Synthesis and Characterization of Low-Cost Adsorbent and used for Alizarin Yellow GG and Alizarin Red S Dyes Removal from Aqueous Solutions

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Abstract. The effective use of a synthetic clay as ionic solid adsorbent for the removal of alizarin yellow GG and alizarin red S from aqueous solutions was investigated as low-cost adsorbent. Anionic clay of layered double hydroxide (LDH) was prepared from the salts of magnesium-aluminium nitrate in a ratio of (5:1) by co-precipitation method, characterized by X-ray diffraction (XRD), FT-IR spectroscopy and thermogravimetric analysis (TGA) to confirm the presence of LDH. The effect of various experimental parameters like contact time, initial and concentrations were investigated. The experiments were carried out in a batch system to optimize operation variables. The Langmuir and Freundlich isotherm equations applied to the data and the isotherm analysis indicated that the equilibrium was fitted to both of Freundlich and Langmuir. The models showed physio – chemical sorption for both of Alizarine yellow GG and Alizarin Red S on LDH surface. Kinetics study was made using laggergreen equations and the results show that the sorption of Alizarine Yellow GG and Red dye uptake on layered double hydroxide (LDH) were fitted with both pseudo-first order until the adsorption reach equilibrium, then, they fellow pseudo second order where the adsorption rates depend on the amount of adsorbent and concentration of adsorbate. The partition coefficient Kd for the sorption of alizarine yellow GG and alizarin red S on LDH were also determined and it was indicated that the value of Kd increases with time until equilibrium is reached and becomes constant because of the extra unoccupied sites until the adsorption reaches equilibrium.

Keywords. Synthesis, Alizarin Yellow GG, Alizarin Red S Dyes, Aqueous Solutions, LDH, TGA, XRD.

1. Introduction
Due to the rapidly growing world population and rapid industrialization, dyes are widely used in many industries, mainly in textile industries. Many of these dyes are commonly considered to be hazardous and toxic to living organisms [1]. The dyes are carcinogenic and toxic which may cause serious effects such as, allergies, skin itching or sores, allergic reaction in the eyes, and
irritation in the mucous membrane and the respiratory tract [2]. Therefore, the removal of dye is an important aspect of wastewater treatment before discharge. The anionic dyes alizarin yellow GG (AY) and alizarin red-S (AR) have a carcinogenic effect causing serious problems to aquatic vegetal life and environmental problems [3], because of the complex aromatic structure of these dyes, that afford high physicochemical and thermal stable to biodegrade [4]. A number of physical, chemical and biological treatment techniques and methods have been developed for the removal of the pollutants in water and wastewater [5]. Although several of these operations and processes are combined in most treatment systems, they are usually considered separately [6]. These include: physical methods such as membrane-filtration processes (nanofiltration, reverse osmosis, electrodialysis) and sorption techniques, chemical methods such as coagulation or flocculation combined with flotation and filtration, precipitation [7-11]. Among these processes the adsorption process considered as one of the most efficient technique for the treatment and removal of organic pollutants in waste water treatment. The advantages of adsorption process, to the other processes due to easy of operation, low costs, high efficiency and low energy requirements [12] and [13]. So, the progress of research and development interest is in testing and using low-cost, easily available materials for dye removal. The main attractions of a synthetic clay as adsorbents which are prepared from layered double hydroxide are its high selectivity and efficiency, cost effectiveness and efficient removal of dyes from large volumes over a number of sorption/desorption cycles. Layered double hydroxides (LDHs) are a group of anionic clay-like materials with unique layered structures [14]. The structure of all LDH consists of positively charged mixed metal hydroxide layers separated by charge balancing anions and water molecules [15]. The general chemical formula of these layered double hydroxides can be written as:

\[ \text{M(II)}_{1-x}\text{M(III)}_x(\text{OH})_2(\text{A}^n)^{n/x} \cdot y\text{H}_2\text{O} \]

