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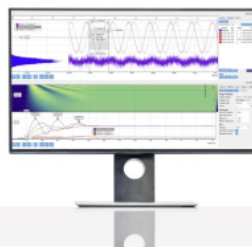
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Preparation of MgAl LDH Intercalated by α -PW₁₂O₄₀³⁻ for Adsorptive Removal of Direct Violet Dye from Aqueous Solution

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Abstract. The preparation of polyoxometalate-intercalated MgAl layered double hydroxides (LDH) has been conducted and employed as an adsorbent for direct violet dye removal from aqueous solution. The MgAl LDH was firstly prepared by coprecipitation method under basic condition then followed by an ion-exchange process with K₃[α -PW₁₂O₄₀].nH₂O solution. The obtained materials were characterized by XRD and TG-DTA. Related to the X-ray diffraction pattern, the synthesized MgAl LDH exhibited typical diffraction peak at 2 θ of 11.56° correspond to the basal distance of 7.72 Å. While after intercalated by [α -PW₁₂O₄₀]³⁻, the typical diffraction peak of MgAl LDH shifted to the lower diffraction angle of 8.88° that corresponds to the basal spacing of 9.95 Å. The utilization of the prepared materials as an adsorbent for direct violet dye removal from aqueous solution showed that the intercalated LDH exhibited better adsorption capacity than the pristine MgAl LDH.

INTRODUCTION

Clean water scarcity is one of the most detrimental problems nowadays [1]. The rapid urbanization and industrialization have caused depletion of clean water resource. Not only for necessity and excessive use, but clean water depletion also caused by the contamination of the water body by various toxic contaminants produced by various anthropogenic activities [2]. Among the contaminants that desecrated the water body and its resources, dyes contamination is the most noticeable one. Dye contamination commonly produced by various industries such as textile, leather goods, plastics, and cosmetics [3]. Among them, Textile industry has reported as the most responsible to the dyes contamination in the environment. As reported before, textile dyeing wastewater has become the third-highest industrial wastewater that contains various toxic substances like detergents, surfactants, and other recalcitrant organic molecules [4]. Moreover, due to the commonly used dyes in the textile industry were complex synthetic dyes, the textile industry effluents are having a highly toxic, carcinogenic, and teratogenic to the living organism and even human being.

As a consequence of the above-mentioned issues, the removal of dyes contamination from textile industry wastewater is highly necessary in order to maintain environmental sustainability. So far, many methods and technologies have been devoted and developed in order to reduce the dyes contamination in wastewater like bioremediation, coagulation, adsorption, oxidation and advance oxidation, and membrane filtration [5–7]. Among them, adsorption is considered as one of the most efficient methods to be applied in dyes contaminated wastewater treatment. However, the efficiency of the adsorption process was profoundly affected by the performance of the used adsorbent material. An adsorbent that has a good performance must have a high surface area or a pore structure and adequate equilibrium uptake time in order to remove the pollutant effectively [8]. The conventional adsorbent that has been used until to date are including alumina, silica gel, zeolite, and activated carbon. Unfortunately, the

above-mentioned material suffered from the high cost and limited regeneration ability. Therefore, various novel alternative materials that have better economical cost and adsorption capacity have been developed in order to get rid of the conventional adsorbent. Layered double hydroxides (LDHs) materials have long been investigated as a high potential adsorbent for removal of numerous pollutant from wastewater. For instance, MgAl LDH with uniform hexagonal thin plate-like form was successfully synthesized and exhibited high performance for congo red dye removal [9]. In this work, we have developed an LDH material that consists of Mg as M^{2+} metal cation and Al as M^{3+} metal cation. Furthermore, the synthesized MgAl LDH was further developed by intercalation method using polyoxometalate (POM) of $PW_{12}O_{40}$ anion in order to increase its interlayer galleries high. The prepared materials then employed as a promising adsorbent for removing the direct violet dyes from aqueous solution as a model of textile wastewater pollutant.

EXPERIMENTAL SECTION

Materials

The chemicals used in this work including hydrochloric acid (HCl), aluminium nitrate nonahydrate ($Al(NO_3)_3 \cdot 9H_2O$), magnesium nitrate hexahydrate ($Mg(NO_3)_2 \cdot 6H_2O$), sodium hydroxide (NaOH), sodium carbonate (Na_2CO_3), potassium chloride (KCl), disodium hydrogen phosphate dihydrate ($Na_2HPO_4 \cdot 2H_2O$), and sodium tungstate dihydrate ($Na_2WO_4 \cdot 2H_2O$) were in reagent grade. All the mentioned chemicals were purchased from Sigma Aldrich.

