



## Preparation and properties of a layered double hydroxide deicing additive for asphalt mixture



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### ABSTRACT

Mg–Al Cl<sup>−</sup> layered double hydroxide (LDH) was prepared from Mg–Al CO<sub>3</sub><sup>2−</sup> LDH by the calcination recovery method. Energy-dispersive spectroscopy spectra show that Cl<sup>−</sup> anions replace CO<sub>3</sub><sup>2−</sup> anions in the LDH structure. X-ray diffraction patterns and scanning electron microscopy images show that the intercalation spacing and platelet thickness of Mg–Al Cl<sup>−</sup> LDH are larger than those of Mg–Al CO<sub>3</sub><sup>2−</sup> LDH. This increase in the structural dimensions occurs partially because a larger quantity of monovalent Cl<sup>−</sup> anions than divalent CO<sub>3</sub><sup>2−</sup> anions is required to balance the positive charges of the LDH metal cations. The results of melting ice point (MIP) tests indicate that Mg–Al Cl<sup>−</sup> LDH is more effective than Mg–Al CO<sub>3</sub><sup>2−</sup> LDH in decreasing the MIP of water. Freezing points (FPs) of asphalt mixtures show that Mg–Al Cl<sup>−</sup> LDH can reduce FPs. At temperatures below the FPs of asphalt mixtures containing Mg–Al Cl<sup>−</sup> LDH, the adhesion forces between ice and asphalt mixtures show that Mg–Al Cl<sup>−</sup> LDH can facilitate ice removal. Immersion tests were conducted to investigate the effect of rain on the melting ice properties of asphalt mixtures. FPs and adhesion force for Mg–Al Cl<sup>−</sup> LDH do not change after immersion, indicating that rain should not affect the properties of melting ice of Mg–Al Cl<sup>−</sup> LDH. Conductivities of immersion liquids show that Mg–Al Cl<sup>−</sup> LDH enables Cl<sup>−</sup> anions to be retained in asphalt mixtures without adversely affecting the surrounding environment and pavement.

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### 1. Introduction

Ice forms easily on the pavement surfaces in cold regions. Ice formation reduces friction coefficient between pavement and tires, frequently resulting in car accidents and subsequent traffic congestion. Chlorine salt is commonly sprayed to eliminate this hazard. However, the chloride anions can potentially pollute the surrounding water, soil and vegetation (Fay and Shi, 2012). Moreover, if it is used on the bridge deck, the steel construction would be corroded seriously (Shi et al., 2009). In addition, chloride anions would weaken the bonding between bitumen and aggregate (Feng et al., 2010), thereby affecting the service life of asphalt pavement (Özgan et al., 2013; Starck and Löfgren, 2007).

To reduce the deleterious effects of spreading chlorine salt on environment and asphalt pavement, a method of integrating chloride deicing additive (Cl<sup>−</sup> DIA) into asphalt mixtures has been developed (Goh et al., 2011; Jones, 1981; Ninomiya et al., 1993; Sheftick, 1991; Szatkowski and Swanson, 1986). When the road surface undergoes

compression, vibration and wear, the chloride component slowly migrates from the inner asphalt layers to the surface layers, thereby preventing or delaying the icing process on the road surface (Giuliani et al., 2012). However, Cl<sup>−</sup> anions are gradually released from the asphalt pavement and dissolve in water, which has a harmful effect on the surroundings and the pavement. Therefore, it is essential to develop a new deicing additive which can prevent the release of Cl<sup>−</sup> anions from the asphalt pavement.

A layered double hydroxide (LDH) consists of hydrotalcite-like compounds, which are composed of the positively charged layers and negatively charged anions (Cavani et al., 1991). The general formula of an LDH is [M<sub>1</sub><sup>2+</sup><sub>1-x</sub>M<sub>x</sub><sup>3+</sup>(OH)<sub>2</sub>]<sup>x+</sup>A<sub>x/n</sub><sup>n-</sup>, where M<sup>2+</sup> is a divalent metal cation, M<sup>3+</sup> is a trivalent metal cation, and A<sup>n-</sup> is an interlayer anion. In our preliminary experiments, Mg<sub>2</sub>Al<sub>4</sub>(OH)<sub>12</sub>CO<sub>3</sub> (which is abbreviated as Mg–Al CO<sub>3</sub><sup>2−</sup> LDH) was found to lower the freezing point of water and can therefore be used to prevent freezing. However, Mg–Al CO<sub>3</sub><sup>2−</sup> LDH has a limited ability to reduce the freezing temperature of water.