Where; M (II) is divalent cation, M (III) is trivalent cation, A is interlayer anion, n- is charge on interlayer ion, and x and y are fraction constants. [16]. The main properties of (LDHs) are their ability to substitute their interlayer anions with preservation of the layered structure [17]. Type of materials are well known for their applications such as catalyst or as catalysts supporters [18] ion exchangers [19] or biosensors based on clay modified electrodes [20]. Also, it is used as an adsorbent to remove dyes from effluents of textile, plastic and paper industries. Thermal treatment of LDH can lead to breakdown of the layered structure and the formation of the mixed metal oxides with surface area [21]. Many researchers have been working for the preparation of low-cost adsorbents. Exploration of good low-cost adsorbent may contribute to the sustainability of the environment and offer promising benefits for the commercial purpose in future. The nature of adsorbent is more effective factor in adsorption progress for removal of pollutants [22]. The purpose of this research is based on searching for economical, available starting raw materials and low-cost adsorbent for removal of Alizarin yellow G and Alizarin red S dyes from aqueous solutions.

2. Materials and methods

2.1. Materials
Alizarin Red S (sodium 3,4-dihydroxy-9,10-dioxo-9,10-dihydroanthracene-2-sulfonat) (97% purity) and Alizarin Yellow G (5-(3-Nitrophenylazo)salicylic acid sodium salt) (94% purity) were purchased from Sigma-Aldrich. And Mg(NO₃)₂.6H₂O, of Al(NO₃)₃.9H₂O and NaHCO₃, were purchased from Fluka. All the chemical reagents were used without further purification.

2.2. Preparation of the adsorbent layered double hydroxide
The adsorbent layered double hydroxide Mg₆Al₂OH₁₆CO₃. The solution 4H₂O was prepared by the co-precipitation method [23] and [24]. A solution containing (2M) NaHCO₃ and (0.2M) NaOH was added to 400 ml of distilled water producing a solution of (pH10). Another solution
was prepared containing a mixture of (46.15g) of Mg(NO₃)₂·6H₂O and (22.5g) of Al(NO₃)₃·9H₂O in 400 ml of distilled water with ratio Mg/Al (5:1), (all chemicals were used from Aldrich). Both solutions were heated to 70 °C and combined together through a Pyrex glass T-piece, adjusting the flow rate to maintain pH 10 with continuous stirring. Once addition was completed, the slurry mixture was stirred at 70 °C for one hour then cooled to room temperature for 24 h. After cooling, the resulting precipitate was filtered and washed several times with distilled water and dried at 70 °C. The dry powder (LDHs) was analyzed by XRD, TGA and FT-IR.

2.3. Preparation of calibration curve
A stock solution of Alizarin yellow GG (AY) and Alizarin red S (AR) dyes have been prepared by dissolving (1 g) of each dye in one liter of distilled water. The stock solutions were appropriately diluted to prepare a series of different concentration at the range (2 – 100) mg/L for AY and from (2 – 40) mg/L for AR. The calibration curve of initial concentrations (Ci) of AY was obtained by measuring the absorbance of known concentration at λ_max = 360 nm and for AR at λ_max = 470 nm using ATI Unicam UV/Vis – spectrometer.

2.4. Effects of contact time
The effect of contact time and initial concentration of AY and AR dyes solution on the sorption of layered double hydroxide (LDH) were studied using two series bottles labeled from (1- 13) at fixed temperature, 25 °C. A 0. One g of LDH adsorbent powder was placed in each bottle then 25 ml volume of 50 mg/L of each dye solution was added to each bottle. At interval times, a required volume from each bottle was taken and centrifuged at 7000 rpm for 10 min, (Hermle Z 160 M centrifuge), then the dye absorbance was measured by UV/Vis-spectroscopy at a fixed wavelength for each dye.

2.5. Adsorption isotherm studies
The adsorption isotherm experiments were carried out at room temperature in batch mode. Exactly 25 ml of the dye solution of each known initial concentration (from 10 – 70 mg /L) were added to 0.1 g of LDH adsorbent then shaken for 45 minutes and left to stand for 1 hr. These were centrifuged for 10 min at 7000 rpm before the solution was measured by UV/Vis – spectroscopy at λ_max = 360 nm for AY and λ_max = 470 nm for AR dye. The amount of adsorbed dye was calculated according to the following equation [25]:

\[
q_e = \frac{(C_i - C_e)V}{m}
\]

Where, \(q_e\) is the amount of dye adsorbed (mg/g) and \(C_i\) is the initial concentration of adsorbed (mg/L), \(C_e\) is the concentration at equilibrium time (mg/L), \(m\) is the mass of adsorbent (g) and \(V\) is the volume of dye solution (ml).

