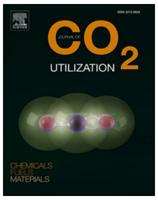
Journal of CO2 Utilization 88 (2024) 102953

Contents lists available at ScienceDirect

Journal of CO2 Utilization

journal homepage: www.elsevier.com/locate/jcou



Strength and leaching behavior of CaO- and MgO-treated Cd-contaminated soils subjected to partial and full carbonation

Wentao Li [a,b,c], Junde Qin [b], Yaolin Yi [b,d,*]

[a] School of Civil Engineering, Architecture and Environment, Hubei University of Technology, 430068, China
[b] School of Civil and Environmental Engineering, Nanyang Technological University, 639798, Singapore
[c] Innovation Demonstration Base of Ecological Environment Geotechnical and Ecological Restoration of Rivers and Lakes, Hubei University of Technology, 430068, China
[d] China-Singapore International Joint Research Institute, Guangzhou, 510530, China

ARTICLE INFO

Keywords:
Contaminated soil
Cadmium
Carbonation
CaO
MgO

ABSTRACT

Cadmium (Cd)-contaminated soils may pose a significant threat on human health. They are often treated with quick lime (CaO) and reactive magnesia (MgO), but these treatments often result in low strength. Hence, in this study, partial and full carbonation are used to enhance the stabilization/solidification of CaO- and MgO-treated Cd-contaminated soils, aiming to achieve CO_2 sequestration, strength improvement, and Cd immobilization. Performance of treated contaminated soils is evaluated through unconfined compressive strength (UCS), leaching, X-ray diffraction (XRD), and thermogravimetric analysis (TGA) tests. The results indicate that carbonation significantly enhances the UCS of both CaO- and MgO-treated Cd-contaminated soils. After carbonation, MgO-treated soils exhibit higher UCS than CaO-treated soils. Partial and full carbonation yield similar UCS in CaO-treated soils, while full carbonation results in higher UCS in MgO-treated soils. For CaO-treated soils, partial carbonation keeps Cd leachability below the 1 mg/kg limit, but full carbonation increases it beyond this limit. In contrast, fully carbonated MgO-treated soils maintain Cd leachability below the limit, though partial carbonation leads to higher leachability. Formation of Ca and Mg carbonates contributes to the strength improvement of soils. $Cd(OH)_2$ and its complex, as well as $CdCO_3$ exist in partially and fully carbonated soils, lowering leached Cd concentration. Overall, partial carbonation is better for CaO-treated soils, while full carbonation is preferable for MgO-treated soils.

1. Introduction

The urbanization and industrialization has led to a widespread environmental challenge of soil contamination from heavy metals, such as unorganized stockpiling and uncontrollable discharge of these wastes [59]. Contaminated soils are difficult to degrade in the natural environment, and easy to release heavy metals into the water, leading to ecological and environmental deterioration. Cadmium (Cd), a poisonous heavy metal, is commonly found in contaminated soils due to the utilization of agricultural inputs, such as fertilizers, pesticides, and biosolids (such as sewage sludge) [2,3,4,48,60,54]. Exposure to excessive Cd will result in the development of painful osteomalacia (a bone disease) and dysfunction of kidneys [53].

The utilization of binders in the stabilization/solidification (S/S) process is an effective method for treating soils contaminated with heavy metals [25]. One of the frequently employed binders is lime (CaO and $Ca(OH)_2$) [10,20,21,24,25,5]. Due to its alkaline nature, lime can react with the heavy metals, forming insoluble hydroxides and their complexes [25,43]. Particularly, the high pH condition (pH > 12) that lime produces, is suitable for the immobilization of Cd as the solubility of Cd hydroxide can reach the minimum value at this pH condition [23,25,40]. However, lime treatment cannot provide enough strength for Cd-contaminated soils [25], posing a limitation on the applicability of lime in the S/S of such contaminated soils. United States Environmental Protection Agency [47] requires that treated waste materials need to have a unconfined compressive strength (UCS) exceeding 350 kPa if such materials intend to be disposed in landfills. In Netherlands and France, a UCS value of 1000 kPa is suggested for the materials disposed in landfill [12].

Magnesia (MgO) has also attracted lots of attention in treating heavy metal-contaminated soils due to the good pH buffering capability [17,25,35,39,45,50]. Nevertheless, the Cd-contaminated soil is an exception

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* Corresponding author at: School of Civil and Environmental Engineering, Nanyang Technological University, 639798, Singapore.
E-mail address: yiyaolin@ntu.edu.sg (Y. Yi).

https://doi.org/10.1016/j.jcou.2024.102953
Received 22 May 2024; Received in revised form 28 September 2024; Accepted 3 October 2024

as the pH range that MgO provides is much lower than the optimal pH (~12) required to minimize the leachability of Cd [25]. Similar to lime treatment, MgO treatment also produces low strength for contaminated soils, which is only 10 %-20 % that of cement-treated contaminated soils [18,51]. Therefore, to better utilize lime and MgO to remediate Cd-contaminated soils, it is necessary to find a method to address the aforementioned problems of low strength and high Cd leachabilities existed in contaminated soils treated by lime and MgO.

One of potential ways is to introduce CO_2 into lime- and MgO-stabilized soils. The technique of MgO+CO_2 (i.e. carbonating MgO) was proposed to remediate Pb-, Zn-, Cd-contaminated soils in the previous studies [26,27]. The result showed that MgO+CO_2 significantly increased the strength of three contaminated soils, producing a similar or higher strength compared to cement for the soils attributed to various formed magnesium carbonates [7,8,32,36,46,55,56,57]. In addition, with the treatment of MgO+CO_2, the leachabilities of Pb, Zn, and Cd in the soils were decreased to different extents. The formation of Pb, Zn, and Cd carbonates contributed to the fixation of these heavy metals. Similarly, introducing CO_2 into lime treatment may have a positive effect on the strength and leaching property of contaminated soils. Haas and Ritter [15] reported that lime carbonation contributed to the strength increase due to the formation of calcium carbonate ($CaCO_3$) in soils stabilization, but no studies were found on the application of lime carbonation to treat contaminated soils. Lu et al. [31] demonstrated that the introduction of CO_2 into calcium-base binder can facilitate the immobilization of potentially toxic elements. It is worthy to investigate the effectiveness of lime+CO_2 (i.e. carbonating lime) in treating Cd-contaminated soil. Particularly, the production of lime and MgO releases enormous CO_2 (1.09 t CO_2 emission /t CaO and 1.1 t CO_2 emission /t MgO, respectively) [34,38]. Carbonating lime and MgO for the treatment of soils can sequester gaseous CO_2, which has a potential to become a carbon-neutral technique.

Nevertheless, the degree of carbonation significantly affects the performance of soils or cementitious materials involving lime and MgO. Yi et al. [56] pointed out that the strength of carbonated MgO-treated soils increased, and then generally reached a plateau with the carbonation degree. However, for some soils specimens, a slight decrease in strength was observed. Unluer and Al-Tabbaa [46] and Wang et al. [49] and found that higher carbonation degree led to higher compressive strength for MgO cement within 7 days carbonation. Although there were some investigation on the strength behavior of MgO-treated soils subjected to full and partial carbonation, the informaion on their leaching behavior is missing. Lime carbonation has been studied by many scholars, who focused on the CO_2 capture efficiencies of pure lime rather than the relationship between performance of lime-treated soils and carbonation degree [11,14,9]. Effect of carbonation degree on strength and leaching characteristics of lime-treated contaminated soils has not been reported in previous studies.

Hence, to better understand strength and leaching characteristics of lime- and MgO-treated contaminated soils with partial and full carbonation, the S/S efficiency of quick lime (CaO)+CO_2 and reactive MgO+CO_2 in treating Cd-contaminated soil is investigated in this study. The strength, leaching behavior, and mineralogical characteristics of the treated Cd-contaminated soils are also assessed. Although Cd usually comes with other metals, other metals might compete with Cd for the S/S efficiency of carbonation, and further affect performance of treated Cd-contaminated soils. Therefore, in this study, only Cd is considered.