In this paper, Mg–Al Cl<sup>−</sup> LDH was prepared from Mg–Al CO<sub>3</sub><sup>2−</sup> LDH by calcination recovery. The effects of Mg–Al Cl<sup>−</sup> LDH and Mg–Al CO<sub>3</sub><sup>2−</sup> LDH on the melting ice point (MIP) of water were investigated. Ice-melting properties of asphalt mixtures with Mg–Al Cl<sup>−</sup> LDH were evaluated.

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## 2. Experimental and methods

### 2.1. Materials

The Mg-Al  $\text{CO}_3^{2-}$  LDH was provided by Jiangyin Ruilaw Chemical Co. LTD, Jiangsu, China, and has the structure formula  $\text{Mg}_2\text{Al}_4(\text{OH})_{12}\text{CO}_3$ . A commercial chloride-based deicing additive ( $\text{Cl}^-$  DIA), which has calcium chloride as its primary component, was obtained from Verglimit Anti-icing Road Surface Co. LTD, Swiss. Binder consisting of SBS modified bitumen was produced by China Best Modified Bitumen Co. LTD, Hubei, China, and had a penetration of 48 dmm at 25 °C, a ductility of 30 cm at 5 °C, a softening point of 81 °C, and a dynamic viscosity of 2.4 Pa·s at 135 °C. Basalt and mineral powder (machine-glazed limestone) were obtained from Jingzhu, Hubei, China.

### 2.2. Preparation of Mg-Al $\text{Cl}^-$ LDH

Mg-Al  $\text{Cl}^-$  LDH was prepared from Mg-Al  $\text{CO}_3^{2-}$  LDH by calcination recovery. The preparation process is shown in Fig. 1. First, 200 g of Mg-Al  $\text{CO}_3^{2-}$  LDH was calcined at 500 °C for 2 h in a muffle furnace to remove  $\text{CO}_3^{2-}$  anions (Cai et al., 2012). Second, a cooling process was performed in a closed storage pan, from which the air had to be continuously evacuated using a vacuum pump to prevent the accumulation of carbon dioxide. Third, the calcined product and 500 mL of NaCl solution (at a concentration of 20 wt %) were mixed in a glass flask by stirring for 30 min. Fourth, the mixture was filtered and the residue was washed repeatedly with deionized water until the final filtrate contained no chlorine anions, which was verified by  $\text{AgNO}_3$  solution. Finally, the residue was dried at 105 °C for 24 h and ground to obtain Mg-Al  $\text{Cl}^-$  LDH.

### 2.3. Characterization of LDHs

#### 2.3.1. X-ray diffraction

X-ray diffraction graphs of the Mg-Al  $\text{CO}_3^{2-}$  LDH and the Mg-Al  $\text{Cl}^-$  LDH were obtained using an X-ray diffractometer (D/MX-III A, Rigaku, Japan) with Cu K $\alpha$  radiation ( $\lambda = 0.15406$  nm; 40 kV, 50 mA). The data were collected in step-scan mode at a scanning rate of 0.02°/s. The diffractograms were scanned from 5° to 20°.

