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Initial pH Influence on Organic Pollutants, Total Suspended Solids, and Nickel Removal from Citarum River Water Using Electrocoagulation

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ABSTRACT

The water of the Citarum river in West Java is used as a water source for three hydropower plants, and some industries operate along the river area. Besides industries, the Citarum river also accommodates domestic and livestock waste. These lead to very high-level pollution in the Citarum river, especially organic and heavy metal waste. One method that can reduce simultaneously organic and heavy metals in water is electrocoagulation. The purpose of the research is to determine the influence of initial pH or pHi on the removal of total suspended solids, total organic pollutants, and nickel ion from Citarum water using aluminum as a cathode and an iron metal as an anode in the electrocoagulation method. During the electrocoagulation process, current density, electrode distance, stirring speed, DC supply voltage, and temperature were kept constant at 118 Am^{-2}, 2 cm, 100 rpm, 18.3 V, and 26-27^{0}C respectively. The nickel-metal ion concentration is determined using Atomic Absorbance Spectrometry (AAS), total suspended solids as TDS by pH/EC/TDS/^{0}C meter, and organic pollutants as TOC by permanganometic titration. It is found that Ni removal is almost 100% after 10 minutes electrocoagulation process at pHi 3, 5 minutes at pHi 5 and 9, and 60 minutes at pHi 7.5. The Maximum total suspended solids, and total organic pollutants removal was obtained after 60 minutes electrocoagulation process. The finest total solid suspended and total organic pollutants removal achieved at pHi 3 are 40 and 84%, respectively. The kinetics of total organic pollutants and total suspended solids removal is suggested obeying the second-order and Lagergren's 2nd order equation, respectively. So, It suggests that the total suspended solids are removed from Citarum river water through an adsorption mechanism.

Keywords: *Citarum Water, Electrocoagulation, Aluminum Cathode, Iron Anode, Water Quality.*

1. INTRODUCTION

The Citarum River has important economic potential for West Jawa, Indonesia. It is used as a water resource for three hydroelectric power plants (Cirata, Saguling, and Jatiluhur), and about 3,236 industries such as textile, leather, food, and beverage, and pharmacies operate along the river area. 20% of them do not have wastewater treatment plants. Consequently, these industries dispose of their wastewater to the Citarum river [1]. Besides industries, domestic, livestock, and agriculture release waste to the Citarum river. Monitoring Citarum water quality shows that some parameters of heavy metals, such as Cu, Ni, Fe, and organics pollutants, do not meet water quality standards [2][3].

One method that can reduce heavy metals and total organic pollutants from water to meet industrial water is electrocoagulation or EC [4]. This method has advantages compared to other methods, such as not requiring chemicals as in the chemical coagulation method. In addition, this method can separate pollutants in various sizes (ionic, molecular, macromolecule, and microparticles) while ion exchange and reverse osmosis is only for ionic sizes and filtration (nano, ultra, and microfiltration) for particles with molecular, macromolecules, and microparticles size range [5]. The drawback of this method is the use of electrical energy in the water purification process. This deficiency can be overcome by developing the use of renewable energy, namely solar energy through photoelectric cells [6].

Several electrodes used in EC have been developed, including Al, Ni, Fe, stainless steel, and carbon. An iron as anode and aluminum as cathode exhibited Ni and Cu metal removal up to 96-99% [7]. Besides, an iron and aluminum price is low and easy to obtain [8]. Some parameters that are affecting EC efficiency are current density, electrodes (material and arrangement, distance between electrode), initial pH (pHi) and conductivity, temperature, reaction time, and steering speed. Because Citarum river water's pH varies and Ni is above the standard industrial water concentration, it needs data on EC efficiency at various pHi and reaction times to remove pollutants from Citarum river using EC. Therefore, the research aims to determine the influence of pHi on total organic pollutants, total suspended solids, and Ni Removal from Citarum water using EC.

