Environmental application of engineering magnesite slag for phosphate adsorption from wastewater

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Abstract
Herein, magnesite slags (MS), which remain after sulfuric acid extraction from light burnt magnesite in the magnesite industry, were used as phosphate adsorbents in wastewater. The MS were calcined under 700 °C to enhance phosphate adsorption. The calcined magnesite slags (CMS) were characterized by nitrogen adsorption–desorption isotherm, X-ray diffraction, and scanning electron microscopy. A series of batch adsorption experiments were carried out to test the phosphate adsorption capacity of CMS. The results showed that the calcific treatment promoted the conversion from Mg, Ca, Fe, etc. compound to metal oxide of the MS. The generated metal oxide particles resulted in 237.4 mg/g increase in the phosphate adsorption capacity. The phosphate adsorption isotherm of CMS fitted the Langmuir model better, and the maximum adsorption capacity of CMS was 526 mg/g. The adsorption kinetics of phosphate on CMS can be described by the pseudo-second-order model. The phosphate removal efficiency was greater than 98% in 300 mg/L phosphate solution. Mechanism investigation results indicated that phosphate was adsorbed by CMS through MgO protonation, electrostatic attraction, Mg-P complexation, and ligand exchange. The results obtained in this work demonstrate that the CMS is a potential effective adsorbent for removal and reutilization phosphate from P-contaminated water, due to it can be employed as a fertilizer after phosphate adsorption.

Keywords Phosphate removal · Magnesite slags · Adsorption · Isotherm · Kinetics

Introduction
Phosphorus is an essential macronutrient for the growth of plant. However, agriculture and industry activities have produced excessive phosphorus; the releasing of phosphorus into surface water such as rivers and lakes may lead to environmental eutrophication which is threatening the ecological balance and human body health (Conley et al. 2009; Huisman et al. 2018; Morelli et al. 2018; Resiere et al. 2018). Phosphorus is a nonrenewable resource on earth; the reserves phosphorus ores on earth are proved to be exhausted without intervention measures (Tang et al. 2017). In addition, phosphorus is an important element for global food security with a highly dissipative (Scholz and Wellmer 2013). It is imperative to develop an effective technology to remove and reutilize phosphorus from P-contaminated water (Li et al. 2018; Zhang et al. 2021c).

Tremendous methods have been developed to remove phosphate such as chemical precipitation, biological treatment, membrane separation, and adsorption. Previous studies have revealed that phosphorus adsorption is an effective technology to capture and reutilize phosphorus from wastewater (Li et al. 2016a). Several mineral slags such as dolomite, bentonite, calcite, and sepiolite waste minerals have been developed as phosphorus adsorbents from wastewater, due to their stability and richness in macronutrient for plant (Mg, Fe, Ca, Zr, etc.). Besides, these metal elements exhibited good affinity to the phosphate via electrostatic interaction, surface complexation, or proton exchange to form P-O-Metal bond (Shakoor et al. 2021; Yin et al. 2021; Zhang et al. 2021b). Furthermore, the mineral adsorbent has a potential application as a slow-release fertilizer after
phosphate adsorption (Li et al. 2020, 2016b; Tang et al. 2018; Yao et al. 2013).

The magnesite slag is a by-product generated in the acid-treating process of light burnt magnesite. In 2020, around 3 million tons of MgSO4·H2O, MgSO4·7H2O, and other magnesium salt had been produced from magnesite in China, whereas the by-production of MS was around 50,000 t which cannot be completely consumed. The main composition of MS is magnesium carbonate, basic magnesium sulfate, silica, calcium carbonate, ferric oxide, aluminum oxide, etc. Many researchers have develop several mineral slag (blast furnace slag (Guo et al. 2017), ferromanganese slag (Jain and Maiti 2021), electric arc furnace slag (Liu et al. 2020), ferrous slag and carbon steel slag (Zheng et al. 2020)) as efficient adsorbent based on its physical and chemical properties. However, there is some lack of exploitation about efficient adsorbent of MS.