Preparation of MgAl LDH

MgAl LDH material was prepared according to work done by Extremera et al. [10] by the coprecipitation method. In brief, 50 mL of 0.3 M $Mg(NO_3)_2 \cdot 6H_2O$ was added into a 250 mL beaker flask containing 50 mL of 0.1 M $Al(NO_3)_3 \cdot 9H_2O$ under vigorous stirring. After homogeneously mixed, the obtained mixed metal solution was further transferred into 500 mL beaker flask containing 50 mL of 1 M Na_2CO_3 under vigorous stirring. In order to maintain the pH of the mixed solution, 2 M NaOH solution was also added simultaneously. After all the mixed metal solution was completely transferred, and the pH of the mixture was recorded constantly at pH 10, the obtained solid (white-colored) was then collected by vacuum filtration. The obtained solid was further rinsed with distilled water in order to remove the remaining mixed metal solution and followed by drying in airflow oven at 80 °C overnight. The final white solid product was then labeled as MgAl LDH.

Preparation of Intercalated MgAl LDH

The polyoxometalate (POM) α - $PW_{12}O_{40}^{4-}$ anion intercalated MgAl LDH was prepared by a simple ion-exchange method. Solution A was prepared by dissolving 1 g of $K_3[\alpha-PW_{12}O_{40}] \cdot nH_2O$ powder into 50 mL of distilled water. Solution B was also prepared by dissolving 2 g of the synthesized MgAl LDH into 25 mL of 1 M NaOH solution. Solution A then transferred into solution B immediately in an N_2 atmosphere condition then stirred for 24 h in room temperature. After finished, the solid product was separated by vacuum filtration and rinsed with distilled water several times. The obtained wet solid was then dried in airflow oven at 80 °C overnight and the final solid product was labeled as MgAl POM.

Materials Characterization

The synthesized materials, including pristine MgAl LDH and MgAl POM, were characterized by X-ray diffraction analysis and TG-DTA analysis in order to investigate the formation of the layered structure of the pristine MgAl LDH and the formation of the POM anion into the interlayer space of MgAl LDH. X-ray diffraction analysis was performed using a Rigaku MiniFlex 600 instrument equipped with CuK α irradiation at 30 kV and 10 mA. The samples were scanned from 2 θ range 5° to 80° with a scanning rate of 5°/min. The TG-DTA analysis was performed by using Shimadzu DTG-60H TG-DTA apparatus under N_2 atmosphere and heating range 25 – 900 °C.

Study of Direct Violet Dye Adsorption

The prepared POM intercalated LDH and the pristine MgAl LDH were further employed as an adsorbent for direct violet dye from aqueous solution. The stock solution of direct violet was firstly prepared by dissolving 1 g of solid direct violet powder with 1000 mL of distilled water. Next, the working dye solution and standard solution was prepared dye diluting the prepared stock solution. In typical, the direct violet dye adsorption was conducted by contacting 0.2 g of adsorbents with 50 mL of direct violet dye solution. The mixture was then stirred vigorously for definite time. After finished, the mixture was separated, and the remaining dye concentration was measured by UV-Vis spectrophotometry technique.

RESULTS AND DISCUSSION

The crystalline properties of the synthesized MgAl LDH and MgAl POM were investigated by X-ray diffraction analysis. **FIGURE 1** showed the X-ray diffraction pattern of both MgAl LDH and MgAl POM samples. It can be observed that the pristine MgAl LDH showed a typical diffraction peaks of double-layered material or *brucite-like* structure at 2θ angle of 11.45° , 22.96° , 29.53° , 32.06° , 34.97° , 48.07° , and 61.60° which correspond to the lattice plane of (003), (006), (009), (012), (015), and (110) respectively. As reported by Balcomb et al. [11], the spacing of lattice plane (003) refers to the interlayer distance of the LDH. In can be calculated that the pristine has interlayer distance of 7.72 Å.

After intercalated by Keggin POM anion, the typical diffraction peak of (003) plane shifted into the lower 2θ angle. This result indicated the expansion of interlayer distance that probably affected by the insertion of POM anion into the interlayer space of MgAl LDH. From the diffraction pattern of MgAl POM, it can be observed that after intercalation the (003) reflection peak moved from 2θ around 11.45° to 8.88° that corresponds with the increase of the interlayer distance from 7.72 Å to 9.95 Å. According to this finding, then can be concluded that the α -PW₁₂O₄₀⁴⁻ anion has been successfully intercalated into the interlayer space of MgAl LDH [12].