2. LITERATURE REVIEW

Electrocoagulation is a process that occurs using electrochemical cells, electrolytic cells. The cell consists of an anode and a cathode, water contains electrolite, and electrical source. Oxidation reaction occurred at an anode that produces electrons and the anode is the positive pole. Reduction reaction occurs at cathode and it is negative pole. Because the oxidation and reduction reactions that occurred in electrolytic cells are not spontaneous, electrical energy is needed from an external circuit using direct current. The chemical equations that describe reactions in Fe as an anode and Al as a cathode are detailed below [9]. At an anode oxidation reaction that can be occurred are

$Fe \rightarrow Fe^{2+} + 2\,e^{-}$ and Fe^{2+} can be oxidized further to form Fe^{3+}

$Fe^{2+} \rightarrow Fe^{3+} + e^{-}$
$2H_2O \rightarrow O_2 + 4H^{+} + 4e^{-}$
At the cathode some reduction reaction can be arised,

$2H_2O + 2\,e^{-} \rightarrow H_2 + 2\,OH^{-}$

$O_2 + 4H^{+} + 4e^{-} \rightarrow 2H_2O$

The total reaction that occurs in the cell is the sum of the reactions at the anode and cathode:

$$Fe \rightarrow Fe^{2+} + 2\,e^{-}$$

$$Fe \rightarrow Fe^{3+} + 3\,e^{-}$$

$$\underline{5H_2O + 5\,e^{-} \rightarrow 2.5\,H_2 + 5\,OH^{-}}$$

$$2\,Fe + 5\,H_2O \rightarrow 2.5\,H_2 + Fe^{3+} + Fe^{2+} + 5\,OH^{-}$$

The three ions, Fe^{2+}, Fe^{3+}, and OH^{-}, will pair up and form nano-sized solids (Floc) that can absorb, adsorb, and co-precipitate pollutants in water (Figure 1).

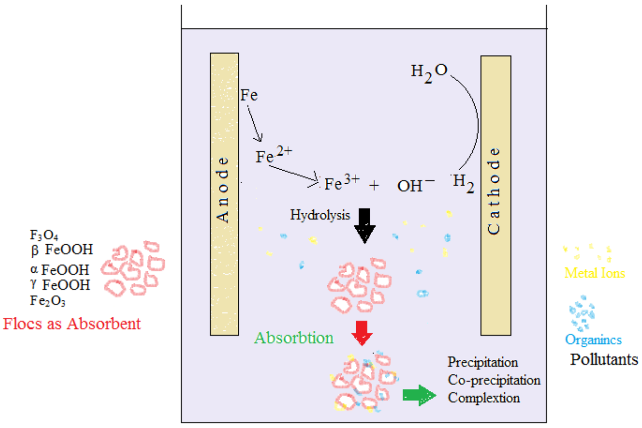


Figure 1 Coagulation process in electrocoagulation cells: electrochemical reactions, floc formation, and pollutants removal

Floc is a material formed from nano-sized Fe(oxy)hydroxides so that it has a high absorption ability. Some types of floc that can be formed are Green rust (GR), magnetite (Fe_3O_4), hematite (α-Fe_3O_4), lepidocrocite (γ-FeOOH), goethite (α-FeOOH), and layered double hydroxides (LDHs). There are 3 zones on the EC in floc formation: acidic, buffer, and alkaline zones. In the acidic zone, anions are formed. In the alkaline zone, cations are formed, and in the buffer zone, hydroxide flocs are formed which are insoluble in water. The H_2 produced by the cathode facilitates floc mass transfer and pollutants deposition. The OH^{-} generated at the cathode affects the tendency to form Fe(II)/Fe(III) hydroxide complexes in solution. Dissolved Fe(II) is easily oxidized to Fe(III) which can also be formed directly at the anode. The pH of the solution is one of the parameters that determine the change of Fe(II) to Fe(III) and the type of floc. The optimum formation of magnetite (Fe_3O_4) and GR-CO_3 occurred at pH 8-9, while lepidocrocite was at pH 7. Thermodynamically, the formation of Fe(II)/Fe(III) species occurred at pH 6-7.