2. Materials and methods

2.1. Materials

A synthetic soil was artificially created, comprising 90 % fine sand and 10 % kaolin by weight. Kaolin and fine sand were sourced from Buildmate Pte Ltd in Singapore and International Scientific (Pte) Ltd in Singapore, respectively. To enhance carbonation, 7.5 % of water content (defined by weight of the dry soil) was applied as per the methodology of Yi et al. [56]. Two binders, namely CaO and reactive MgO, were employed in treating Cd-contaminated soils. Fine powder CaO and reactive MgO were sourced from Sigma-Aldrich Pte Ltd, Singapore and Meishen Chemical Co. Ltd, Xingtai, China, respectively. The reactivity of MgO (46 seconds) was evaluated following the method described by Shand (2006). Table 1 shows chemical compositions for kaolin, MgO, and CaO obtained by X-ray fluorescence (XRF) testing.

The Cd-contaminated soil was prepared through incorporating cadmium chloride hydrate ($CdCl_2 \cdot H_2O$) into the soil. $CdCl_2 \cdot H_2O$, was used to formulate Cd solutions by being dissolved into deionized water to obtain Cd concentrations of 1000, 2000, 4000, 8000, and 16,000 mg/kg of (dry) soil weight. These concentrations were chosen to simulate a wide range of Cd concentrations in natural and/or contaminated soils.

2.2. Sample preparation

In this study, a binder content (5 % by weight of the dry soil) was employed to treat the contaminated soil. The remediation process involved several steps. Initially, either CaO or MgO was mixed with dry soil for 5 minutes to ensure uniformity, using a laboratory mixer operating at a rotation rate of 60 revolutions per minute. Subsequently, the $CdCl_2 \cdot H_2O$ solution was introduced and thoroughly mixed for an additional 10 minutes. Then, the resulting mixture was promptly placed in a steel mold with a size of Φ50 mm×100 mm, and compacted using a mini-compactor that was applied in prior studies [25,27]. Each test sample underwent compaction in three layers, with each layer receiving five blows. This compaction method allowed for the generation of a compaction energy of 582.51 kJ/m^3 per unit volume, consistent with the energy generated from the standard Proctor compaction test [1]. Finally, the molded specimen was hermetically sealed in plastic bags and cured at 25°C for 2 hours (for carbonation) or for 28 days curing (for conventional curing without carbonation).

2.3. Carbonation process

As depicted in Fig. S1 shown in the supplementary material, the carbonation was processed following the experimental setup described in Li and Yi [27]. The test specimen was placed inside the mold and covered with plastic caps. Both caps were equipped with a flow tap to enable gaseous CO_2 to travel through the specimen. During the carbonation test, the specimen was exposed to 50 kPa of CO_2 over a period of 7 days and the weight of the mold, specimen, and caps was always recorded. The carbonation test was performed three times for each condition. Degree of carbonation, denoted by CO_2 uptake/CaO (or MgO) by weight, is expressed as per Eq. 1.

$$CO_2\ uptake \Big/ CaO\ (or\ MgO) = \frac{M_t - M_0}{M_m} \tag{1}$$

Where, M_0 represents the initial weight before carbonation; M_t stands for the weight of the at carbonation time "t". $M_t - M_0$ signifies the weight of CO_2 uptake. M_m represents the weight of CaO (or MgO) added in the specimen.

2.4. Tests

UCS tests were conducted in triplicate on either carbonated or 28-day conventionally cured specimens. The loading rate was maintained at 1 mm/min until the peak strength was achieved during the test. Subsequently, the specimens used in the UCS test were crushed to prepare samples smaller than 4 mm in size, as stipulated by the one-stage batch leaching test requirements [6]. Then, these samples were mixed with distill water and used in the leaching test following the procedure depicted in [6]. After leaching, the pH of the soil-water mixture was swiftly measured by using a Mettler Toledo pH meter, and the leachate

Table 1
Chemical compositions of materials (% by weight).

Composition	CaO	SiO_2	Al_2O_3	Fe_2O_3	MgO	K_2O	SO_3	Others	Loss on ignition
Kaolin	0.06	64.70	30.17	ND	0.80	3.57	0.05	0.65	0
MgO	0.50	ND	ND	0.03	89.50	ND	0.60	1.72	7.65
CaO	93.50	ND	ND	ND	0.74	ND	1.76	0.50	3.50

ND: not detected.

was filtered with a 0.45 mm membrane syringe filter. The filtrate was then subjected to triplicate analysis to determine the Cd leachability. The other crushed samples underwent freeze-vacuum drying, then ground and passed through a 0.075 mm sieve. The obtaned powder samples were utilized in X-ray diffraction (XRD) and thermogravimetric analysis (TGA) to ascertain their mineralogical compositions.

3. Results and analysis

3.1. CO_2 uptake

Fig. 1(a) to (d) show the CO_2 uptake for CaO- and MgO-treated soils subjected to full and partial carbonation. Li et al. [26] has reported that the introduction of $CdCl_2$ did not considerably decerlarate the carbonation of MgO-treated soils, as shown in Fig. 1(a). Additionally, the final CO_2 uptake of MgO-treated contaminated soils rose with escalating initial Cd concentrations due to that acidic $CdCl_2$ enhanced the hydration degree of MgO, leading to the increase of the final CO_2 uptake [26].

From Fig. 1(b), the full carbonation process of CaO-treated sois are similar to that observed in MgO-treated soils. The CO_2 uptake of the CaO-treated uncontaminated soil increases notably in the initial 12 hours, and then gently until the CO_2 uptake/CaO ratio reaches 0.65 at the 168th hour. This trend also exists for the other CaO-treated soils, which however, achieve a stable CO_2 uptake/CaO ratio after 90 hours. The introduction of $CdCl_2$ does not noticeably slow down the carbonation process for CaO-treated soils, and the CO_2 uptake/CaO ratio reaches a stable value before the 96th hour. After carbonation for 7 days (i.e.168 hours), the values of CO_2 uptake for Cd-contaminated soils treated with CaO are generally similar (~65 %) among different initial Cd concentrations. This finding is different from the case in MgO-treated Cd-contaminated soils, of which the values increases with rising initial Cd concentrations. This could be attributed to that the acidic nature of $CdCl_2$ does not impact the hydration of CaO. Hence, with the increasing Cd concentration in soils, the consumption of CO_2 is similar.

Fig. 1(c) and (d) provide the curves of CO_2 uptake for soils treated by CaO and MgO subjected to partial carbonation. It takes 2 or 3 hours for CaO-treated soils to achieve a CO_2 uptake/CaO value of 33 %-38 %, while 3 hours for MgO-treated soils to achieve a CO_2 uptake/MgO value of 36 %-43 %. These values are about half of CO_2 uptake/CaO (or CO_2 uptake/MgO) value for the corresponding soils subjected to full carbonation. Regarding CaO-treated contaminated soils, the carbonation process slows down as the initial Cd concentrations increase within