3. Results and discussions

3.1. Characterisation of LDH
LDHs contains two or more varieties of metallic cations. Anionic species are included in the primary layers as well as in the hydrated interlayer domains [26]. Mg-Al/CO₃ has been synthesized by co-precipitation method as shown by the following chemical reaction:

\[
5\text{Mg(NO}_3\text{)}_2\cdot6\text{H}_2\text{O} + 2\text{Al(NO}_3\text{)}_3\cdot9\text{H}_2\text{O} + 13\text{NaOH} + \text{NaHCO}_3 \rightarrow \text{Mg}_5\text{Al(OH)}_{13}\text{CO}_3 + 13\text{NaNO}_3 + 15\text{H}_2\text{O}
\]
Figure 1 shows XRD pattern of the hydrotalcite type phase being characterized by synthesized layered double hydroxide, more intensive and sharper reflections of the (003) and (006) planes lower 2Θ value (11°) and (23°) respectively. Broad asymmetric reflections at higher 2Θ (34 - 66°) can be observed in the XRD patterns. The peaks for the (003) and (006) plans indicate that it has the best crystallinity. The difference in intensities of the reflection shows different degree of crystallinity. The resulting spectrum is in accordance with reference library of patterns. The (009) reflection overlaps with the (012) resulting in a abroad signal.

The functional groups on the adsorbent play a very important role in the adsorption and understand the adsorption process. Figure 2 shows the FT-IR spectrum of Mg-Al/CO₃ LDH, a broad band located at 3450 cm⁻¹ is stretching vibration both of hydroxide group layers and interlayer water molecules. The strong band at 1383 cm⁻¹ is characteristic of the intercalated carbonate group. A sharp band located at 790 cm⁻¹ is assigned to the Mg-O vibration [27]. However, the peak position can be varied with different ratio of cations. A decrease in the cationic molar ratio has the effect of shifting to lower wave number values and the reverse is also true [28].

Thermal analysis (TGA, DSC) in figure 3 shows the thermal decomposition of the hydrotalcite-like layered Mg-Al/CO₃ LDH. A heating rate of 10 °C min⁻¹ in air atmosphere and a temperature range of 30 °C 850 °C with ceramic sample holder has been used. Dehydration, dihydroxylation (loss of lamellar hydroxyls) and loss of interlayer carbonate are the steps of thermal decomposition of hydrotalcite. Mg₅Al(OH)₁₅CO₃ LDH undergo thermal decomposition at higher
temperatures shows that the mass changes up to 200 °C due to the removal of interlayer and adsorbed water. Step two of TGA curve between 200 and 500 °C, indicates to the decomposition of interlayer carbonate and hydroxyl groups of the lamellae [29].

Figure 3. TGA analysis of Mg₅Al(OH)₁₃CO₃ synthesised at pH 12.

3.2. Effect of contact time

The effect of contact time and dye concentration on the removal of AY and AR dyes by LDH from aqueous solution is illustrated in Figure 4, showing a plot between the amount of adsorption (mg/g) and interval time. Fifty mg/L of each dye conduct with the 0.1 g of LDH adsorbent at the same volume. It was observed that the adsorption of each dye on the surface of LDH was rapid in the initial stages and up to the first 15 minutes contact at all initial dye concentrations understudy. After the 15 minutes, the adsorption efficiency reached equilibrium and stabilized at a maximum value.

Figure 4. Effect of contact time for the adsorption of AY and AR adsorption on LDH at 25 °C.
3.3. Calibration curve
A calibration curve for Alizarin yellow GG and alizarin red S have been prepared by plotting the measured absorption against concentration at fixed maximum wave length as shown in Figure 5 and Figure 6. The chemical structures of each dye and the absorption coefficient for AY and AR have been determined with value of (4.39 L mg⁻¹ cm⁻¹ and 1.08 L mg⁻¹ cm⁻¹).