2.1. Mechanism of floc formation

In an anaerobic solution (poor O_2), GR will be formed. When there is O_2 (dissolved O_2 or DO from the air produced by cathode) in the solution, GR will change into γ-FeOOH. And this form will turn into a mixture of α-FeOOH and γ-FeOOH. If there is no DO in the solution, at pH 7 GR will not change and end up in Fe_3O_4. GR-CO_3 can be formed in the presence of anions CO_3^{2-}, and has a hexagonal crystal structure. Unlike other Fe species, GR has an internal surface which results in greater surface area and reactivity. Therefore, polar molecules can be absorbed on the internal and adsorbed on the surface of the GR. Metal ions, such as Cu(II), Ni(II), and Zn(II), replace Fe ions during GR formation (co-precipitation) [10].

2.2 *Pollutants Removal Kinetics*

To describe the kinetics of pollutants removal, kinetics data of pollutants can be evaluated using zero, pseudo-first, second, and Lagergran's first and second-order for adsorption mechanism. The rate law of pollutants concentration reduction in batch EC is presented by Equation (1) [11], [12].

$$\frac{dC}{dt} = -kC^n \qquad (1)$$

k is a constant rate, C is concentration, and n is reaction order. The integrated form of Equation (1) is Equation (2) for zero order.

$$\frac{dC}{dt} = -k \rightarrow C_t = C_0 + kt \qquad (2)$$

Where C_t and C_0 are concentration at time (t) and initial concentration, respectively. After integrating, the first-order reaction (n=1) becomes Equation (3).

$$\frac{dC}{dt} = -kC \rightarrow \int_{C_0}^{C_t} \frac{dC}{C} = -kdt$$

$$\rightarrow \mathrm{Ln}\,\frac{Ct}{Co} = -kt \qquad (3)$$

Solution of integrating equation for the second-order (n=2) is Equation (4).

$$\frac{dC}{dt} = -kC^2 \rightarrow \int_{Co}^{Ct} \frac{dC}{C^2} = -kdt$$

$$\frac{1}{C_t} - \frac{1}{C_0} = kt \qquad (4)$$

If C_t, Ln $\frac{C_t}{C_0}$, or $\frac{1}{C_t}$ are plotted against t, they will get a straight line equation.

Supposing that the pollutants removal to be through an adsorption mechanism, the Lagergren's first- and second-order reaction rate equations can be used. Lagergren's first-order equation is Equation (5).

$$-\frac{dq}{dt} = k(q_e - q) \qquad (5)$$

Where q_e and q_t (mg/g) are the amounts of adsorbed at equilibrium and at time t, respectively; k_1 is the equilibrium rate constant in the first-order model (min^{-1}). The integrated form of Equation (5) is Equation (6).

$$\mathrm{Ln}\,(q_e - q_t) = \mathrm{Ln}\,(q_e) + kt \qquad (6)$$

Lagergren's equation for second-order is Equation (7).

$$-\frac{dq}{dt} = k(q_e - q_t)^2 \qquad (7)$$

Equation (7) can be integrated and will become Equation (8).

$$\frac{t}{q} = \frac{1}{k\,q_e^2} + \frac{1}{q_e}\,t \qquad (8)$$

Where k is rate constant ($min.g.mg^{-1}$). By making a curve of $\frac{t}{q}$ to t, it will get the equation of a straight line. q_e can be determined by inverting the value of the gradient of the line and k is calculated by using the intercept of the line equation.

3. RESEARCH METHOD

The parameters of Citarum river water were determined before conducting EC. The pH and conductivity are 7.5 and 0.2 mS cm^{-2}, respectively.

