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# Preparation of calcium carbonate nanoparticles from waste carbide slag based on $CO_2$ mineralization

Wenxiu Li<sup>a</sup>, Yan Huang<sup>c</sup>, Tao Wang<sup>a,b,\*</sup>, Mengxiang Fang<sup>a,b</sup>, Yan Li<sup>c</sup>

<sup>a</sup> State Key Laboratory of Clean Energy Utilization, Zhejiang University, Hangzhou, 310027, PR China

<sup>b</sup> Qingshan Lake Energy Research Base, Zhejiang University, Hangzhou, 311300, PR China

<sup>c</sup> Shanxi Guohua Jinjie Energy Co., Ltd., Yulin, 719000, PR China

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### ABSTRACT

This study proposes a novel method for simultaneously dealing with the waste carbide slag (WCS) and  $CO_2$  mitigation. In addition, the ultrafine vaterite CaCO<sub>3</sub> with high economic value was produced under mild conditions of 25 °C and 0.1 MPa. Ammonium sulfate ((NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>) was employed to efficiently extract calcium from WCS into the solid phase calcium sulfate dehydrate (CaSO<sub>4</sub>·2H<sub>2</sub>O). Subsequently, it was subjected to "dropwise carbonation" leading to the formation of a calcium carbonate with controllable particle size between 100 nm–1 µm through multiple crystalline phase transformations. The results showed that nano-sized CaCO<sub>3</sub> with nearly 90% purity and whiteness was prepared at 25 °C, CO<sub>2</sub>-500 mL/min, and NH<sub>4</sub><sup>+</sup>/Ca<sup>2+</sup> = 2.4 conditions. Results of TG and FTIR analyses confirmed that CaCO<sub>3</sub> polymorphs with vaterite and metastable were formed. Based on the calcium conversion ratio of the entire process, treatment with 1 ton of WSC captured ~0.5 tons of CO<sub>2</sub> and yields about 1.15 tons of nano-sized CaCO<sub>3</sub> per each cycle operation. This study provides a valuable method for identifying potential application for scale preparation of vaterite nanoparticles from low cost but abundant calcium resource such as WCS and gypsum.

### 1. Introduction

Carbon capture and storage (CCS) is a promising technology, which can not only minimize the effects of climate change caused by carbon dioxide (CO<sub>2</sub>) emissions but also make industrial processes sustainable, benefiting future generations (Galina et al., 2019). Among the CCS options, CO<sub>2</sub> mineralization can permanently sequester CO<sub>2</sub> in the form of carbonate minerals without long-term monitoring and effectively generate high-value-added products (Xu et al., 2019).

Waste carbide slag (WCS) as a typical Ca-containing alkaline material, is a by-product of the hydrolysis of calcium carbide with a yield of ~20 tons (90 wt% of water content) for each ton of polyvinyl chloride (PVC) produced (Wang et al., 2020). As one of the typical industrial solid wastes, it usually occupies land and causes resource waste and environmental pollution (Cheng et al., 2009). Currently, some efforts have been made to reuse WCS converting it into harmless and useful materials (Guo et al., 2017; Miró et al., 2014). Because of its high content of calcium oxide (CaO) (about 85 wt%), synthesizing high-value calcium carbonate nanoparticle (CCNP) by carbonation can simultaneously address the WCS treatment and CO<sub>2</sub> emission, which is believed to be a more significant contributor to  $CO_2$  mitigation (Goeppert et al., 2012; Wang et al., 2011). It is well known that  $CaCO_3$  has three polymorphs: calcite, aragonite and vaterite, among which vaterite particles exhibit high hydrophilicity and hierarchical structures (Bo et al., 2019). It can be used to enhance the smoothness and ink adsorption in papermaking and improving the mechanical properties of plastic (Jimoh et al., 2018). Other applications in PVC, paint, rubber have also been extensively explored (Chen et al., 2011; Zhang et al., 2012).

Currently, direct and indirect carbonation as major processes are employed to produce calcium carbonate (CaCO<sub>3</sub>). Because single-step direct carbonation with slower reaction kinetic rates and lower conversion ratio (Liu et al., 2021), requiring high reaction temperature or pressure, the indirect aqueous carbonation process with relatively mild conditions has attracted considerable attention. It involves extracting calcium components from raw materials using acids, bases, and salts, followed by carbonation by bubbling CO<sub>2</sub>, which can effectively produce high purity and uniformly shaped CaCO<sub>3</sub> particles with narrow particle size distributions (PSD) under ambient conditions (Kezuka et al., 2018). Moreover, this technique is a better choice to meet industrial demands owing to its reliable operation and easy control of process

\* Corresponding author., Institute for Thermal Power Engineering, Zhejiang University, Hangzhou, 310027, PR China. *E-mail address:* oatgnaw@zju.edu.cn (T. Wang).