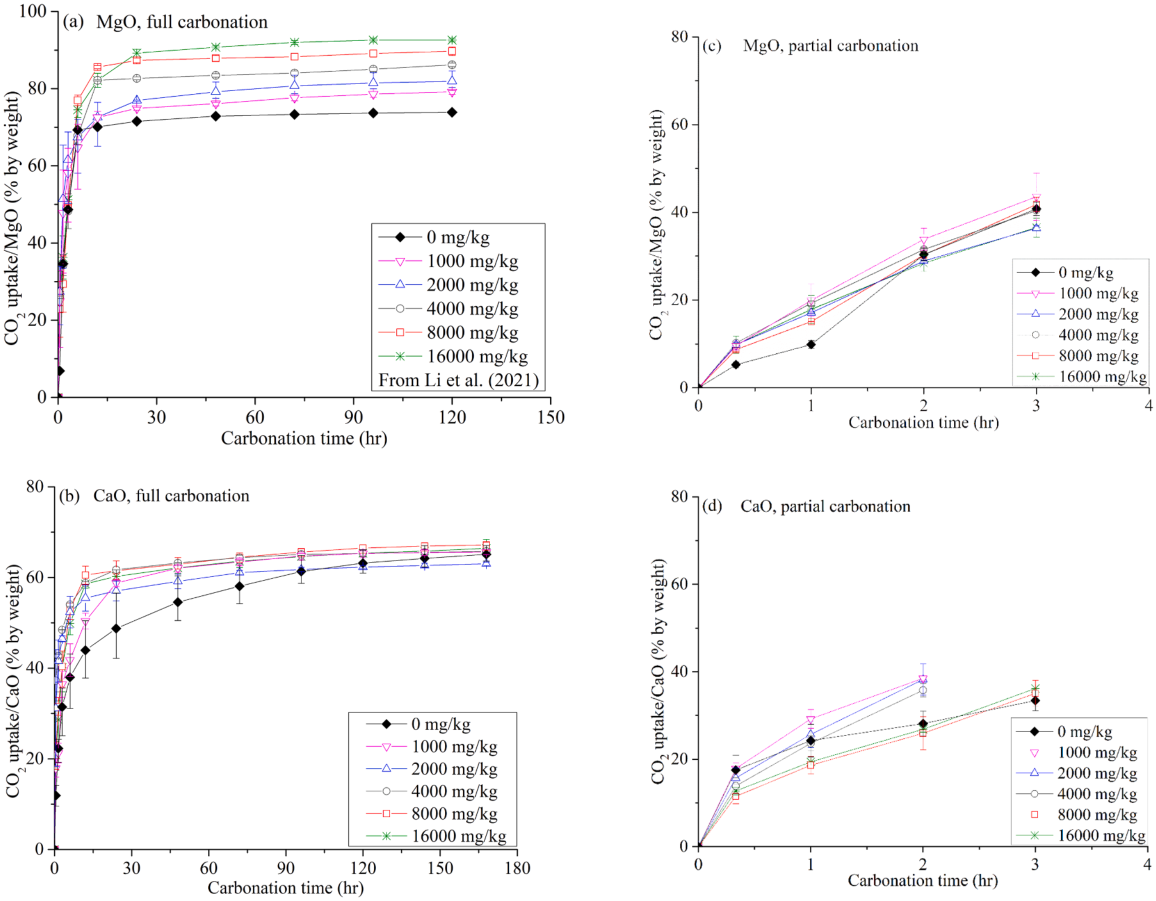

Fig. 1. CO_2 uptake for Cd-contaminated soils treated with (a) MgO, full carbonation [26], (b) CaO, full carbonation, (c) MgO, partial carbonation, (d) CaO, partial carbonation.

the time period of 3 hours, that is, higher CO_2 uptake/CaO value can be achieved in this time period for soils with lower Cd concentration. However, this phenomenon is not observed in MgO-treated contaminated soils, where the elevation of initial Cd concentration does not notably influence the carbonation process in the first 3 hours. The reason is linked with the similar solubilities (as shown in Table 2) of Cd $(OH)_2$ and $Mg(OH)_2$ [26], and the introduction of $CdCl_2$ will not significantly change the carbonation rate of MgO-treated soils. In contrast, the solubility of $Cd(OH)_2$ is considerably inferior to that of Ca $(OH)_2$ (Table 2), resulting in that Cd decelerates the carbonation of the CaO-treated soil. Higher Cd concentration would result in increased deceleration. The relationship between the deceleration effect and the solubility has been explained in previous studies [26,27].

3.2. XRD

Fig. 2 displays XRD patterns of uncontaminated and contaminated soils with 16,000 mg/kg of Cd treated by CaO and MgO subjected to full and partial carbonation. The primary minerals identified in the uncontaminated soil include quartz, kaolinite, and muscovite. As seen in Fig. 2 (a), calcite ($CaCO_3$), calcium carbonate hydrate (CCHA, $CaCO_3{\cdot}6\,H_2O$), and portlandite ($Ca(OH)_2$) are clearly identified in CaO-treated soil subjected to partial carbonation without the addition of Cd. Due to the co-existence of $CaCO_3$ and $Ca(OH)_2$, calcium carbonate hydroxide (CCHO) is formed from the chemical reactions between them. When the soil is fully carbonated, $CaCO_3$ is detected but $Ca(OH)_2$ is absent, illustrating that $Ca(OH)_2$ could completely transform to $CaCO_3$.

With the addition of Cd, $CaCO_3$, $Ca(OH)_2$, and CCHO exist under partial carbonation for CaO-treated contaminated soil. Meanwhile, multiple Cd compounds, e.g. cadmium carbonate ($CdCO_3$), cadmium hydroxide ($Cd(OH)_2$), cadmium hydroxide chloride (CdHCl), are identified. Under full carbonation, $CdCO_3$, $Cd(OH)_2$, and CdHCl are still present in CaO-treated Cd-contaminated soil, but $Ca(OH)_2$ is not detectable. The formation of these compounds illustrates that the partial and full carbonation process could precipitate some free Cd^{2+} into insoluble $CdCO_3$, $Cd(OH)_2$, and CdHCl.

The minerals of uncontaminated and MgO-treated contaminated soils subjected to full carbonation have been analyzed in previous study [26], which are provided here for comparison purpose. Under full carbonation, magnesium carbonates are identified in both uncontaminated and contaminated soils with MgO. Various Cd carbonates (e.g. $CdCO_3$ and $CdMg(CO_3)_2$ (cadcium magnesium carbonate)), along with CdHCl are also formed in contaminated soils. These findings demonstrate that the treatment (i.e. MgO, full carbonation) transforms the soluble salt (i.e. $CdCl_2$) into insoluble carbonates and a $Cd(OH)_2$ complex. Under partial carbonation, brucite ($Mg(OH)_2$) presents in the MgO-treated soils, and $Cd(OH)_2$ forms in the contaminated soil, indicating that $Cd(OH)_2$ and brucite do not react with CO_2, and are left in the partially carbonated MgO-treated soils.

Overall, XRD results show that $CaCO_3$, $Ca(OH)_2$, $CdCO_3$, and Cd $(OH)_2$ exist in partially carbonated CaO-treated soils, while $Ca(OH)_2$ is absent in fully carbonated soils, and the other three minerals are still detectable. This indicates $Ca(OH)_2$ continuously reacts with CO_2 until fully carbonated. The formed Ca carbonates and the absence of $Ca(OH)_2$ will affect the strength and pH environment of soils, further influencing the Cd leachability of soils. For MgO-treated contaminated soils, the full carbonation produces Cd and Mg carbonates from the reaction of Cd $(OH)_2$ (and $Mg(OH)_2$) and CO_2, and the complex of $Cd(OH)_2$ (i.e. CdHCl) is also formed due to the reaction between $Cd(OH)_2$ and $CdCl_2$. Under partial carbonation, Cd and Mg carbonates, $Mg(OH)_2$, along with Cd $(OH)_2$ are identified. The formation of Ca and Mg carbonates facilitates strength development of soils. Higher carbonation degree results in more Mg and Cd carbonates are formed and a new mineral (CdHCl) is produced. The existence of $CdCO_3$, and CdHCl in carbonated soils facilitates the reduction of leached Cd concentration.

Table 2
Data on the solubility of hydroxides and carbonates for Mg, Ca, and Cd.

Chemicals	log K_{sp} (25 °C)	Calculated solubility (g/L) at 25 °C
$Mg(OH)_2$	−11.25 [28]	6.49×10^{-3}
$Ca(OH)_2$	−5.3 [28]	0.166
$Cd(OH)_2$	−13.64 [33]	2.62×10^{-3}
$MgCO_3$	−5.17 [28]	0.219
$CaCO_3$	−8.47 [28]	5.8×10^{-3}
$CdCO_3$	−12.01 [33]	1.70×10^{-4}

Note: K_{sp} refers to the solubility product. NIST stands for National Institute of Standards and Technology (US).

