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# Adsorption of Direct Yellow Dye from Aqueous Solution by Ni/Al and Zn/Al Layered Double Hydroxides

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**Abstract.** The synthesis of layered double hydroxides has been conducted by coprecipitation method using $M^{2+}/M^{3+}$ metal cations in aqueous medium, where $M^{2+}$: $Ni^{2+}$, $Zn^{2+}$ and $M^{3+}$: $Al^{3+}$. Ni/Al and Zn/Al layered double hydroxides were characterized using FTIR and X-Ray powder analyses. Ni/Al and Zn/Al layered double hydroxide were applied as adsorbent of direct yellow dye in aqueous medium. The results show the data of kinetic parameter was appropriate with pseudo-second-order. Freundlich isotherm model was fitted to adsorption of direct yellow dye by Ni/Al and Zn/Al layered double hydroxide then Langmuir isotherm model. Thermodynamic parameters indicated the adsorption process was endothermic and spontaneous. The results showed that Ni/Al and Zn/Al layered double hydroxides are potential adsorbent for treatment of dye on wastewater.

## INTRODUCTION

Natural water damage is one of the world's issues of concern such as metal ions dyes, pesticides and chemical synthetic materials. This is because the problems can harmful to environmental and human due to nondegradable these substance[1]. Natural water can be polluted caused by synthetic dyes [2]. Synthetic dyes can be produced from industrial process laboratory and home industries such as home textile industry. Home textile industry was found in several locations in Indonesia including South Sumatera. Synthetic dyes used are dyestuffs that have aromatic structures that cause these dyestuffs to be stable and hard to biodegradable [3] therefore, that dyes should be removed from solution. The methods were usually used to dyes removal are adsorption, coagulation, electrocoagulation and photodegradation [4]. One of the most widely used processes is the adsorption process.

The adsorption process was chosen because this process tends to be easier, cheaper and effective [5,6]. The successful process of adsorption is depending on adsorbent. Various adsorbent to removed dyes in wastewater such as activated carbon [7], layered double hydroxides [8], graphene [9] and bentonite[10]. Therefore the researches were searched an effective adsorbent with low cost [7]. The most effectively adsorbent to adsorp dyes is layered double hydroxides [11]. Layered double hydroxides has the advantage of being easily synthesized and can be easily used in the desired process compared to the naturals adsorbent [12].

Hydrotalcite is a class of synthetic anionic clays whose represented by the general formula $[M^{2+}_{(1-x)}M^{3+}_{x}(OH)_2](A^{n-})_{x/n}{\bullet}nH_2O$ [13] with the identities of $M^{2+}$ and $M^{3+}$ are divalent and trivalent metal cation and $A^n$ is interlayer anion as counter balancing the metals cations[14].Layered double hydroxides had more advantages is can be easily anions exchanges, this advantages caused the effectivically layered double hydroxides as adsobent [15]. The anion can be taking up by layered double hydroxides uses three mechanism such as anion exchanges in interlayer, surface adsorption and recontruction layered duble hydroxides calcined by 'memory effect'. Layered double hydroxides as adsorbent has been reported used layered double hydroxides Ni/Fe and Mg/Al to adsorp congo red dye[8,13]. The result was shown layered double hydroxides can be removed congo red in waste water effectivelly. In this research, layered double hydroxides NiAl and ZnAl were characterized using FTIR

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Spechtrophotometer and XRD analysis. The adsorption of dye on layered double hydroxides was studied by the kinetic and thermodynamic parameters by measuring residual concentration and adsorbed using spectrofotometers UV-Visible.

## EXPERIMENTAL SECTION

### Chemicals and Instrumentation

Chemicals was used are analytical grade from Merck and Sigma Aldrich such as nickel nitrate, zinc nitrate, aluminum nitrate, sodium hydroxide, sodium carbonate. Water was supplied from Integrated Research Laboratory, Universitas Sriwijaya using water system Purite® ion exchange pH 7. FTIR spectrophotometer was conducted using Shimadzu FTIR Prestige-21 using KBr disk and scanning was performed at wavenumber 400-4000 $cm^{-1}$. XRD was conducted using Rigaku Miniflex-600 and scanned at 0.1 deg $min^{-1}$. Analysis of direct yellow was used UV-Vis spectrophotometer double beam EMC-61PC.