In this study, MS was activated by calcination process. The aim of this work was to develop a phosphate adsorbent from MS for the recovery of phosphate from aqueous solution. Specially, effects of solution pH, adsorption isotherm, and kinetics were investigated with batch experiments; subsequently the efficiency of phosphate-contained adsorbent as a fertilizer were examined. At last, the adsorption mechanism of as-prepared CMS was investigated.

Materials and methods

Materials

Magnesite slags (MS) used in the experiments are a magnesite waste generated in the H2SO4-treating process of light burnt magnesite obtained from Yingkou Magnesite Chemical Ind Group Co., Ltd., China. The MS was washed by purified water three times to minimize the presence of solvent, then dried, smashed, and sieved; the MS powder with a particle size between 80 and 100 mesh was collected for use. Monopotassium phosphate (KH2PO4), hydrochloric acid (HCl), sodium hydroxide (NaOH), and other analytical chemical reagents of analytical grade were purchased from Aladdin Industrial Corporation. Purified water was employed to prepare aqueous solutions.

Calcined magnesite slag preparation

The raw MS were transferred into a muffle furnace and calcined at 10 °C/min up to 700 °C for 2 h. After cooling down to the room temperature, the calcined magnesite slags (CMS) were sealed in a desiccator for further experiment tests. To investigate the effect of calcination temperature on the transformation of MS, calcined magnesite slags (CMS) prepared at 600, 700, and 800 °C were expressed as CMS 600, CMS 700, and CMS 800, respectively. As a contrast, magnesite slags (80–100 mesh) without calcination were tested.

Calcined magnesite slag characterization

The main elemental composition of the MS determined by X-ray fluorescence (XRF, S4 Pioneer, Bruker AXS, Germany) were summarized in Table 1; the raw MS is mainly consisted of Mg, Si, S, Ca, Fe, Al, and other elements. The surface morphology and elements of the CMS were analyzed by scanning electron microscopy (SEM) and energy dispersive spectrometer (EDS) (FESEM-EDS, S-4800, Hitachi, Japan). The crystallographic structure of CMS was characterized by X-ray diffractometry (XRD, D8 Advance Sol-X, Bruker Co., USA); the samples were scanned in a 2 theta 5–85°. The BET-specific surface area, total pore volume, and average pore diameter of the MS and CMS were characterized by nitrogen adsorption–desorption isotherms at 77 K on automated adsorption equipment (BSP-PM2, Beishide Instrument, China). To investigate adsorption mechanism, the CMS before and after loading phosphate was analyzed by X-ray photoelectron spectroscopy (XPS, K-Alpha, Thermo Scientific, USA) and Fourier transform infrared spectroscopy (FT-IR, IN10, Thermo Scientific, USA).

Phosphate adsorption experiments

Batch adsorption experiments were performed to obtain the phosphate adsorption behavior of CMS. Firstly, phosphate stock solutions were prepared by dissolving a certain amount of KH2PO4 in deionized water to get an initial concentration of 1000 mg L−1. For adsorption isotherm experiment 0.1 g, CMS were accurately weighed and placed in a stoppered 250-mL conical flask, and 100 mL of different initial phosphate concentrations (50, 100, 150, 200, 250, 300, 350, 400, 450, and 500 mg L−1, respectively) were mixed in the conical flask. Then the mixtures were shaken at 180 rpm in a 25 °C air bath for 24 h. After adsorption, the supernatant was separated by filtering through a 0.45 μm microfiltration membrane for phosphate concentration analysis. Phosphate concentrations in solutions before and after adsorption were analyzed by the molybdenum blue method with a UV–Vis spectrophotometer (UV1900, Shimadzu, Japan). The absorbance was determined at a wavelength of 880 nm and according to absorbance of the samples that calculate the equilibrium concentration of phosphate. Equilibrium phosphate adsorption capacities qe (mg-P g−1) were calculated by the mass balance as expressed in Eq. (1).

| Table 1 Composition of the MS (mass %) |
|--------------------------|-------|-----|-----|-----|-----|
| Mg | Si | S | Ca | Fe | Al |
| 13.177 | 6.234 | 5.192 | 4.445 | 1.391 | 1.021 |
where \( C_0 \) is the initial phosphate concentration (mg L\(^{-1}\)), \( C_e \) is the equilibrium phosphate concentration (mg L\(^{-1}\)), \( V \) is the volume of phosphate solution (L), and \( m \) is the adsorbent mass (g).