3.3. TGA

Fig. 3 provides TGA and DTG (derivative thermogravimetry) curves of uncontaminated and contaminated soils with 16,000 mg/kg of Cd treated by CaO and MgO subjected to full and partial carbonation. Kaolinite decomposition, occurring at ~500℃, is detected in all treated soils and should be sourced from kaolin. With the treatment of CaO, a noticeable DTG peak for $CaCO_3$ is evident between 720 and 760℃ in CaO-treated soils during both partial and full carbonation. As shown in Fig. 3(a), under partial carbonation, it is challenging to distinguish portlandite at ~500℃ for both uncontaminated and contaminated soils because the weight loss at around this temperature is likely caused by the decomposition of kaolinite. $Cd(OH)_2$ or its complex (cadmium chlorite hydroxide, CdHCl) forms in partially carbonated contaminated soil with CaO as they both decompose at ~330℃. The presence of $CdCO_3$ can be confimed by its characterized decomposition temperature of ~380℃, after which the formed CdO further decomposes at ~570℃. Additionally, calcium chlorite ($CaCl_2$) is detected at the temperature of ~900℃, indicating that the chemical reaction between CaO and $CdCl_2$ occurred when CaO was used to treat Cd-contaminated soils. Comparatively, the contaminated soil experiences a greater weight loss (5.4 %) than uncontaminated soil, indicating the formation of complex Cd compounds in CaO-treated contaminated soil during carbonation. When the CaO-treated soils are fully carbonated (Fig. 3(b)), $CaCO_3$ is identified at ~760℃ for both contaminted and uncontaminted soils. In the Cd-contaminted soil treated with CaO, $CdCO_3$ is detected confidently but the presence of $Cd(OH)_2$ remains uncertain as there is not characterized decomposition temperature for $Cd(OH)_2$ and its complex.

In Fig. 3(c), during partial carbonation of MgO-treated soils, $MgCO_3$ is indentified at ~450℃, along with various magnesium carbonates (nesquehonite, dypingite, and hydromagnesite) detected between 100 and 250℃. The complex of $Cd(OH)_2$ (CdHCl) is observed in MgO-treated contaminated soil under partial carbonation, decomposing at ~195℃. The presence of $CdCO_3$ and its complex ($CdMg(CO_3)_2$) is uncertain due to their characterized decomposition temperatures (~380℃ and 600℃), which may overlap with the decomposition of $MgCO_3$ and CdO. In Fig. 3(d), the TGA results of MgO-treated soils between partial and full carbonation genearlly are very similar, showing that similar minerals are occurred between these soils. The above TGA results generally agree with the XRD results on the mineralogical products of CaO- and MgO-treated soils subjected to partial and full carbonation.

Table 3 summarizes TGA-induced weight loss for each soil during the heating from 30 to 900 ℃. Regardless of the binder type and carbonation degree, Cd-contaminated soils consistently exhibit higher weight loss than uncontaminated soils due to the decomposition of Cd compounds, confirming the existence of Cd compounds. Full carbonation leads to higher weight loss compared to partial carbonation for the same soil (uncontaminated or contaminated), indicating more carbonates (e.g., $CaCO_3$, $MgCO_3$, and $CdCO_3$) formed in fully carbonated soils. For example, regarding CaO-treated Cd-contaminated soils, the full carbonation causes the weight loss of 24.6 %, while the partial carbonation induces 23.2 %. This probably results from the formation of more $CaCO_3$ and $CdCO_3$ in the fully carbonated CaO-treated contaminated soils. Similar to XRD results, more $CaCO_3$ and $CdCO_3$ will affect

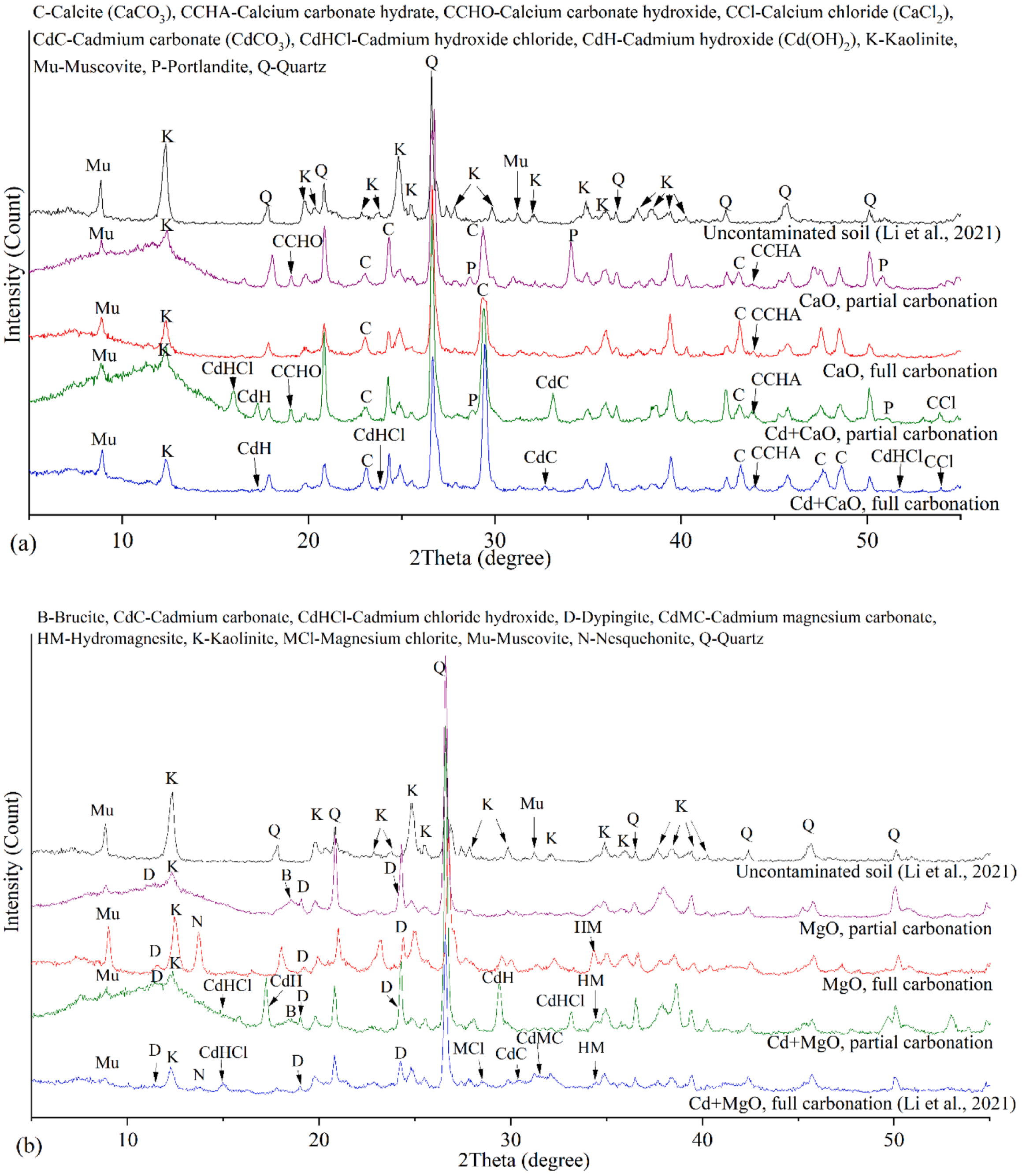

Fig. 2. XRD curves for soils treated by (a) CaO and (b) MgO under partial and full carbonation.

the strength and pH environment of soils, further influencing the Cd leachability of soils. Furthermore, MgO treatment results in greater weight loss than CaO treatment, indicating that MgO facilitates the formation of more decomposable carbonates, leading to increased weight loss. This result also illustrates that MgO can absorb more CO_2 than CaO, facilitating better strength development and lower Cd leachability for Cd-contaminated soil.

3.4. UCS results

Fig. 4 shows the UCS of uncontaminated and Cd-contaminated soils treated with CaO and MgO undergoing both full and partial carbonation. MgO-treated Cd-contaminated soils produce comparable UCS values to those treated with CaO under 28-day conventional curing, and the UCS values keep stable (less than 150 kPa) with the increasing initial Cd concentration.