The electrocoagulation cell consists of a power supply rectifier, electrodes, stirring, and 6 L plastics reactor. Fe as anode and Al cathode size was 45 mm × 75mm × 3mm (width x height x thickness). The arrangement of 3 pairs of Al and Fe were mono-polar parallel (MP-P) configuration [13], and the distance between the electrodes was fixed at about 2 cm (Figure 2). The current was adjusted using a DC rectifier (GW Instek GPR-1810HD). Electrocoagulation was conducted at room temperature (26-27°C). The pH, total suspended solids as TDS, and conductivity of the river water were determined using a pH/EC/TDS/°C meter (HI9813-5). River water is filtered using filter paper and whatman size 42 to decrease river water pH and adjust to 3 and 5 was used HCl and to increase pH to 9 was used 0.1 N NaOH. The electrocoagulation cell was stirred using a magnetic stirrer and kept constant at 200 rpm. Electrodes were sand and washed by dilute HCl and water before conducting every experiment. Electrocoagulation was performed in 60 minutes and 100 mL of water samples were taken and filtered by whatman 42 at 5, 10, 20, 30, 40, and 60 minutes for analysis. The concentrations of Ni were determined by AAS (Atomic Absorbance Spectrometer, Shimadzu AA 7000) method. Organic pollutants as TOC was determined using permanganometric titration and total suspended solids as TDS was determined by pH/EC/TDS/°C meter. The percentage of Ni, organic pollutants, and total suspended solids removed from the river water is calculated using Equation (9).

$$\%\ \text{removal}\ (t) = (C_0 - C_t)/\ C_0 \times 100\% \qquad (9)$$

The amount of organic pollutants and total suspended solids adsorbed on and absorbed into flok is evaluated using Equation (10).