The adsorbed amount of phosphate was analyzed with two isotherm equations: Langmuir and Freundlich models, which expressed as follows:

**Langmuir.** \( q_e = \frac{V (C_0 - C_e)}{m} \) (1)

**Freundlich.** \( q_e = \frac{K_F C_e^{1/n}}{1 + K_F C_e} \) (3)

where \( K_L \) and \( K_F \) are the constants of Langmuir and Freundlich, respectively. \( q_m \) is the maximum adsorption capacity of the adsorbent, and \( n \) is the heterogeneity factor.

Kinetics of phosphate adsorption on CMS were determined by performing batch adsorption experiments. Mixing a series of 0.5 g CMS with 100 mL phosphate solutions (initial pH = 9, 300 mg L\(^{-1}\) phosphate diluted from phosphate stock solution). The mixtures were shaken at 25 °C, 180 rpm, filtered at selected time intervals. The phosphate concentration of the filtrates was analyzed as described above. Pseudo-first-order and pseudo-second-order models were used to illustrate the adsorption mechanism of CMS.

**Pseudo – first – order.** \( q_t = q_e(1 - \exp(-K_{p1}t)) \) (4)

**Pseudo – second – order.** \( q_t = \frac{K_{p2}q_e^2}{1 + k_{p2}q_et} \) (5)

where \( K_{p1} \) and \( K_{p2} \) are the pseudo-first-order and pseudo-second-order rate constants, respectively. \( q_e \) and \( q_t \) are the phosphate adsorption amount of the adsorbent at equilibrium and time \( t \), respectively.

**Results and discussion**

**Impact of calcination temperature on phosphate adsorption capacity**

Calcination activation has many advantages such as simple operation, low cost, high efficacy, and limited secondary pollution. To get the best calcination temperature, the pristine MS was calcined at different temperatures (600 °C, 700 °C, and 800 °C), and CMS 600, CMS 700, and CMS 800 were compared based on morphology and phosphate adsorption capacity (Fig. 1). As shown in Fig. 1a–c, with an increase in the calcination temperature from 600 to 700 °C, the SEM figures indicate a visible increase of nanoparticles formation on the surface of the CMS after calcination. However, the increment of calcination temperature from 700 to 800 °C caused a slightly aggregation of the particles. Similarly, it is evident that there was significant difference in the phosphate adsorption capacity of MS, CMS 600, CMS 700, and CMS 800; the phosphate adsorption capacities of MS, CMS 600, CMS 700, and CMS 800 were 59.5 mg g\(^{-1}\), 215.4 mg g\(^{-1}\), and 59.5 mg g\(^{-1}\), respectively.
296.9 mg g⁻¹, and 267.2 mg g⁻¹, respectively (Fig. 1d). The calcined magnesite slag exhibited much higher adsorption capacity than the pristine MS. Based on the TG and XRD results of CMS, the high-temperature calcination process could in situ generate metal oxide particles on the magnesite slag surfaces, which can significantly increase the affinity to phosphate in solution. Thus, the calcination treatment can effectively enhance the phosphate adsorption activity and capacity of magnesite slag. Among three investigated calcination temperatures, CMS-700 exhibited best phosphate adsorption capacity. Thus, CMS-700 was selected for further investigation.

Physical and chemical properties of CMS

The XRD spectra of MS samples (Fig. 2) demonstrated the presence of MgCO₃ (JCPDS card No. 08–0479), Mg₅(OH)₆SO₄·8H₂O (No. 53–1039), SiO₂ (No. 75–0443), etc.; the XRD analysis revealed that the composition of MS was consistent with that result of XRF. After calcination, the XRD spectra of CMS peaks indicated the formation of MgO particles from MgCO₃ and Mg₅(OH)₆SO₄·8H₂O during pyrolysis; SiO₂ (No. 75–0443) was also observed which remains from the raw magnesite waste. The highly dispersed MgO particles on CMS were ascribed to the dehydrogenation and decomposition of MgCO₃ and Mg₅(OH)₆SO₄·8H₂O compounds (Choudhary et al. 1992), which could provide efficient adsorption site for phosphate in water (Jiang et al. 2019; Luo et al. 2021).