When CO_2 is introduced, the soils treated with MgO or CaO present higher UCS than those under 28-day conventional curing. For CaO-carbonated soils, UCS is similar for partial and full carbonation (Fig. 4 (a)), indicating that Ca carbonates boost soil strength, but increased carbonation does not lead to a substantial enhancement in strength. This is probably linked with the formation of $CaCO_3$ and $Ca(HCO_3)_2$. Initially, formed $CaCO_3$ enhances soil strength by binding particles. However, full carbonation leads to the production of $Ca(HCO_3)_2$, causing a decrease in CaO-treated soil strength [41]. Notably, $Ca(HCO_3)_2$ is not confirmed by XRD and TGA tests, likely because it easily decomposes to $CaCO_3$ in the dry state and is difficult to exist in the soil mixture [16,58].

Under full carbonation, the UCS of CaO-treated contaminated soils decreases from 942 to 330 kPa with increasing initial Cd concentration (0–4000 mg/kg), and then fluctuates between 330 and 536 kPa (4000 to

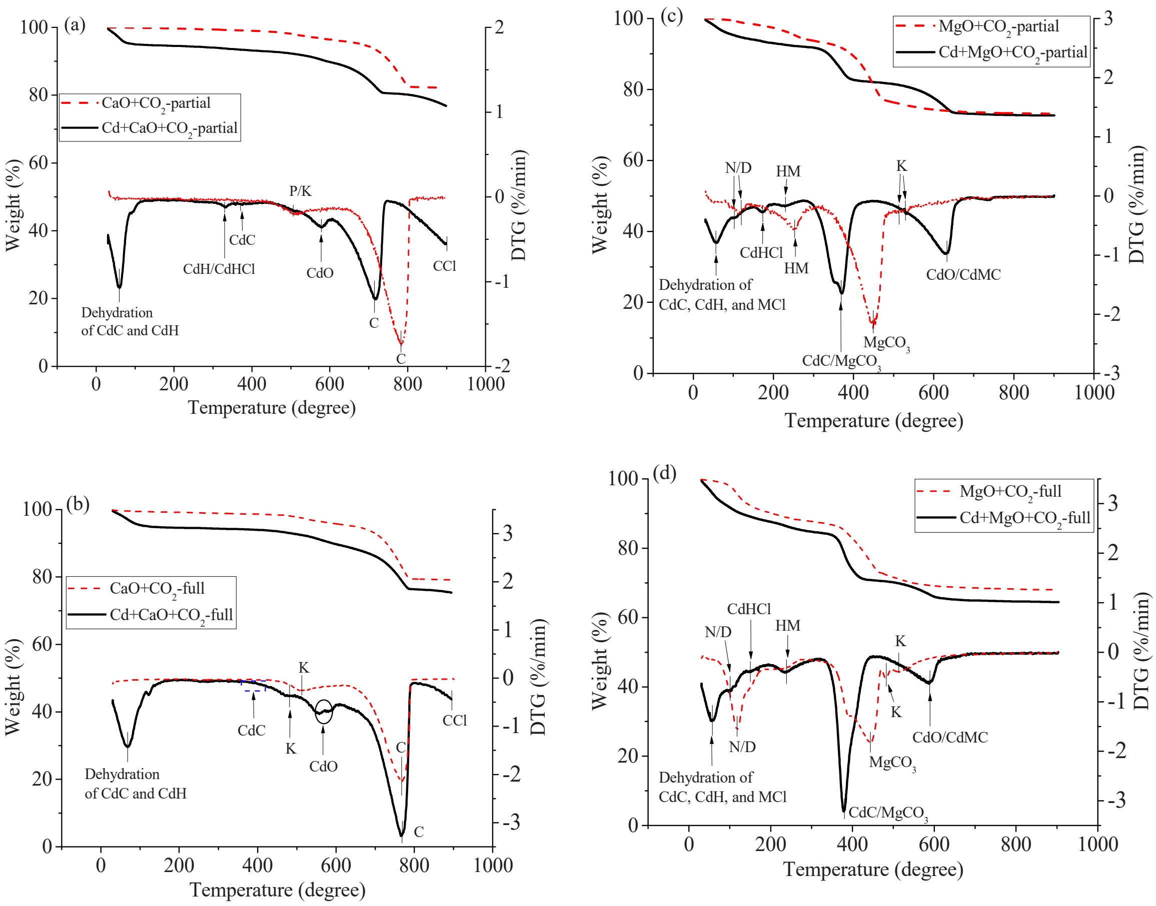

Fig. 3. TGA curves for soils treated by (a) CaO, under partial carbonation, (b) CaO, under full carbonation, (c) MgO, under partial carbonation, and (d) MgO, under full carbonation. (Note: C-Calcite ($CaCO_3$), CCl-Calcium chloride ($CaCl_2$), CdC-Cadmium carbonate ($CdCO_3$), CdH-Cadmium hydroxide ($Cd(OH)_2$), CdHCl-Cadmium hydroxide chloride, CdMC-Cadmium magnesium carbonate, CdO-Cadmium oxide, D-Dypingite, HM-Hydromagnesite, K-Kaolinite, MCl-Magnesium chlorite, N-Nesquehonite, P-Portlandite).

Table 3
The weight loss of soil during TGA test.

Binder	Carbonation degree	Soil type	Weight loss (%)
CaO	Partial	Uncontaminated	17.8
		Contaminated	23.2
	Full	Uncontaminated	20.8
		Contaminated	24.6
MgO	Partial	Uncontaminated	26.9
		Contaminated	27.3
	Full	Uncontaminated	32.0
		Contaminated	35.6

16,000 mg/kg). Under partial carbonation, as Cd concentration rises from 0 to 2000 mg/kg, UCS of these soils slightly increases from 595 to 629 kPa but drops to 260 kPa at 4000 mg/kg, followed by fluctuations (4000 to 16,000 mg/kg). This suggests that the heavy metal Cd generally weakens the strength of CaO-treated soils under both carbonations, though the specific reasons remain unknown.

As seen in Fig. 4(b), the soils containting MgO always present higher UCS values than those containing CaO irrespective of the carbonation degree as the formation of Mg carbonates contributes to the strength enhancement of soils. The UCS for MgO-treated soils subjected to full carbonation decreases from 2191 to 434 kPa as the initial Cd concentration increases from 0 to 16,000 mg/kg. Although the UCS range of 780–932 kPa under partial carbonation is much narrower than that of soils under full carbonation, the UCS under partial carbonation generally decreases with the escalating initial Cd concentration. Again, the result indicates that Cd exerts an adverse impact on the strength of MgO-treated soils. Additionally, the higher the degree of carbonation, the greater the strength of MgO-treated soils.

Overall, as long as CO_2 is introduced, contaminated soils treated with CaO or MgO generally satisfy the strength requirement (i.e. 350 kPa) outlined by USEPA [47] if such soils intend to be disposed in landfills.