### Synthesis of Ni/Al Layered Double Hydroxides

Synthesis of Ni/Al layered double hydroxides using co-precipitation method [17]. Nickel nitrate (100mL, 0.3 M) and alumunium nitrate (100 mL, 0.1 M) with the molar ratio 3:1 were stirring for a hour. Then, 100 mL of sodium carbonate solution 0.3 M at 80 $^{o}$C was added slowly. The solution was added with 25 mL of sodium hydroxides 2 M and the mixtures was slowly stirring at 80 $^{o}$C for 17 hours. The solution kept at pH 10 to form blue solid bulky material. After that, the blue material was washed with water and kept overnight at 60 $^{o}$C to obtain Ni/Al layered double hydroxides. Characterization of Ni/Al layered double hydroxides was performed using XRD powder analysis and IR spectroscopy.

### Synthesis of Zn/Al Layered Double Hydroxides

Synthesis of Zn/AL layered double hydroxides using co-precipitation method at pH 10. The mixtures solution zinc nitrate (10 mL, 0.75 M) and alumunium nitrate (10 mL, 0.25 M) added dropwise 10 mL of solution sodium hydroxides 2 M to form white gel. The white gel was kept at 80 $^{o}$C for 4 hours and then wash with water to form white material. White solid material was kept at 60 $^{o}$C overnight to obtain Zn/Al layered double hydroxides. Characterization of Zn/Al layered double hydroxides was performed using XRD powder analysis and IR spectroscopy.

### pH *PZC Analysis*

pH point zero charges (pH PZC) was conducted using sodium chloride 0.1 M. pH solution of sodium chloride 0.1 M was adjusted from pH 2 to 11 by addition of sodium hydroxide or hydrochloric acid 0.1 M. In the series of sodium chloride solution with various pH, Zn/Al layered double hydroxides was added. The mixtures were shaken for 24 hours then pH of filtrate was determined by pH meter. pH PZC was obtained by comparison initial and final pH solution.

### Adsorption Experiment

In order to determine the parameters adsorption of Ni/Al and Zn/Al layered double hydroxides, adsorption was studied by the effect of adsorption times, initial concentrations, temperatures and pH solutions. Stock solution of direct yellow 1000 mg/L was prepared by 1 g direct yellow dye dilute with deionized water. Standart solution of direct yellow was prepared ranging from 150-20 mg/L was prepared.
*Effect of pH adsorption*

The pH of direct yellow solution was adjusted by varying pH 2-11. Layered double hydroxides was added to the dye on the various pH, respectively. Then, shaking in equilibrium time and after that the sample were filtered. Filtrate was analyzed using UV-Visible Spectrophotometer.

*Effect of adsorption time*

Adsorption kinetics was studied by varying adsorption times. Dye with concentration 50 mL was added into 0.05 g Ni/Al and Zn/Al layered double hydroxides respectively. Then shaking in varying time after that the sample was filtered. Time of adsorption was adjusted for 10, 20, 30, 40, 50, 60, 90, 120, 150 and 180 minutes. Filtrate was analyzed using UV-Visible Spectrophotometer.

*Effect of initial concentration and temperature*

Thermodynamics was studied by varying initial concentration of direct yellow. Various concentration of dyes was added to 0.05 g Ni/Al and Zn/Al layered double hydroxides respectively. Then, shaking in equilibrium time and varying temperature ranging 303-343 K after that the mixture was filtered. Filtrate was analyzed using UV-Visible spectrofotometer.

## RESULTS AND DISCUSSION

### Adsorbent Characterization

*X-ray Diffraction*

The XRD pattern of Ni/Al and Zn/Al layered double hydroxides was shown in Fig. 1. Fig. 1 (a) showed diffraction of Ni/Al layered double hydroxide and Fig. 1 (b) showed diffraction of Zn/Al layered double hydroxide. The unique structure of layered double hydroxides for both Ni/Al and Zn/Al was identified at 11° as interlayer space. The data of interlayer gallery showed that Ni/Al and Zn/Al layered double hydroxides have basal spacing 7.65 Å and 7.57 Å, respectively.