$$q(t) = (C_0 - C_t) \qquad (10)$$

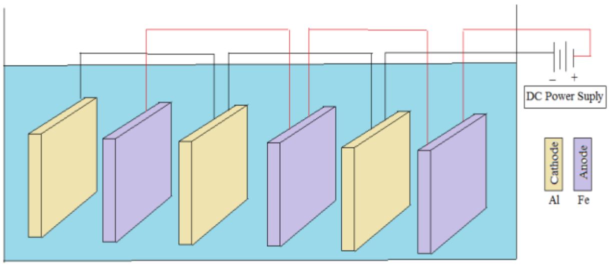


Figure 2 Fe and Al configuration in EC conducted

4. RESULTS AND DISCUSSION

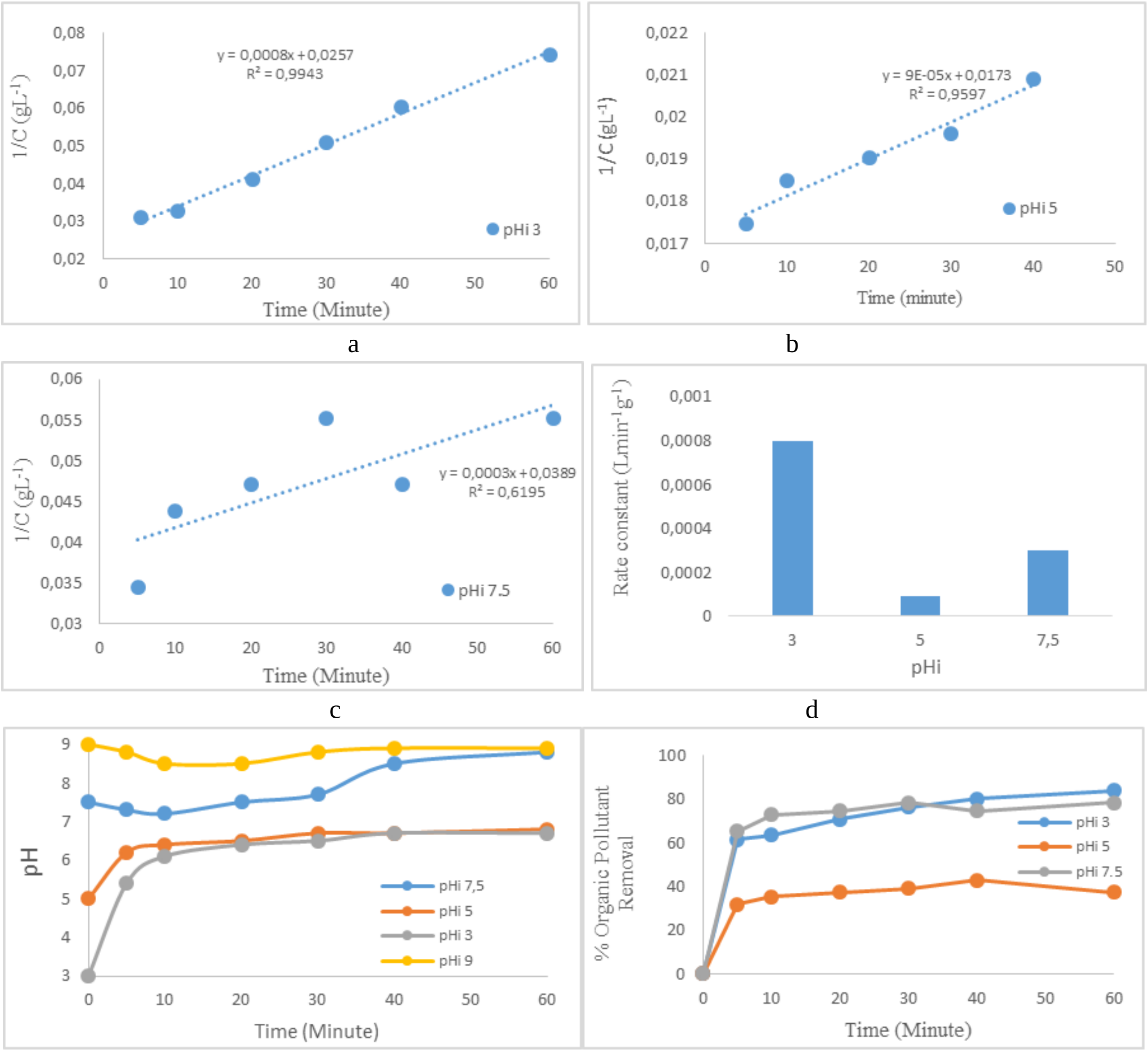
The reaction kinetics of reducing total organic pollutants and total suspended solids in water was carried out using the zero, pseudo-first, second-order reaction rate laws, and Lagergren's second-order rate equations on the kinetic data. From the curves such as Figure 3. a-b-c that plotted $\frac{1}{C_t}$ over t, the correlation value of kinetic data with linear equations or R^2 (the squared-correlation coefficient) was obtained and tabulated in Table 1.The largest R^2 value in the EC process with pHi 3, 5, and 7.5 occurs at the rate law of order 2nd. Therefore, it is assumed that the organic pollutants reduction process goes along with order 2nd. It is appropriate with other research reports [14] [15] [16]. The largest rate constant occurs at pHi 3. Since the rate law does not meet the rate law Lagergren's second order, it is suggested that the organic pollutants reduction mechanism does not obey the adsorption mechanism on the surface of the floc.

Tabel 1. R^2 value of zero**,** pseudo-first**,** 2nd, and Lagergren's 2nd order equation in organic pollutants Removal

pHi	R^2				Rate Constan (g (Lmin)$^{-1}$) 2nd order
	Zeroth-order	Pseudo-first order	2nd order	Lagergren's 2nd order	
3	0.9353	0.9766	0.9943	0.9976	0.0008
5	0.3743	0.3644	0.6195	0.989	9.00E-05
7.5	0.9939	0.6011	0.9597	0.0987	0.0003

At pHi 3, 5, and 7.5, with a solution pH of 5-7, the floc is in the GR form. It has a large internal surface area so that total organic pollutants can be absorbed onto the internal surface. At pHi 3, the pH of the solution was more acidic than pHi 5, while the pH of the solution at pHi 7.5 was more alkaline (Figure 3. e). This causes the interaction of total organic pollutants with the floc to be more stimulated. The interaction is strengthened by acidic and alkaline conditions. It is shown by the rate constant for total organic pollutants reduction at pHi 3 and 7.5 is greater than at pHi 5 (Figure 3. d). This is also supported by the % total organic pollutants removal at pHi 3 and 7.5 greater than at pHi 5.