3.5. Cd leachability

Fig. 5 shows the leachate pH of Cd-contaminated soils treated by CaO and MgO under full and partial carbonation. When treated with CaO, the pH of soils decreases with the rise in Cd concentrations, as depicted in Fig. 5(a). As the degree of carbonation increases, the pH tends to decrease. For example, pH ranges from 12.39 to 12.31 for contaminated soils treated with CaO with 28-day conventional curing, 12.38–12.13 for CaO subjected to partial carbonation, and 11.24–9.8 for CaO subjected to full carbonation. This pH reduction is due to CO_2, an acidic gas. Greater CO_2 absorption, associated with higher carbonation degree, leads to decreased pH in CaO-treated soils. Saturated CaO and $CaCO_3$ have pH values of 12.5 and ~10, respectively, so partially carbonated CaO-treated soils with pH $>$ 12 suggest the presence of $CaCO_3$ and $Ca(OH)_2$, aligning with XRD analysis. The pH of 11.24 and 10.74 for fully carbonated soils indicates that both $CaCO_3$ and $Ca(OH)_2$ likely exist at lower Cd concentrations, specifically 1000 and 2000 mg/kg. However, at higher Cd concentrations (i.e., 4000, 8000, and 16,000 mg/kg), only $CaCO_3$ is formed in the soils due to their pH values ranging from 10 to 9.8. As stated earlier, CO_2 is consumed for the formation of $CaCO_3$ and $Ca(HCO_3)_2$ in CaO-treated soils. The pH values

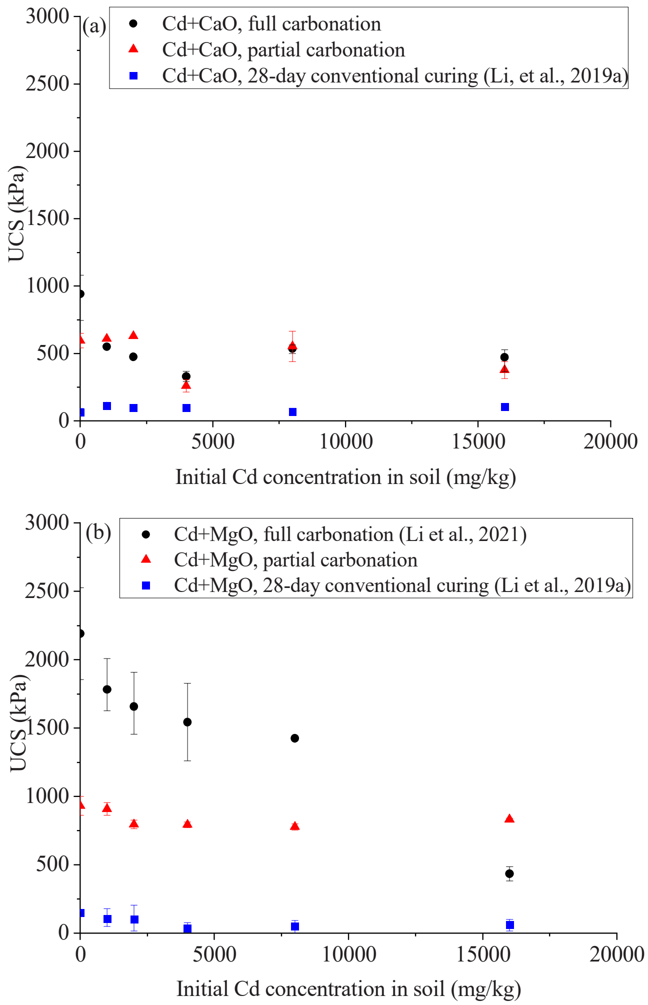

Fig. 4. UCS of carbonated soils containing (a) CaO and (b) MgO.

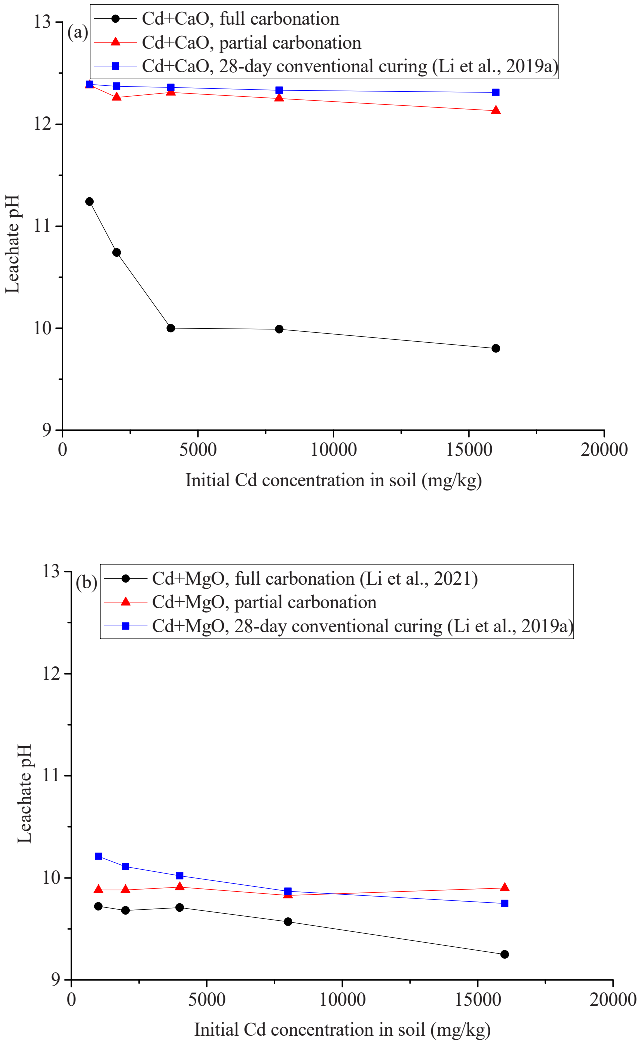

Fig. 5. Leachate pH of carbonated soils containing (a) CaO and (b) MgO.

of $Ca(HCO_3)_2$ and $CaCO_3$ are 8–9 and ~10, respectively, which are lower than the pH (~12.45) of CaO, so the formation of $CaCO_3$ and $Ca(HCO_3)_2$ will decrease the pH of carbonated CaO-treated soil. Additionally, the acidic nature of $CdCl_2$ will lead to the decrease of pH with the increasing Cd concentration. The combination of above two factors, therefore, contributes to the trend of the leachate pH of carbonated CaO-treated soil.

Fig. 5(b) illustrates that MgO-treated soils have lower pH values than CaO-treated soils because of the inherent characteristics of MgO and CaO (MgO and CaO with pH of ~10 and 12.5, respectively). The pH of contaminated soils under three conditions gradually decreases with the increasing Cd concentration. Like CaO-treated soils, soils treated with MgO have a similar pH trend, wherein the pH decreases with the increase of the carbonation degree. The highest pH appears in soils treated with MgO with 28-day conventional curing, ranging from 10.21 to 9.75. The second highest is for MgO-treated soils subjected to partial carbonation, ranges from 9.90 to 9.83. The lowest pH, from 9.72 to 9.25, is found in those soils subjected to full carbonation. This pH difference is attributed to the formation of $MgCO_3$, which has a pH of approximately 9.3 [42], lower than that of $Mg(OH)_2$ (i.e., 9.5–10). As the degree of carbonation increases, more $Mg(OH)_2$ is consumed to form Mg carbonates, leading to a decrease in the pH for MgO-treated soils. The second highest pH in partially carbonated MgO-treated soils suggests the coexistence of $MgCO_3$ and $Mg(OH)_2$, consistent with XRD results.

Fig. 6 provides the Cd leachability of contaminated soils treated with CaO and MgO subjected to full and partial carbonation, as well as two limits specified by the Standard EN [13]. From Fig. 6(a), the Cd leachability slowly decreases from 0.12 to 0.045 mg/kg with the rise of initial Cd concentrations for CaO-treated soils with 28-day conventional curing, while increases from 0.042 to 0.18 mg/kg for those subjected to partial carbonation. In both cases, the Cd leachability remains below the limit for non-hazardous waste (i.e., 1 mg/kg), but exceeds 0.04 mg/kg, a limit for inert waste outlined by EN [13]. However, a different scenario emerges when full carbonation is achieved. For fully carbonated CaO-treated soils, the Cd leachability sharply increases from 0.029 to 48.5 mg/kg with rising initial Cd concentrations. Notably, with higher intial Cd concentrations (i.e., 8000 and 16,000 mg/kg), the Cd leachability surpasses the 1 mg/kg limit. These results illustrate that the partial carbonation and 28-day conventional curing are more suitable for the treatment of CaO-treated soils than the full carbonation, probably due to the high pH that the soils these two treatments produce. Johnson [19] have indicated that the leachability of heavy metals is influenced by pH, with the optimal pH for minimizing Cd leachability reported to be around 11. This pH value is comparable to that of CaO-treated soils subjected to 28 days curing and partial carbonation, along with two soils (i.e. 1000 and 2000 mg/kg Cd) subjected to full carbontion.