*Infrared Spectroscopy*

The FTIR spectra of Ni/Al and Zn/Al layered double hydroxides are shown in Fig. 2. The intensity at wavenumber 3448 $cm^{-1}$ is assigned to the OH stretching vibration, at wavenumber 1635 $cm^{-1}$ is OH bending in the interlayer water molecule. Also, the intent band 1380 $cm^{-1}$ is shown as stretching of nitrate anion in the interlayer. A strong band at 1000 $cm^{-1}$ was identified as M-O vibration where M is Zn and Al. The weak shoulder at 424 $cm^{-1}$ is identified as Zn-O-Al and bands in the range 600 $cm^{-1}$are attributed to Ni-O-Al.

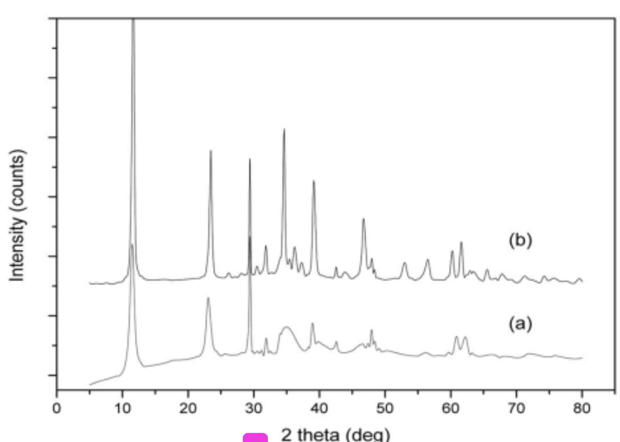


**FIGURE 1**. XRD pattern of Ni/Al and Zn/Al layered double hydroxides

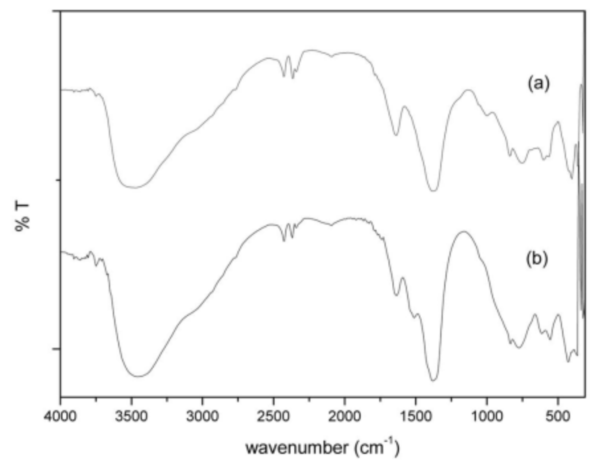


**FIGURE 2**. FTIR Spectrum of NiAl (a) and ZnAl (b) layered double hydroxides

## Effect of pH

The pH is an important parameter in adsorption process since it can control the uptake mechanism of the adsorbate as well as surface site on adsorbent. The effect of pH adsorption of direct yellow dye into NiAl and ZnAl layered double hydroxides was studied at pH range of 2-11. The adsorption was shown in Fig. 3. Fig. 3 shows decreasing adsorption capacity at the same time increasing pH. The result showed that the maximum at around pH 4 and 5 for each NiAl and ZnAl layered double hydroxides, respectively. The adsorption of anionic dye can be maximum in positive charge sites on adsorbent surface. As the pH of system increases, the concentration of $H^+$ is decreases. Therefore, the surface sites on adsorbent become negative charge. So, adsorption capacity of adsorbent decreasing. The adsorption of dye molecules was facilitated by positive charge density on adsorbent surface.