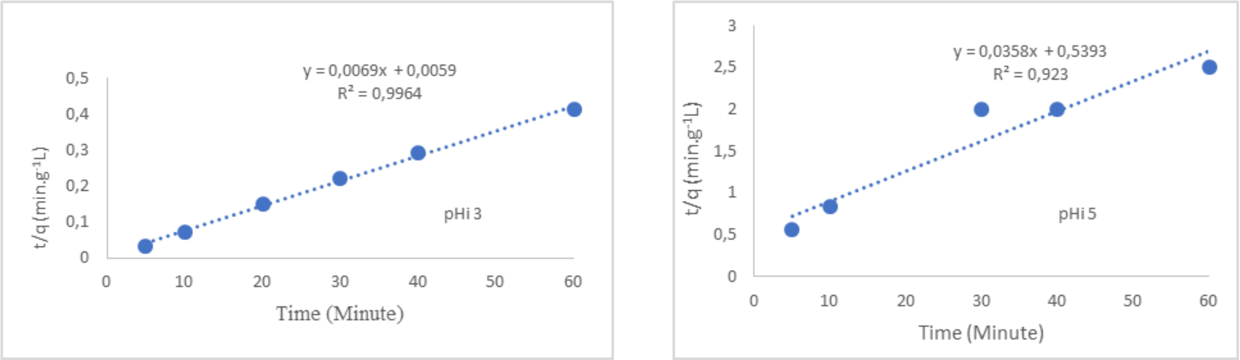
It is not appropriate with adsorption organic compounds such as chlorophenol on γ-FeOOH. In this study, the amount of chlorophenol adsorbed decreases along with the increase of pH [17]. During 60 minutes EC processing, the removal of total organic pollutants at pHi 3, 7.5, and 5 are 84, 78%, and 39%, respectively (Figure 3. f). Compared to the EC study using bilge water, the maximum organic removal reaches 89% [18] and in this study 78% at the natural Citarum water river (pHi 7.5). Another study on real textile water using EC at optimal conditions shows that total organic compound and total suspended solids removal are 42.5 and 64.7%, consecutively.



a b c d e f

Figure 3 a-b-c Removal 2nd order kinetics, d. Rate constant of 2nd order, e. pH solution in various pHi, f. Organic pollutants removal

The kinetics of total suspended solids removal was conducted as same as total organic pollutants. To evaluate kinetic of pollutants removal, the kinetics data was applied using rate equations of zero, pseudo-first, 2nd-order, and the Lagergren's equation of order 2. The linear regression curve was obtained for each equation, an example is a curve that uses Lagergren's 2nd order equation (Figure 4 a-b-c-d).



