption models can be applied to the adsorption process of iron (II) ions using calcium carbide residue adsorbent. The Langmuir isotherm adsorption model at 25 minutes contact time obtained by the equation $\boldsymbol{q_e} = \frac{1{,}1764C_e}{0{,}719C_e+1}$, while the Freundlich isotherm adsorption model obtained by the equation $\boldsymbol{q_e} = 2{,}6705\boldsymbol{C_e^{3{,}6296}}$,. The calculation indicates that the isotherm adsorption pattern in adsorbing iron (II) ions using adsorbent of calcium carbide residue follows the pattern of Langmuir and Freundlich isotherms. It because the active side of the surface of calcium carbide residue was heterogeneous and the adsorption area was occurred on the surface of adsorbent on a mono layer.

Adsorption kinetics

In order to determine the mechanism of iron (II) ions removal process using calcium carbide residue adsorbent, there are two kinetics models that are often used, namely the kinetic model of the pseudo-first order and pseudo-second order developed by Lagergren. Both models are generally used as kinetic models to determine the reaction of the adsorption system [17, 18,19]

Pseudo-first order kinetics model according to Lagergren, generally stated as Eq. (12).

$$\frac{dq_1}{dt} = k_1(q_e - q_t) \tag{12}$$

Where, q_e (mg/g): the amount of adsorption of Fe (II) ions per gram of adsorbent at equilibrium, q_t (mg / g): adsorption capacity at time t (min), k_1 (min-1): constant rate for kinetics at the pseudo-order level.
In the linear form, the pseudo first-order kinetics model can be expressed in Eq. (13).

$$\ln(q_e - q_t) = \ln q_e - k_1\, t \tag{13}$$

Then a graph was made between ln (qe-qt) vs t, therefore the value of qe (calculated) and k_1 were obtained. Where qe was adsorbate (solute) capacity absorbed at equilibrium (mg/g), qt was the amount of adsorbate (solute) absorbed at (t) minute, k_1 was the first order adsorption constant (minute-1).

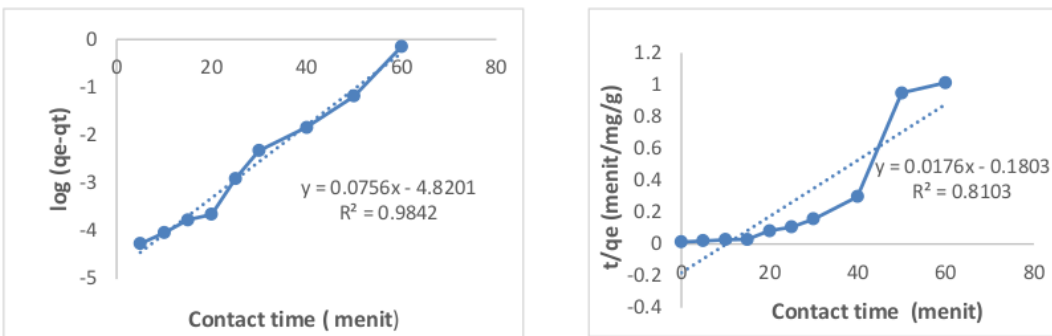
The pseudo second-order kinetics model was used to test the presence of chemical reactions on the active side of the calcium carbide residue adsorbent which controls the rate of removal of iron (II) ions in synthetic acid waste water solutions. The pseudo second-order kinetics model was stated in Eq. (14).

$$\frac{dt}{dt} = k_2\,(q_e - q_t)^2 \tag{14}$$

The linear form of the pseudo second-order kinetics model was expressed in Eq. (15).

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e}\, t \tag{15}$$

The adsorption kinetics model can be used to determine the variables involved in adsorption and the mechanism that occurs. Figures 6(a) and 6(b) show kinetic plots for adsorption of iron (II) ions on calcium carbide residue using a pseudo-first-order, pseudo second-order model. Parameter values and correlation coefficients obtained are in Table 3.



(a) (b)

Figure 6. (a) Pseudo kinetic first order. (b) Pseudo kinetic second order

TABLE 3. Linear kinetic model and correlation values (R^2)

Initial concentration Co (mg/L)	First order kinetic model			Second order kinetic model		
	Linear Equation	R^2	k_1	Linear Equation	R^2	k_2
100	Y=-0.0756X+4.8201	0.9842	0.0756	Y = 0.0111x - 0.1271	0.6879	0.0111
80	Y=-0.0787X+4.3333	0.9773	0.0787	Y = 0.0176x - 0.1803	0.8103	0.0176
60	Y=-0.0881X+3.9781	0.9311	0.0881	Y = 0.0341x - 0.3215	0.87	0.0341
40	Y=-0.0951X+3.6968	0.9186	0.0951	Y = 0.1038x - 1.3112	0.5333	0.1038

Table 4, shows that the Fe (II) ion adsorption model takes place according to the first-order pseudo kinetics model with R^2 = 0.9842, at an initial concentration of 100 mg/L, 0.9773 at a concentration of 80 mg/L, 0.9311 at a concentration of 60 mg/L and 0.9186 at a concentration of 40 mg/, respectively. The adsorption constant (k) increasing

when the initial concentration is small, thus the adsorption kinetics model in this study is the pseudo-order 1 kinetics model means that the initial concentration of Fe (II) ion solution affects the rate of reaction.

CONCLUSIONS

Calcium carbide residue is suitable to be used as an absorbent to reduce iron (II) ion concentration. There is a decrease of 99.73% for iron (II) with the initial concentration of 40 mg/L and 98.84% for initial concentrations of 100 mg/L. The Calcium carbide residue also can increase the pH of acidic water from 2.5 to 11.8.

The process of adsorption of Fe (II) ions by calcium carbide residue can follow the adsorption model of Langmuir and Freundlich Isotherms. The best adsorption process occurs at 25 minutes contact time with the highest R^2 value of 0.9933 for the equation of the Langmuir isotherm and for 0.9726 for the Freundlich Isotherm equation. The results of calculations using the equation of the adsorption model of Langmuir isotherm are obtained: $q_e = \frac{1,1764C_e}{0,719C_e+1}$. The kinetic adsorption of Fe (II) ions in calcium carbide residue are in accordance with first order pseudo adsorption kinetics with adsorption rate constants $k_1 = 0.0756$ and $R^2 = 0.9842$ for initial concentrations of 100 mg/L, $k_1 = 0.0787$, $R^2 = 0.9773$ for initial concentrations of 80 mg/L, $k_1 = 0.0881$ and $R^2 = 0.9311$ for initial concentrations of 60 mg/L, and $k_1 = 0.0951$ and $R^2 = 0.9186$ for initial concentrations of 40 mg/L

q_e prices continue to increase in line with the increase in pH values from pH values of 2.5 to pH 12.8, the tendency of increasing qe prices is still visible. The optimum interaction time occurred at 25 minutes, which showed the number of Fe (II) ions adsorbed per gram of calcium carbide residue with q_e value of 0.976 mg / g for initial concentrations of 40 mg/L and 1.136 mg/L for initial concentrations of 60 mg/L. Whereas for initial concentrations of 80 mg/L and 100 mg/L, q_e prices were 1.580 mg/L and 1.977 mg/L, respectively.

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