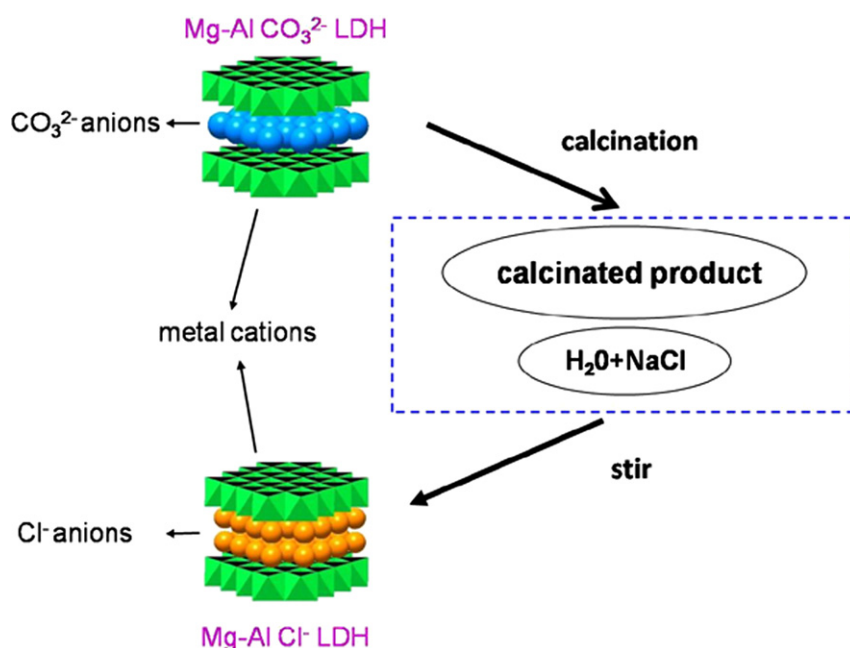


Fig. 1. Preparation of Mg-Al  $\text{Cl}^-$  LDH.

#### 2.3.2. Scanning electron microscopy

The Mg-Al  $\text{CO}_3^{2-}$  LDH and the Mg-Al  $\text{Cl}^-$  LDH powder were attached to stubs with a carbon-type double-sided adhesive. To enhance their electrical conductivity, the surfaces of the samples were sputtered with a thin film of platinum using a sputter coating system (Baltec MED020, Vaduz, Liechtenstein). The sample morphologies were investigated by scanning electron microscopy (Quanta 250 FEG, FEI, Oregon, USA) and an elemental analysis was performed using energy-dispersive spectroscopy (JEOL-EDS System, Oxford Instruments, Tokyo, Japan).

#### 2.3.3. Melting ice point tests of LDHs slurries

Melting ice point (MIP) is more constant than freezing point due to the supercooling effect (Kozłowski, 2009). Different amounts of Mg-Al  $\text{CO}_3^{2-}$  LDH or Mg-Al  $\text{Cl}^-$  LDH were added to distilled water. The slurries were placed in a refrigerator at  $-20$  °C until they were completely transformed to ice phase. The slurries were then left at room temperature. When the slurries melted into ice and water mixture, their melting temperatures were measured with a thermometer whose precision is 0.1 °C. The MIP of LDH slurry was obtained by three parallel tests.

### 2.4. Preparation of asphalt mixture containing deicing additives

#### 2.4.1. Gradation composition of asphalt mixture

An AC-16 asphalt mixture is commonly used in the surface layer of asphalt pavement. According to the standard JTG F40-2004 (Technical Specification for Construction of Highway Asphalt Pavements, the Ministry of Transport of the People's Republic of China), designed gradation of control mixture containing 6 wt% mineral powder is shown in Fig. 2 and composition of different aggregates is shown in Table 1.

The optimum bitumen content was determined to be 4.8 wt% from the Marshall test. The particle size distributions of the deicing additives (Mg-Al  $\text{Cl}^-$  LDH and  $\text{Cl}^-$  DIA), and the mineral powder were given in Table 2. The asphalt mixture was prepared by replacing the mineral powder with equal weights of the two deicing additives because the particle sizes of Mg-Al  $\text{Cl}^-$  LDH and  $\text{Cl}^-$  DIA were similar to that of mineral powder. In this experiment, the mixtures of 3 wt% deicing additive/3 wt% mineral powder, 4 wt% deicing additive/2 wt% mineral powder, 5 wt% deicing additive/1 wt% mineral powder, and 6 wt% deicing additive were added into asphalt mixture respectively.

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