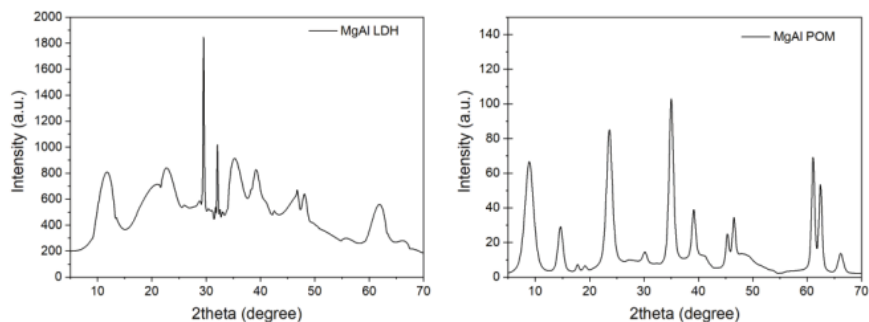


FIGURE 1. X-ray diffraction pattern of MgAl LDH and MgAl POM

The TG-DTA profile of both MgAl LDH and MgAl POM was presented in **FIGURE 2**. The TGA profile of MgAl LDH showed a strong endothermic peak at 100°C that corresponds to the release of the water molecule that presented in the interlayer space of LDH. The relatively weak endothermic peak observed at around 300°C correspond to the decomposition of interlayer anion such as NO_3^- or CO_3^{2-} that lied in the interlayer space of MgAl LDH. While the endothermic peaks observed at 500°C and 700°C correspond to the dehydroxylation and decomposition of MgAl LDH Structure [13]. The TGA profile of MgAl POM showed almost similar profile as MgAl LDH. However, the new endothermic peaks that observed at temperature around 180°C might be ascribed to the decomposition of the POM anion [12] as well as the increase of the endothermic peak at temperature around 320°C . That finding in agreement with the result of XRD analysis which concluded the success of POM anion intercalation into MgAl LDH interlayer space.

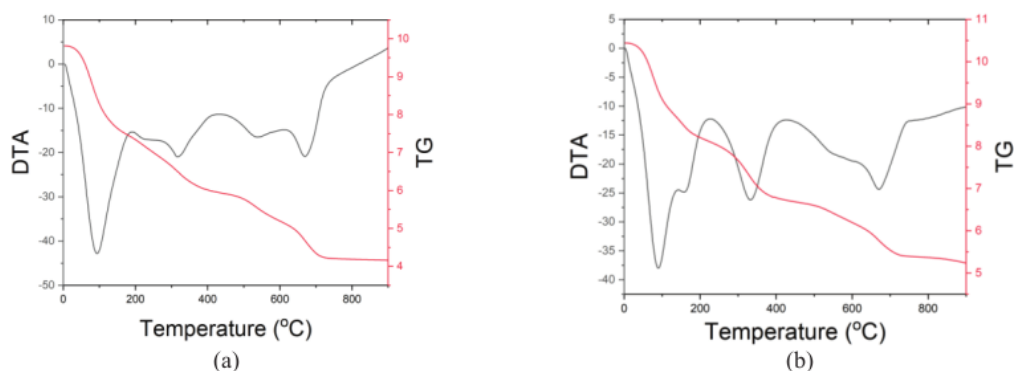


FIGURE 2. TG-DTA profile of (a) MgAl LDH and (b) MgAl POM

Next, the synthesized materials were tested as an adsorbent for direct violet dye removal from aqueous solution. The obtained results as the function of contact time to the removal of direct violet dye were presented in **FIGURE 3**. It can be observed that by increasing the contact time from 0 to around 40 minutes, the adsorption process occurred rapidly then increased gradually until the adsorption process achieved the equilibrium state after 120 minutes. It also observed that the MgAl POM exhibited slightly higher adsorption capacity rather than the pristine MgAl LDH. In the equilibrium time, the MgAl POM could adsorbent direct violet dye around 82 mg/L, while the pristine MgAl LDH only 80 mg/L. This finding indicated that the intercalated LDH exhibited a slight increment performance compared with the pristine LDH that probably affected by the small increase of the LDH interlayer space during the intercalation.

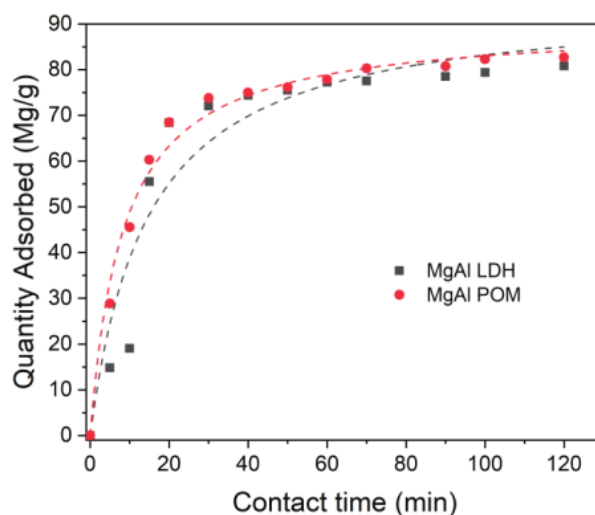


FIGURE 3. Effect of contact time to the adsorption performance of MgAl LDH and MgAl POM

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CONCLUSION

The MgAl LDH and α -PW₁₂O₄₀ intercalated MgAl LDH (MgAl POM) have been successfully synthesized by facile coprecipitation and ion exchange method. The materials characterization revealed that after intercalated by α -PW₁₂O₄₀ anion, the interlayer space of MgAl LDH increased from 7.72 Å to 9.95 Å that indicated incorporation of PW₁₂O₄₀ anion into the interlayer space of MgAl LDH.

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