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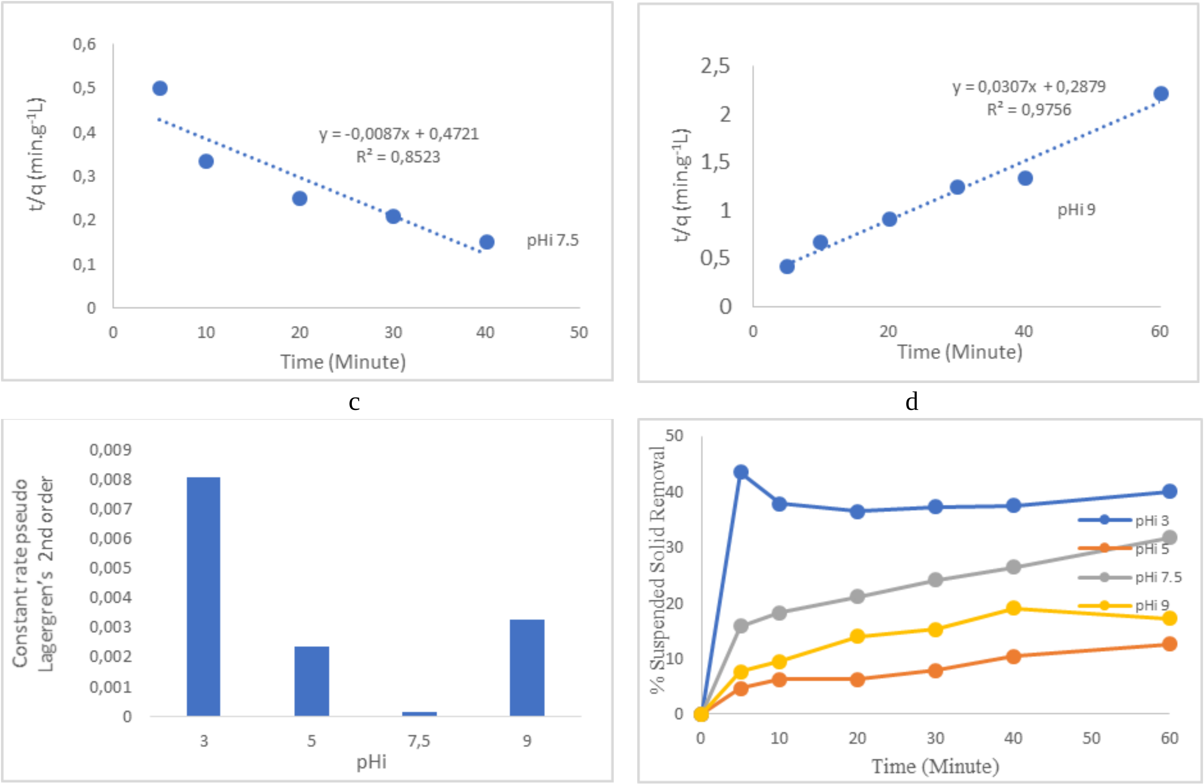

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Figure 4 a-b-c-d Removal kinetics using 2nd Lagergren's equation, e. The rate constant of Lagergren'**s** 2nd order, f. Solid suspended removal

The R^2 values of the straight-line zero, pseudo-first, 2nd order, and the 2nd Lagergren's equation are tabulated in Table 2. The largest R2 values at some pHi obey Lagergren's 2nd order equation. It is confirmed that the kinetic of total suspended solids Removal agree with Lagergren's 2nd order. So that, it is proposed that the mechanism of solids Removal is through an adsorption mechanism.

Tabel 2. R^2 value of zero, pseudo-first**,** 2nd, and Lagergren's 2nd order equation in suspended solids removal

pHi	R^2				Rate Constan (g (Lmin)$^{-1}$) Lagergren's 2nd order	q_e (mg)
	2nd order	Pseudo-first order	Zeroth-order	Lagergren's 2nd order		
3	0.0476	0.4342	0.0385	0.9964	0.00807	144.9
5	0.9599	0.9599	0.8245	0.923	0.00238	27.9
7.5	0.9983	0.9972	0.9939	0.8466	0.00009	114.9
9	0.7508	0.7511	0.8811	0.9756	0.00327	32.6