Understanding the calcination processes of MS was crucial for insight into the active site formation of material. The calcination behavior of CMS generated from MS was analyzed by TG-DTG (5 °C/min to 800 °C). As depicted in Fig. 3, the turning temperature points indicated different stages of MS decomposition; the first step ranged from room temperature to 120 °C was ascribed to the loss of water and volatiles from the MS (Luo et al. 2021). The second step varied from 120 to 318 °C was the dissociation of crystal water. The third stage within the range of 318 to 451 °C was attributed to the decomposition of hydroxyl (Wang et al. 2020). The fourth stage ranged from 451 to 681 °C was the conversion of carbonate into CO₂. The decomposition of MS during the thermal treatment is shown in the following reaction.
The morphology and microstructure of the MS and CMS were processed using SEM and EDS (Fig. 4). The SEM proved that the morphology between MS and CMS was significantly different. As shown in Fig. 4a, the SEM image of MS showed irregular plates with a smooth surface, as well as sharp edges and corners. Figure 4b shows the in situ generated homogeneous nanometal oxide particles on the surface of CMS; the obtained nanoparticles provided more adsorption sites and improve phosphate adsorption capacity; the phases of MS and CMS were further confirmed by EDS analyses.

The elemental contents of pristine MS and CMS were analyzed by EDS (Fig. S1c, d), and the EDS analysis showed the amounts of Mg, O, Ca, Si, Al, and Fe elements in the composites. As would be expected, the O% in the CMS decreased, and the Mg, Si, Ca, and Al% increased after calcination.

It is well known that Mg, Ca, Fe, and Al have a high chemical affinity to phosphate. The MgO, as one of main components of CMS, can easily react with phosphate to form Mg-PO₄ (Mg₃(PO₄)₂, Mg(H₂PO₄)₂, MgHPO₄) composition via precipitation reaction (Liu et al. 2019). The CaO, Fe₂O₃, and Al₂O₃ can react with phosphate ions to form Fe–O–P and Al–O–P complexes by surface complexation (Liu et al. 2019; Peng et al. 2019). Therefore, the CMS used in this work contains MgO, CaO, and Fe₂O₃ which was promising for phosphate removal from wastewater.

Table S1 lists the porosity-related characteristics (e.g., BET surface area, pore volume, and average pore diameter) of MS and CMS. The MS and CMS were classified as type II isotherms (Fig. S2) with distinct hysteresis loop according to the International Union of Pure and Applied Chemistry classification. Compared to raw MS (specific surface area 4.69 m² g⁻¹, total pore volume 0.0315 cm³ g⁻¹), CMS featured triflingly increased textural properties (specific surface area of 4.73 m² g⁻¹ and total pore volume of 0.0356 cm³ g⁻¹). The specific surface area, total pore volume, and average pore diameter of the MS and CMS were approximately similar. The BET data suggested that the in-situ generated metal oxide particles did not impact the specific surface area of CMS.

**Impact of pH on phosphate adsorption capacity**

The solution pH strongly influenced the adsorption capacity of adsorbent in adsorption process (Zheng et al. 2020). In this work, the effects of pH on phosphate adsorption capacity of CMS were determined by adsorption isotherms; the obtained equilibrium data were fitted by using Langmuir (Eq. 2) and Freundlich (Eq. 3) isotherm models (Fig. 5), and the adsorption isotherm parameters were presented in Table S2, as compared to the regression coefficient ($R^2$); the Langmuir adsorption model showed more satisfactory fitting to the phosphate adsorption isotherms on CMS, which indicated that phosphate adsorbed on CMS is primarily monolayer adsorption. With the increase of pH from 5 to 11, the phosphate adsorption capacity increased and then slight decreased. When the pH value was 9, the highest phosphate capacity for CMS was 526 mg/g; similar results had been reported by other studies (Liu et al. 2020; Luo et al. 2021; Xu et al. 2018; Yang et al. 2014). Thus, CMS had a wide range of pH values from 5 to 11; alkaline wastewater was more favorable for the phosphate adsorption process. Some studies reported that the main existent of phosphate was PO₄³⁻ and HPO₄²⁻ ions at pH 9 value. The MgO and other metal oxide can generate electrostatic attraction with PO₄³⁻ and HPO₄²⁻ at alkaline environment (Deng et al. 2020).
Impact of initial concentration on phosphate adsorption capacity