![Calibration curve of alizarin yellow GG (AY).](image)

![Calibration curve of alizarin red S (AR).](image)

3.4. Kinetic studies
The adsorption kinetic study is very important to identify the efficiency and the mechanism of adsorption as it describes the solute uptake rate at which the solid-solution interface and the possible rate-controlling. In the present study, the adsorption kinetics of AY and AR on the surface of LDH adsorbent have been studied. The adsorption kinetics of both dyes onto LDH adsorbent were investigated with two kinetic models, namely the Lagergren pseudo—first—order and pseudo—second—order model [21]. The Lagergren rate equation is one of the most widely used adsorption rate equations for the adsorption of solute from a liquid solution. The pseudo—first—order kinetic model (Eq.3) can be expressed by the following equation:

\[
\log(q_e - q_t) = \log q_e - \frac{k_1}{2.303} t
\]

where \(q_t\) is the amount of dye adsorbed in mg g⁻¹ at time (t), \(q_e\) is the maximum adsorption capacity and \(k\) is the first-order rate constant in (min⁻¹). The plot versus time (t) gives a slope equal to (-k1/2.303) and the intercept equal to (log \(q_e\)) to determine the adsorption capacity and adsorption rate constants as shown in Fig. 7 for the adsorption of AY on LDH and Fig. 8 for the adsorption of AR on LDH.

The second—order kinetics of adsorption was analyzed by using Largerger equation expressed as [30].

\[
\frac{dq}{dt} = k_2 (q_e - q_t)^2
\]

(4)

When arranging equation (4), to the following equation is obtained:

\[
\frac{d(q_e-q_t)}{(q_e-q_t)^2} = -k_2 dt
\]

(5)
When we apply the conditions of integration if \( t=0 \), \( q_t =0 \), the dye is not adsorbed on the adsorbent at time equal to zero, but after duration of time the molecules of dye are adsorbed on adsorbent surface at \( t=t \), \( q_t=q_e \) the integrated form of equation (4) becomes:

\[
\frac{1}{(q_e-q_t)} = \frac{1}{q_e} + k_2 t
\]  

Equation (7) can be rearranged to obtain a linear equation form:

\[
\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e}
\]

where \( K_2 \) is the rate constant of second order in \( \text{mg g}^{-1} \text{min}^{-1} \) and \( q_e \) is the equilibrium adsorption capacity in \( \text{mg} / \text{g} \). The values of \( q_e \) and \( k_2 \) are obtained from the slope and intercept, respectively when \((t/q_t)\) is plotted against time \((t)\) to determine the adsorption capacity and adsorption rate constants as shown in Figure 9, for the adsorption of AY on LDH, and Figure 10 for the adsorption of AR on LDH. The kinetic adsorption of both dyes shows that the best correlation for the system provided by the pseudo-first order model from the starting of adsorption up to 20 minutes might be due to more empty sites on the surface of adsorbent and the high concentration of adsorbate. After the 20 minutes, the data were best fit to pseudo second order kinetic according to the correlation factors and that it might be due the occupation of all sites of the adsorbent surface and adsorption reached to equilibrium. The results of kinetics parameters are listed in Table (1)

**Figure 7.** First order kinetic plot for the sorption AY on LDH.  
**Figure 8.** First order kinetic plot for the sorption AR on LDH.  
**Figure 9.** Second order kinetic plot for the sorption AYG on LDH.  
**Figure 10.** Second order kinetic plot for the sorption AR on LDH.
Table 1. Kinetic parameters for sorption of Alizarin Yellow GG (AY) and Alizarin Red S (AR) on layered double hydroxide (LDH) adsorbents.

| Adsorbents | AY | AR |
|------------|----|----|
| R²         | 0.9269 | 0.9346 |
| 1st order  | K (min⁻¹) | 0.158 | 0.122 |
|            | q_{max} (mg/g) | 53.05 | 38.61 |
|            | R²         | 0.9868 | 0.9906 |
| 2nd order  | K₂(L.mg⁻¹.min⁻¹⁻¹) | 0.071 | 0.008 |
|            | qₑ (mg/g)  | 26.31 | 27.01 |

3.5. Partition coefficient (Kd)

The Partition Coefficient is defined as the concentration of solute in the adsorbed phase divided by the concentration of the solute in the solution phase. Thus, Kd is a factor related to the partitioning of a solute between the solid and the aqueous phases which can be determined by the following equation:

\[ K_d = \frac{C_s}{C_e} \]  \quad (8)

where, \( K_d \) = adsorbent/water equilibrium partition coefficient, \( C_s \) = dye concentration in solid adsorbent, and \( C_e \) = dye concentration in water. The value of \( K_d \) for the sorption of each dye on LDH with time interval were determined and listed in Table 2. The results show that the \( K_d \) values increases with time until the sorption reaches equilibrium and becomes constant. The proportion for both dyes of AY and AR on LDH adsorbent increased possibly due to the large number of adsorption sites that are available on the adsorbent surface from the beginning of adsorption until when sites were filled up by dye molecules over time [31].