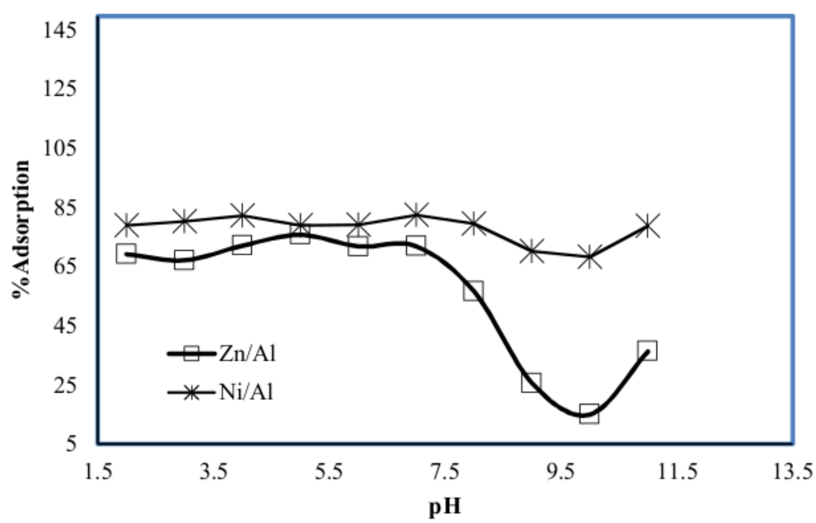


**FIGURE 3**. Effect of pH adsorption of direct yellow dye into NiAl and ZnAl layered double hydroxides.

## Effect of Contact Time and Kinetic Parameters

In order to establish the equilibrium time for maximum adsorption, the adsorption of direct yellow dyes was investigated as the function of contact time. The adsorption of direct yellow by Ni/Al and Zn/Al layered double hydroxides are shown in Fig. 4. Fig. 4 shows that Ni/Al and Zn/Al layered double hydroxide increased slowly after an hour with maximum uptake 79.02 mg/g and 76.23 mg/g, respectively. The fast removal adsorption can happen because the adsorbent surfaces have large number of site to adsorb dye solution, then the rate of adsorbent was decreased when the surface site full of dye accumulated.

In order to identify the kinetics parameter adsorption process, two kinetic models i.e, Pseudo-first-orde and pseudo-second-order are applied. The kinetics parameters is calculated using the pseudo-first-order and pseudo-second-order equations as follows:

$$\log (Q_e - Q_t) = \log Q_e - \left(\frac{k_1}{2{,}303}\right) t \quad (1)$$

$$\frac{t}{Qt} = \frac{1}{k_2 Qe^2} + \frac{1}{Qe} t \quad (2)$$

Where qe and qt are capacity of adsorbed (mg/g) at equilibrium and t is contact time (min), $k_1$ is the rate constant of Pseudo-First-Orde. Then, $k_2$ is the rate constant of pseudo-second-order.

The result was shown in Table 1. Table 1 was calculated value of $k_1$ and $k_2$, $qe_{exp}$, $qe_{calc}$ together with $R^2$. The value of correlation coefficient NiAl and ZnAl $R^2$ is 0.999 for pseudo-second-order model was better fitted than pseudo-first-orde for adsorption direct yellow by Ni/Al and Zn/Al layered double hydroxides, respectively.

**TABLE 1**. Kinetics Parameters Adsorption of Direct Yellow Dye by Ni/Al and Zn/Al Layered Double Hydroxides

| kinetics models | parameters | Adsorbent | |
|---|---|---|---|
| | | Ni/Al | Zn/Al |
| Pseudo-first-order | $qe_{exp}$ (mg/g) | 82.782 | 79.871 |
| | $qe_{calc}$ (mg/g) | 17.172 | 24.066 |
| | $k_1$ (min$^{-1}$) | 0.015 | 0.024 |
| | $R_2$ | 0.842 | 0.925 |
| Pseudo-Seconds-order | $qe_{exp}$ (mg/g) | 82.782 | 79.871 |
| | $qe_{calc}$ (mg/g) | 83.951 | 82.040 |
| | $k_1$ (min$^{-1}$) | 0.002 | 0.002 |
| | $R_2$ | 0.999 | 0.999 |