The effect of initial concentration on phosphate adsorption of CMS was presented in Fig. 6. The results indicated that the adsorption capacity was affected by phosphate concentration. As the initial phosphate concentration ranging from 50 to 550 mg L\(^{-1}\), the phosphate adsorption capacity on CMS increased significantly. Test with initial phosphate concentration of 50 mg/L showed 99% removal efficiency on CMS. However, the phosphate removal efficiencies were still greater than 90% when the solution concentration was 500 mg L\(^{-1}\). The decrease of phosphate removal efficiency combined with the increase of adsorption capacity with increasing initial phosphate concentrations has been reported (Blanco et al. 2016; Gan et al. 2015).

Impact of adsorption time on phosphate adsorption capacity

The changes in phosphate removal efficiency and adsorption amount with different adsorption time were presented in Fig. 7a and b. The phosphate removal efficiency and adsorption amount increased with time and reached equilibrium after 25 min approximately. Nearly 64% and 90% of phosphate was removed within 5 min and 15 min, respectively. The CMS reached the maximum adsorption efficiency of 98.3% after 55 min. To better understand the adsorption mechanism and kinetics of CMS, kinetics modeling of adsorption helps to characterize the adsorption process. The pseudo-first-order (Eq. 4) and pseudo-second-order (Eq. 5) rate equations were used to model phosphate adsorption on CMS. The fitting results of the adsorption experimental data were represented in Fig. 7b and Table S3. The pseudo-second-order better described the adsorption process, which was consistent with other reported works (Cui et al. 2020;
Zhang et al. 2021a). The adsorption of phosphate on CMS was dominated by chemical interaction with adsorbate.

### Possible phosphate adsorption mechanism

To further understand the possible mechanism of phosphate adsorption process, XRD, FT-IR, and XPS were applied to investigate the internal connection between CMS and CMS-P. As shown in Fig. 8a, the XRD pattern of CMS-P shows main phase of SiO$_2$ and MgHPO$_4$(H$_2$O)$_3$. This revealed the phase of MgO in CMS transformed to MgHPO$_4$(H$_2$O)$_3$ phase during phosphate adsorption process, while the SiO$_2$ phase still remained. The diffraction peaks of MgO significantly reduced after the phosphate adsorption, suggesting that MgO particles might primarily manifest the prominent contributions in the adsorption.

The changes of functional groups before and after adsorption was confirmed by the FT-IR characterization (Fig. 8b). The hydroxyl-OH vibration peaks (~3440 cm$^{-1}$, 1627 cm$^{-1}$) were assigned to the absorbed water in materials. The peak
at 1022 cm\(^{-1}\) corresponds to stretching vibration of Si–O-Si bond in SiO\(_2\) (Yang et al. 2021). The major differences between spectra of CMS and CMS-P are summarized as follows: (1) Peaks in CMS-P (~1434 cm\(^{-1}\) and 3689 cm\(^{-1}\)) significantly reduced compared with CMS. (2) New peak (~1064 cm\(^{-1}\)) appeared in CMS-P. After adsorption, the peak located at 1064 cm\(^{-1}\) was contributed to stretching of P-O bond in HPO\(_4^{2-}\) (Zhang et al. 2019, 2021b), which indicated the phosphate was successfully adsorbed by the CMS. The wavelength at 3689 cm\(^{-1}\) and 1434 cm\(^{-1}\) of CMS corresponded with Mg-O of MgO, while after adsorption these peaks reduced, we suggested that the MgO particles in CMS involved in the adsorption of phosphate, which transformed to Mg-P complexes after adsorption. This idea is consistent with the XRD result and other works (Park et al. 2017; Xia et al. 2016a).