The adsorption equilibrium constant can be determined from the line intercept and q_e values calculated using the line gradient values (Equation 8). Figure 4. e shows the adsorption rate constant at pHi 3, 5, 7.5, and 9. The lowest total suspended solids adsorption rate constant reached at pHi 7.5. It means that the slowest equilibrium state of adsorption reaches at pHi 7.5. It is also confirmed by the change of % total suspended solids removal patterns over time at pHi 7.5 (Figure 4. F). Even though EC at pHi 7.5 is the slowest to reach an equilibrium state, the q_e value (adsorbed total suspended solid in an equilibrium state) and % total solids suspended removal is greater than at pHi 5 and 9. Not only the highest q_e and % total solid suspended removal but also reach the equilibrium state fastest due to adsorption rate constant (Figure 4. f) occurred at pHi 3. In general, the % of total suspended solid removal is lower than the % of total organic pollutants removal. The largest % of total solid suspended removal (40%) occurred at pHi 3. In this research, suspended solid removal is lower than suspended removal (76.6%) from synthetic water using an aluminum electrode [19].

Figure 5. shows the % removal of Ni from Citarum river water in the EC process at pHi 3, 5, 7.5, and 9. In

the first 5 minutes Ni removal reached 100% at pHi 5 and 9, 80% at pHi 7.5, and <20% at pHi 3. At the same time, the largest % removal of total suspended solids and total organic pollutants among all pHi conducted are at pHi 3. In the first 5 minutes, floc form is GR, which has a wide outer and inner surface. The determination of kinetics shows that the mechanism of reducing total organic pollutants and total suspended solids from river water accord to adsorption on the outer surface and absorption onto the inner of the floc. On the other hand, the mechanism of Ni uptake in floc follows the mechanism of Fe ion replacement (co-precipitation). Due to a large number of total organic pollutants and total suspended solids being adsorbed on and absorbed onto the floc, it will prohibit Ni ions from entering the interior of the floc to replace Fe.

At the beginning of EC, the research shows that % Ni removal is inversely proportional to % of total organic pollutants and total suspended solids removal. During the first 5 minutes at pHi 3 and 7.5, % of total organic pollutants and total suspended solids removal reach 64 and 44%. At the same pHi and time, the % Ni removal does only 15%. This was since, at the same time, organic pollutants covered the inner surface of the floc, and the outer surface was covered by solids suspended so that it was difficult for Ni to access the interior of the floc to replace Fe in the floc. The longer the EC is carried out, the more the number of floc forms, and the pH becomes alkaline. In the alkaline condition, Ni can co-precipitate, either replacing the Fe position or settling as unsoluble Ni hydroxide. Through this removal mechanism, the % of Ni removal can still reach 100%. Under optimal conditions, the EC carried out on metal plating wastewater reached 100% of Ni removal in 20 minutes [7], simulated water reached 100% in 40 minutes [9], artificial metal plating wastewater was almost 100% [20].

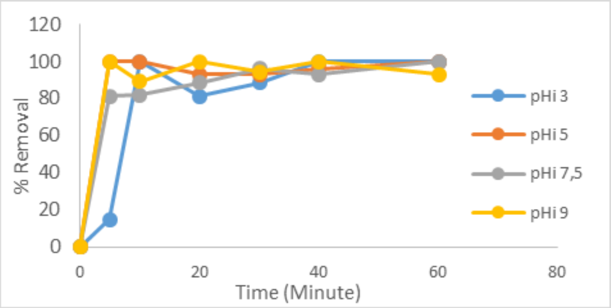

Figure 5 The influence of pHi in Ni removal

5. CONCLUSION

Ni removal from Citarum river water reached 100% at all pHi but at different times. Removal of 100% can occur in a shorter time, 5 minutes at pHi 5 and 9 and 10 minutes at pHi 3. EC conducts at natural pH (pHi 7.5) requires a longer processing time (30-60 minutes). Removal of total organic pollutants can reach 83% at pHi 3 and 78% at pHi 7.5. Removal of total suspended solids is relatively low, maximum 40% at pHi 3. The kinetics of total organic pollutants and total suspended solids removal is suggested to obey the 2nd order and Lagergren's 2nd order, respectively. It is suggested that suspended solids remove from Citarum river water through an adsorption mechanism.

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