The treatment methods involving MgO generally provide poorer S/S efficiency than those involving CaO as shown in Fig. 6(b), because the leached Cd concerntrations of MgO-treated soils are mostly higher than those of CaO-treated soils. The Cd leachability in fully carbonated MgO-treated soils exhibits a slight fluctuation, ranging from 0.31 to 0.72 mg/

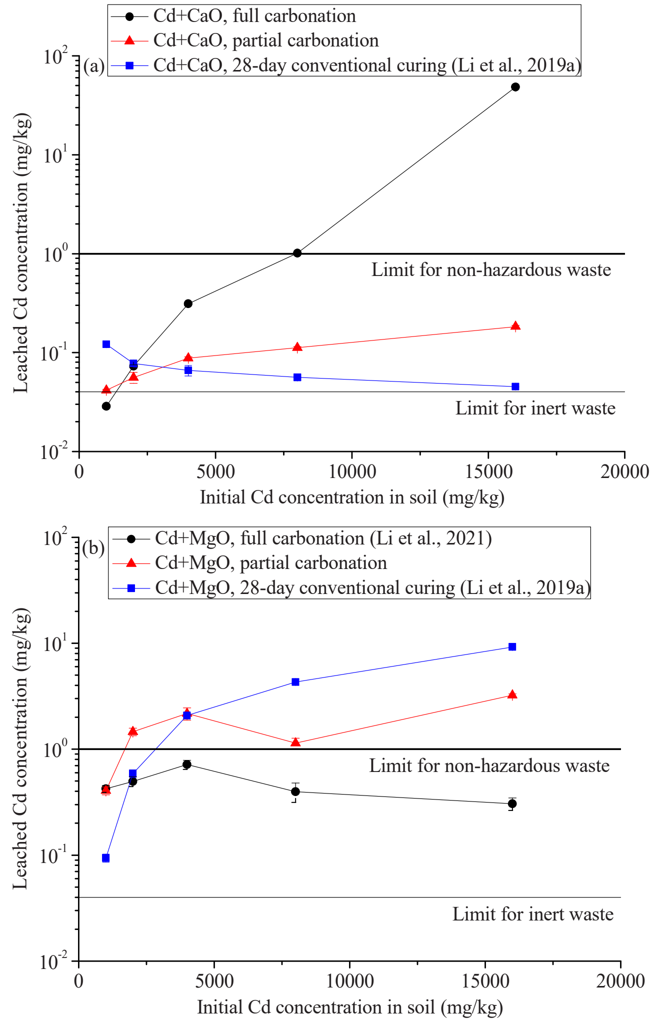

Fig. 6. Cd leachability of carbonated soils containing (a) CaO and (b) MgO.

kg with increasing initial Cd concentration. However, all these values are below the limit of 1 mg/kg specified by EN [13]. The MgO-treated soils subjected to 28-day conventional curing and partial carbonation generall produce higher Cd leachability than those subjected to full carbonation at initial Cd concentrations of 2000, 4000, 8000, 16, 000 mg/kg. Particularly, the leached Cd concerntrations of soils subjected to 28 days curing (4000, 8000, and 16,000 mg/kg) and partial carbonation (2000, 4000, 8000, and 16,000 mg/kg) exceed the limit for non-hazardous watse specified by EN [13]. Usually, $CdCO_3$, $Cd(OH)_2$, and CdHCl existing in partially and full carbonated soils facilitate the reduction of leached Cd concentration.

Fig. 7 shows the correlation between pH and Cd leachability for contaminated soils treated by CaO and MgO subjected to 28-day conventional curing, full and partial carbonation. pH plays a crucial role in the precipitation-dissolution and coordination-dissociation equilibrium of Cd compounds in soils, ultimately affecting their speciation and leachabilities [44]. The theoretical solubility curves of $Cd(OH)_2$ and $CdCO_3$ obtained from [52] and Johnson [19] are also provided in Fig. 7. These curves reflect the trend of solubility with pH for $Cd(OH)_2$ and $CdCO_3$, which are used to analyze the type of the formed minerals. The leached Cd concentrations genearlly distribute around the solubility curve for $Cd(OH)_2$ and are much higher than the solubility curve of $CdCO_3$. This result illustrates that $Cd(OH)_2$ may form in all treated soils and the formation of $CdCO_3$ is probably limited in the treated soils, even in soils subjected to partial and full carbonation. Hence, the Cd leachability of these treated soil is likely controlled by the formation of $Cd(OH)_2$, but can be decreased to some extent by the carbonation process.

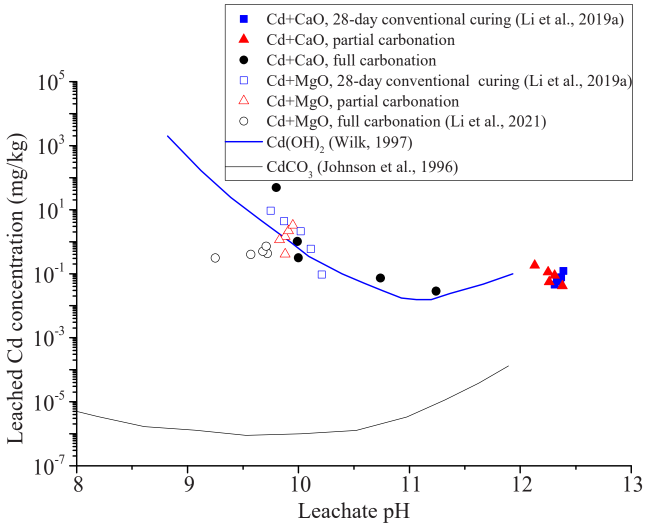

Fig. 7. Leachate pH VS leached concentrations for Cd-contaminated soils treated by CaO and MgO under 28-day conventional curing, partial and full carbonation.

4. Discussion

Both CaO and MgO treatment, the carbonation degree has an effect on the strength and leaching properties of soils. The UCS of CaO-treated soils with partial and full carbonation is generally similar, but the leached Cd concentrations of partially carbonated CaO-treated soils are lower than those of fully carbonated CaO-treated soils. As shown in XRD and TGA results (Figs. 2 and 3), $CaCO_3$, $Ca(OH)_2$, $CdCO_3$, and $Cd(OH)_2$ are all detected in partially carbonated CaO-treated soils, while $Ca(OH)_2$ is absent in fully carbonated soils, and the other three minerals are still detectable. It can be deduced that residue $Ca(OH)_2$ existed in partially carbonated CaO-treated soils transforms into $CaCO_3$ when the full carbonation is achieved. However, $Cd(OH)_2$ is difficult to completely transform into $CdCO_3$ under the condition of full carbonation. This phenomenon could be explained as below. When $CdCl_2$ is mixed with CaO and soil before carbonation, the dissolved Cd^{2+} in pore water of soil will react with OH^- produced from CaO hydration (Eq. 1) to form the precipitation of $Cd(OH)_2$ (Eq. 6). In doing so, the OH^- concentration in the pore water will decrease and hence the hydration degree of CaO will increase. With the introduction of CO_2, the formed H^+ from Eq. 3 will bind OH^- in the pore water (Eq. 4), and simultaneously, $CaCO_3$ (Eq. 5) and $CdCO_3$ (Eq. 7) will form and precipitate. Due to the decrease of Cd^{2+}, Ca^{2+}, and OH^- concentrations, Eqs. 1, 2, and 6 will lose equilibrium and produce more Cd^{2+}, Ca^{2+}, and OH^- to reach new equilibrium. As $Cd(OH)_2$ has much lower solubility than $Ca(OH)_2$ (as shown in Table 2), it will release OH^- more slowly and hence $Cd(OH)_2$ is hard to completely transform to $CdCO_3$ within certain time period. The formation of $CaCO_3$ facilitates strength development of soils. Although fully carbonated CaO-treated soils has more $CaCO_3$ than partially carbonated CaO-treated soils, the UCS of both soils is generally similar. This is probably due to that some $CaCO_3$ transforms into soluble $Ca(HCO_3)_2$ under full carbonation, which negatively affects the soil strength, but cannot be detected by XRD tests. Additionally, as the existence of $CdCO_3$ and $Cd(OH)_2$ in partially and fully carbonated CaO-treated soils, the leached Cd concentrations of both soils are controlled by the solubilities of $CdCO_3$ and $Cd(OH)_2$. As known from Fig. 7, the Cd leachability of these soils is likely controlled by the formation of $Cd(OH)_2$, of which the solubility depends on pH environment of soils and the minimum value occurs at pH of ~12. The existence of $Ca(OH)_2$ in partially carbonated CaO-treated soils induces a pH of 12.13–12.38, which close to 12, resulting in lower Cd leachabilities compared to fully carbonated CaO-treated soils with a lower pH range of 9.8–11.24. Several studies [29, 37] have shown that $CaCO_3$ or its other forms (e.g. alcite, aragonite, and vaterite) can absorb Cd through physisorption and chemisorption, which may contribute to the decrease of Cd leachabilities, but the amount of $CaCO_3$ and its other forms contained in treated Cd-contaminated soils remains unclear. Moreover, some $CaCO_3$ transforms into soluble $Ca(HCO_3)_2$ under full carbonation, possibly resulting in the increase of leached Cd concentrations. Therefore, the effect of $CaCO_3$ on the Cd leachability cannot be clearly identified.