XPS was employed to analyze phosphate adsorption process. Figure 8c and d showed the XPS spectra of CMS and CMS-P. As shown in Fig. 8c, after adsorption of phosphate, a new peak for P 2p (134.4 eV) appeared in the XPS spectra of CMS-P, which indicated the presence of Mg-P compounds in the CMS-P. The P2p spectra was separated into two species (Fig. 8d) located at 134.8 and 133 eV, which attributed to the presence of HPO\(_4^{2-}\) and PO\(_4^{3-}\), respectively (Xia et al. 2016b). This phenomenon indicated the successful nutrients uptake of CMS.

Based on the above analysis, the adsorption mechanism of phosphate by the CMS was illustrated in Fig. 9; the adsorption mechanism of CMS mainly included the following. (1) In MgO protonation, the MgO on CMS were protonated to MgOH\(^+\); the positively charged MgOH\(^+\) had strong affinity to phosphate anions in aqueous solution (Liu et al. 2021). (2) In electrostatic attraction, as shown in Fig. 9, phosphate ions was captured by MgOH\(^+\) through electrostatic attraction (Xiao et al. 2020). (3) In Mg-P complexation formation, the previous investigation demonstrated that the MOH\(^+\) groups (i.e., Mg, Ca, Fe) can form the complexes with phosphate ions. As the reaction continued, MgOH\(^+\)–HPO\(_4^{2-}\) was formed by complexation. (4) For ligand exchange, the FT-IR analysis observed the decrease of Mg-O on the surface of CMS. The OH groups of MgOH\(^+\) were replaced by HPO\(_4^{2-}\) during ligand exchange. This coordination process may be the key for phosphate adsorption mechanism (Liu et al. 2021). In summary, the mechanism of phosphate adsorption on CMS can be classified into four aspects, MgO protonation, electrostatic attraction, Mg-P complexation, and ligand exchange.

**Plant growth as a response to P fertilizer applications**

To assess the possible utilization of CMS-P as a fertilizer, the CMS-loaded phosphate (CMS-P) was employed as a phosphate-contained fertilizer for agriculture. 0.5 g/kg and 1.0 g/kg of CMS-P was applied to the pot for the treatment; garlic growth was measured after 15 days. As represented in Fig. 10, the average growth height of garlic seedling above-ground parts in the CMS-P (CMS-P-0.5 (42 cm), CMS-P-1 (44 cm)) was higher than those in the control (30 cm). These
results clearly demonstrated that the CMS-P has the potential application of substitute for phosphate fertilizers.

**Comparison with other engineering slags/wastes adsorbents**

To date, many engineering mineral slags/wastes have been developed for phosphate adsorption with favorable features. We summarized some similar investigations for comparison with this work; results are represented in Table 2. From the data in the table, we can find that many engineering slag/waste adsorbents were developed complicated or with lower phosphate adsorption capacity. In contrast, the CMS developed in this work is relatively efficient and accessible. Therefore, the CMS is an attractive adsorbent for treating phosphate-rich wastewater.

**Conclusions**

In this work, magnesite slags from the engineering waste of acid extraction of light burnt magnesite were used to prepare phosphate adsorbent by simply calcination process. The calcination process promoted the decomposition of metal compounds impurity and bring active adsorption sites for phosphate. CMS had a wide range of pH values from 5 to 11; the adsorption capacity of CMS increased with pH value; alkaline wastewater was more favorable for the phosphate adsorption process. Equilibrium adsorption isotherm at pH = 9 was fitted by Langmuir; the highest phosphate capacity for CMS was 526 mg/g. The adsorption process conformed to the pseudo-second-order kinetics better. Phosphate was adsorbed by CMS through MgO protonation, electrostatic attraction, Mg-P complexation, and ligand exchange. The solid waste after adsorption proved to be a well fertilizer for plant. This investigate demonstrated that the calcined magnesite slag possessed noteworthy potential in phosphate recovery application.
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