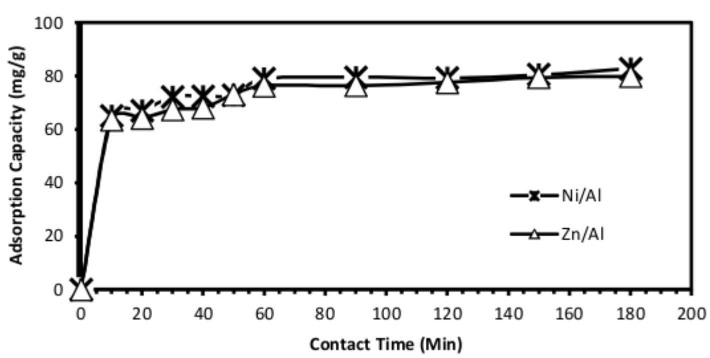


**FIGURE 4.** Time adsorption of direct yellow by Ni/Al and Zn/Al layered double hydroxides

## Adsorption Isotherm and Thermodynamic Study

The adsorption isotherms for direct yellow dye using Ni/Al and Zn/Al layered double hydroxides at varying temperature 303 K, 323 K and 343 K were shown in Fig. 5. The isotherms models Langmuir and Freundlich are used for this data. The Langmuir assumed that adsorbate was occupied into monolayer. Its used equation as follows:\

$$\frac{C_e}{q_e} = \frac{1}{kL\, q_{max}} + \frac{C_e}{q_{max}} \quad (3)$$

Where $q_e$ is the equilibrium adsorption, $C_e$ is equilibrium concentration, $q_{max}$ is the maximum adsorption and $k_L$ is the equilibrium adsorption constant. Then, the essential features of Langmuir isotherm namely $R_L$ (equilibrium parameters). Value $R_L$ has indicated the models of isotherm. If irreversible, the $R_L$ calculated zero ($R_L = 0$), liniear when $R_L = 1$, and favorable when $0 > R_L > 1$.

The Freundlich isotherm model identified the heterogenous adsorbent surface. The equation is following:

$$\text{Log } q_e = \text{Log } k_F + n \text{ Log } C_e \quad (4)$$

Where $k_F$ is adsorption capacity when equilibrium. Therefore, both the isotherm models are shown in Table 2. Fig. 5 shows that the Freundlich models fits the experimental data better than Langmuir models were indicated that the adsorbate interacted each other on surface sites of Ni/Al and Zn/Al layered double hydroxides, respectively. So this phenom is physisorption as principal adsorption process. Tabel 2 shows the Freundlich isotherm models of $n_F$ values and Langmuir $R_L$ obtained support the favorable adsorption of direct yellow dye by Ni/Al and Zn/Al layered double hydroxide.

The thermodynamic parameters were shown in Table 3. The values of enthalpy were investigated endothermic. The entropy was decreased with increasing concentration. Each concentration, energy changes of adsorption direct yellow by Ni/Al and Zn/Al layered double hydroxides is more negative with increases temperature. This phenom was indicated the spontaneity of adsorption process. The thermodynamic was analyzed by temperature effect shows that the adsorption process is endothermic and spontaneous.

**TABLE 2**. Langmuir and Freundlich Isotherm Models

| Correlation Parameter | T= 303 K | | T= 323 K | | T= 343 K | |
|---|---|---|---|---|---|---|
| Langmuir | Ni/Al | Zn/Al | Ni/Al | Zn/Al | Ni/Al | Zn/Al |
| $Q_{max}$ | 255.1028 | 307.3420 | 121.8437 | 166.9001 | 145.3543 | 164.4710 |
| $K_L$ | 0.0071 | 0.0046 | 0.0409 | 0.0156 | 0.0553 | 0.0228 |
| $R_L$ | 0.00026-0.00078 | 0.00021-0.00065 | 0.000054-0.00016 | 0.000039-0.00011 | 0.000045-0.00013 | 0.000045-0.00012 |
| R2 | 0.817 | 0.345 | 0.847 | 0.713 | 0.829 | 0.818 |
| Freundlich | Ni/Al | Zn/Al | Ni/Al | Zn/Al | Ni/Al | Zn/Al |
| $K_f$ | 3.286 | 2.604 | 16.593 | 6.882 | 21.301 | 9.571 |
| n | 1.303 | 1.265 | 2.507 | 1.676 | 2.431 | 1.780 |
| $R^2$ | 0.992 | 0.949 | 0.906 | 0.937 | 0.905 | 0.953 |