$$\mathrm{CaO}_{(s)}(\mathrm{MgO}_{(s)}) + H_2O_{(l)} \rightarrow \mathrm{Ca}^{2+}{}_{(aq)}(Mg^{2+}{}_{(aq)}) + 2OH^{-}{}_{(aq)} \tag{1}$$

$$\mathrm{Ca}^{2+}{}_{(aq)}(\mathrm{Mg}^{2+}{}_{(aq)}) + 2OH^{-}{}_{(aq)} \leftrightarrow \mathrm{Ca}(OH)_{2(s)}(Mg(OH)_{2(s)}) \tag{2}$$

$$\mathrm{CO}_{2(g)} + H_2O_{(l)} \leftrightarrow CO_3^{2-}{}_{(aq)} + 2H^{+}{}_{(aq)} \tag{3}$$

$$H^{+}_{(aq)} + OH^{-}_{(aq)} \leftrightarrow \mathrm{H_2O}_{(l)} \tag{4}$$

$$\mathrm{Ca}^{2+}{}_{(aq)}(\mathrm{Mg}^{2+}{}_{(aq)}) + CO_3^{2-}{}_{(aq)} \leftrightarrow \mathrm{Ca}CO_{3(s)}(MgCO_{3(s)}) \tag{5}$$

$$\mathrm{Cd}^{2+}{}_{(aq)} + 2OH^{-}{}_{(aq)} \leftrightarrow \mathrm{Cd}(OH)_{2(s)} \tag{6}$$

$$\mathrm{Cd}^{2+}{}_{(aq)} + CO_3^{2-}{}_{(aq)} \leftrightarrow \mathrm{Cd}CO_{3(s)} \tag{7}$$

For MgO-treated contaminated soils, $CdCO_3$ and Mg carbonates, Mg $(OH)_2$, along with $Cd(OH)_2$ are identified under partial carbonation (Fig. 2). When the full carbonation is achieved, $CdCO_3$, Mg carbonates, and CdHCl are detected, while $Mg(OH)_2$ and $Cd(OH)_2$ are absent. It is suggested that with the increase of carbonation degree, $Mg(OH)_2$ and Cd $(OH)_2$ existed in partially carbonated MgO-treated soils generally transform into $CdCO_3$ and Mg carbonates, except some $Cd(OH)_2$ reacts with $CdCl_2$ to form the complex (i.e. CdHCl). The formation of Mg carbonates enhances the soil strength. Fully carbonated MgO-treated soils have more Mg carbonates than partially carbonated MgO-treated soils, leading to a higher UCS for fully carbonated MgO-treated soils. Different from CaO-treated Cd-contaminated soils, $Cd(OH)_2$ has similar solubility with $Mg(OH)_2$ (as shown in Table 2) in MgO-treated Cd-contaminated soils. Hence, $Cd(OH)_2$ can release OH^- at a similar rate with $Mg(OH)_2$, resulting in $Cd(OH)_2$ can completely transform to $CdCO_3$ under full carbonation and no $Cd(OH)_2$ is detected in fully carbonated MgO-treated soils. The presence of $Cd(OH)_2$, $CdCO_3$, and CdHCl controls the Cd leachability of fully and partially carbonated MgO-treated soils. As shown in Fig. 7, the solubility of $CdCO_3$ is much lower than that of Cd $(OH)_2$, so fully carbonated MgO-treated soils, which has more $CdCO_3$, will produce lower Cd leachability in comparison to partially carbonated MgO-treated soils at pH of 9.90–9.25. Additionally, Mg carbontes has a potential to absorb heavy metals (i.e. chromium, copper, and arsenic) [22,30], which may contribute to the lower Cd leachabilities for fully carbonated MgO-treated soils as more Mg carbonates may form in such soils compared to partially carbonated MgO-treated soils.

Overall, partial carbonation fits for treating CaO-treated Cd-contaminated soils, while full carbonation is more suitable for MgO-treated soils.

5. Conclusions

The MgO-treated soils present higher UCS values than CaO-treated soils under carbonation. Both soils generally comply with the strength requirement of 350 kPa outlined by USEPA. The UCS of CaO-treated soils with partial and full carbonation is generally similar, in a range of 942–330 kPa, while the UCS for MgO-treated soils subjected to full carbonation is higher than that subjected to partial carbonation. The Cd leachability of MgO-treated soils are mostly higher compared to that of CaO-treated soils. The leached Cd concerntrations of partially carbonated CaO-treated soils are within 0.042–0.18 mg/kg, below the limit for non-hazardous waste . However, when the full carbonation is achieved, the Cd leachability of soils sharply increases, and even exceed the limit. The fully carbonated MgO-treated soils have lower Cd leachability than the limit, but partially carbonated MgO-treated soils generally produce higher Cd leachability, and some exceed the limit.

XRD and TGA results show that $CaCO_3$, $Ca(OH)_2$, $CdCO_3$, and Cd $(OH)_2$ exist in partially carbonated CaO-treated soils, while $Ca(OH)_2$ is absent in fully carbonated soils, and the other three minerals are still detectable. For MgO-treated contaminated soils, the full carbonation produces Cd and Mg carbonates, but CdHCl is also formed. Under partial carbonation, Cd and Mg carbonates, $Mg(OH)_2$, along with $Cd(OH)_2$ are identified. The formation of Ca and Mg carbonates contributes to strength improvement of soils. For MgO-treated soils, the higher carbonation degree results in higher strength, but this trend is not observed for CaO-treated soils. The existence of $CdCO_3$, $Cd(OH)_2$, and CdHCl in partially and full carbonated soils facilitates the reduction of leached Cd concentration. The presence of $Cd(OH)_2$ and CdHCl likely controls the Cd leachability. Consequently, partial carbonation is more suitable for treating CaO-treated Cd-contaminated soils, while full carbonation is more suitable for MgO-treated soils. Results of the leaching test highlight the importance of cadmium speciation, which, however, is not well revealed in this study and should be further investigated using other tests, such as sequential extraction.

CRediT authorship contribution statement

Wentao Li: Writing – original draft, Methodology, Investigation, Formal analysis. **Junde Qin:** Writing – review & editing, Methodology, Investigation. **Yaolin Yi:** Writing – review & editing, Supervision, Project administration, Funding acquisition, Formal analysis, Conceptualization.

Acknowledgement

The authors appreciate support from The Key Research and Development Program of Hubei Province, China (No. 2023BAB024), and Joint Funds of the Natural Science Foundation of Hubei Province, China (No. 2022CFD130), and China- Singapore International Joint Research Institute (No. N2052401–A204).

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be made available on request.

Appendix A. Supporting information

Supplementary data associated with this article can be found in the online version at doi:10.1016/j.jcou.2024.102953.

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