Table 2. Partition coefficients (Kd), for the adsorption of Alizarin Yellow GG and Alizarin Red S on layered double hydroxide adsorbents.

| Time (min) | AY | AR |
|------------|----|----|
| 3          | 1.05 | 0.025 |
| 6          | 1.05 | 0.052 |
| 9          | 1.11 | 0.113 |
| 12         | 1.43 | 0.431 |
| 15         | 2.15 | 1.823 |
| 18         | 2.96 | 16.937 |
| 21         | 17.19 | 22.666 |
| 27         | 23.66 | 22.666 |
| 30         | 12.21 | 13.5 |
| 40         | 14.5 | 8.34 |
| 50         | 17.46 | 4.33 |

3.6. Adsorption isotherms

The experimental data have been analyzed by the linear forms of the Freundlich and Langmuir model isotherms represented by the following equations. Freundlich isotherm;

\[ \log q_e = \log K_f + \frac{1}{n} \log C_e \]  \quad (9)

Where, \( q_e \) is amount of adsorbed at equilibrium mg g⁻¹, \( C_e \) is the concentration at equilibrium mg L⁻¹, \( K_f \), Freundlich constant refers to the adsorption capacity and \( n \) is Freundlich constant related to the intensity of adsorption. The plot \( \log q_e \) against \( \log C_e \) gives a slope equal to \( (1/n) \), with intercept equal to \( \log K_f \) as shown in figures (11) and (12). And Langmuir isotherm is equal to:

\[ \frac{C_e}{q_e} = \frac{1}{K_L b} + \frac{C_e}{K_L} \]  \quad (10)

Where \( K_L \) is Langmuir constant related to adsorption capacity mg g⁻¹ and \( b \) Langmuir constant related to intensity of adsorption L mg⁻¹. Freundlich and Langmuir adsorption isotherms were applied by using straight line equations (9) and (10) for evaluated adsorption isotherm constants of the sorption of AY and AR on LDH. The plot \( C_e/q_e \) against \( C_e \) gives a straight line producing a slope equal to \( (1/K_L) \) with intercept equal to \( (1/b K_L) \) as shown in figures (13) and (14). The adsorption isotherms parameters were determined and listed in table (3). The results show that the
adsorption of both AY and AR dye on LDH were more fit to Langmuir model. These results suggest that the adsorption of AY and AR dyes on LDH are a chemical adsorption due to forming a monolayer on the LDH surface conducted strongly to dye molecules [32].

Table 3. Isotherms constants of Freundlich & Langmuir for adsorption of Alizarin yellow GG and Alizarin Red S on Layered double hydroxide (LDH) at 25 °C.

| Adsorbates | Freundlich constants | Langmuir constants |
|------------|----------------------|---------------------|
|            | K_f (mg/g) | n (L/mg) | R² | K_L (mg/g) | b (L/mg) | R² |
| AYG        | 4.683      | 3.79      | 0.8636 | 14.14      | 0.089 | 0.9491 |
| ARS        | 0.782      | 0.942     | 0.8185 | 29.41      | 0.040 | 0.8661 |

4. Conclusion
Low cost of clay magnesium aluminum layered double hydroxide (Mg/Al CO₃) adsorbent has been prepared by coprecipitation method and characterized with XRD, FT-IR and analysis. The ability of this adsorbent was used for Alizarin yellow GG and Alizarin Red S removal from aqueous solution. The adsorption isotherms and kinetic equilibrium for adsorption of AY and AR dyes on LDH surface were studied. The results show that the adsorption isotherms were more fit with Langmuir model than Freundlich model which indicted to chemisorption type. In addition, the kinetic equilibrium study of the adsorption of both dyes on LDH were investigated and the results show that the kinetics of the process followed the pseudo-second-order before the
adsorption reached equilibrium, but when the adsorption reached the equilibrium, they followed the pseudo second order model.

5. References
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