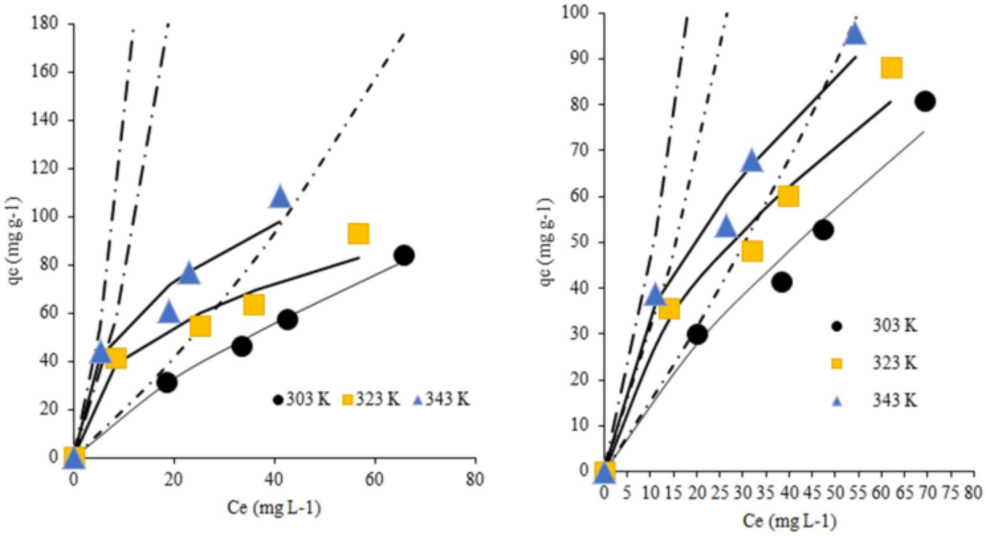


**FIGURE 5.** Adsorption isotherm, Langmuir and Freundlich models Ni/Al (a) and Zn/Al (b) layered double hydroxides. The solid line represents the model fitting Freundlich, the dash line represent the model fitting Langmuir, and the symbol represents experimental data.

**TABLE 3**. Values of Thermodynamic Parameters for The Adsorption of Direct Yellow Dye By Ni/Al And Zn/Al Layered Double Hydroxides

| T (K) | Concentration (mg/L) | Ni/Al ΔG (kJ/mol) | Ni/Al ΔS (J/mol.K) | Ni/Al ΔH (kJ/mol) | Zn/Al ΔG (kJ/mol) | Zn/Al ΔS (J/mol.K) | Zn/Al ΔH (kJ/mol) |
|---|---|---|---|---|---|---|---|
| 303 | | -1.506 | | | -1.049 | | |
| 323 | 50 | -3.906 | 120.004 | 34.855 | -2.348 | 64.961 | 18.634 |
| 343 | | -6.306 | | | -3.647 | | |
| 303 | | -0.831 | | | -0.199 | | |
| 323 | 80 | -2.075 | 62.181 | 18.010 | -1.104 | 45.216 | 13.501 |
| 343 | | -3.318 | | | -2.008 | | |
| 303 | | -0.066 | | | -0.245 | | |
| 323 | 100 | -1.758 | 54.704 | 15.911 | -1.175 | 46.498 | 13.844 |
| 343 | | -2.852 | | | -2.105 | | |
| 303 | | -0.501 | | | -0.378 | | |
| 323 | 150 | -1.579 | 53.563 | 15.722 | -0.984 | 30.336 | 8.814 |
| 343 | | -2.650 | | | -1.591 | | |

## CONCLUSION

The adsorption of direct yellow dye by Ni/Al and Zn/Al layered double hydroxides was used pseudo-second-orde with the rate is 0.002 $min^{-1}$. The adsorption of direct yellow dye by Ni/Al layered double hydroxide have better adsorption capacity than Zn/Al layered double hydroxide. The surface site of adsorbent was investigated by pH when pH decreases the surface site of adsorbent is positively and whereas pH increases the surface site of adsorbent is negative. Ni/Al and Zn/Al layered double hydroxides have a maximum at pH 4 and 5, respectively. The thermodynamic parameters were indicated that the adsorption process is endothermic and spontaneous. Therefore, Ni/Al and Zn/Al layered double hydroxides have effective adsorbent for direct yellow dye in wastewater.

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