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parameters (Chen et al., 2012). The pH-swing technology was implemented as a means of accelerating the dissolution of minerals and carbonate precipitation (Azdarpour et al., 2015). Among the recent studies (Hu et al., 2017; Jo et al., 2014; Said et al., 2013; Zhang et al., 2020), acidic ammonia salt (NH4NO3, NH4Cl, NH4HSO4 and (NH4)2SO4) as a calcium extractant was considered the most promising approach. Wang and Maroto-Valer (2011) found that nearly 100% reactive components  $(Ca^{2+}, Mg^{2+})$  were extracted from feedstocks with 1.4 M ammonium bisulfate (NH<sub>4</sub>HSO<sub>4</sub>) in 3 h at 100 °C, and high purity of CaCO<sub>3</sub> was prepared in the carbonation stage. Recently, the thermal reduction technology of multi-stage precipitation was proposed, in which the blast furnace slag was firstly roasted with ammonium sulfate ((NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>) at 300-450 °C to convert the calcium component into sulfates, and then carbonated with ammonium bicarbonate/ammonium carbonate  $(NH_4HCO_3/(NH_4)_2CO_3)$  solution at relatively mild conditions (Hu et al., 2017; Wang et al., 2018). Moreover, our previous work showed that calcium was selectively extracted from WCS with (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> aqueous solution, and the calcium conversion ratio reached approximately 90% in about 30 min under ambient conditions. From a technical point of view, ammonia salt as an extractant is feasible, and high purity of CaCO<sub>3</sub> can be obtained by adjusting the pH. However, properties of the produced carbonates such as particle size, phase, morphology, etc. are of little concerned.

The precipitation of CCNP was associated with early-stage nucleation rate of CaCO<sub>3</sub>. The nucleation rate is mainly dependent on the supersaturation of the reaction solution, which is also affected by parameters, such as the initial ion concentration, stirring speed, reaction time, etc., (Babou-Kammoe et al., 2012). In previous studies, nano-sized CaCO<sub>3</sub> was mainly synthesized from the supersaturated solution with calcium and carbonate ions assisted by additives (Konopacka-Lyskawa et al., 2019; Nagaraja et al., 2014). Wang et al. (Wang et al., 2020) found that organic acid salts such as citrate could promote the extraction of calcium ions and exert a strong surface effect on the crystallization of nanocrystals. The additives such as polyols were found to reinforce the early nucleation rate by increasing supersaturation and decreasing the nucleation barrier (Svenskaya et al., 2018; Trushina et al., 2014, 2016). Besides, polyphosphates could also prolong the induction period probably by enhancing complexation with  $Ca^{2+}$  (Bang et al., 2011). Incorporation of ethylene diamine tetra acetic acid (EDTA) at a mass ratio of 2.5%, resulted in CaCO<sub>3</sub> particles with good dispersity. So far, no study has explored the simultaneously effect of carbonation based on industrial solid waste and nano-sized CaCO<sub>3</sub> preparation.

Here, we propose a stepwise mild route for the preparation of nanosized  $CaCO_3$  from WCS based on  $CO_2$  mineralization. Firstly, the calcium component was extracted from WCS using  $(NH_4)_2SO_4$  solution. Next, impurities were separated from the leaching products by flotation. Finally, CCNP was prepared by  $CO_2$  mineralization using purified  $CaSO_4$ ·2H<sub>2</sub>O. This study aimed to determine the optimum conditions for producing CCNP and the possible factors affecting the particle size and calcium conversion ratio. Additionally, the actual  $CO_2$  uptake of the WCS was also evaluated and the results showed that the proposed route effectively achieved  $CO_2$  fixation. This work offers a new approach that can address the WCS by converting it into high-value products while fixing  $\mathrm{CO}_2$ .

# 2. Materials and methods

# 2.1. Materials

The WCS sample was obtained from an acetylene plant in Yulin, China. All the samples were dried in an oven at 80 °C until the weight was constant. The PSD presented in Fig. 1a shows that 84.26% of particles were smaller than 425  $\mu$ m. In addition, large particles (>425  $\mu$ m) had remarkably high levels of impurities, such as carbon residue. Thus, the feedstocks were blended into a fine powder that was sieved through a 150 µm sieve, thereby avoiding the effect of the impurities. The pretreated WCS was kept in a vacuum dryer to prevent the reaction with CO<sub>2</sub> in the air. The chemical composition was determined using X-ray fluorescence (XRF, Panalytical) and the results were listed in Table 1. In addition, all the raw materials (ammonium sulfate, glycerol (Gly), sodium tripolyphosphate (STP) and EDTA) were analytical grade and purchased from Sigma-Aldrich. The ASDP was synthesized using the equimolar EDTA and STP system. Ultra-high-purity (UHP) CO<sub>2</sub> was purchased from Hangzhou Jingong Materials Co., Ltd. (Hangzhou, Zhejiang, China).

Based on XRF analysis, detailed calcium content in the WCS was calculated. The calcium contents included calcium hydroxide  $(Ca(OH)_2)$  76.38%, CaCO<sub>3</sub> 9.23%, free CaO 19.49%, and calcium sulfate (CaSO<sub>4</sub>) 0.77%, respectively, which are shown in Fig. 1b.

## 2.2. Preparation experiments

The WCS was firstly extracted using 2.0 M (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution, as a recyclable leaching agent. Optimized leaching was conducted at 40 °C with a dosage of 105 g/L, accompanied with continuous stirring for 30 min, and then the solid phase was transferred to a 150 mL micro-column cell for impurity flotation. The purified product was the source of calcium for the preparation of CCNP in the subsequent carbonation process, which was conducted in a double jacketed glass container. Finally, the purified product produced in the leaching process was dissolved in a certain amount of distilled water under stirring at the desired temperature. After 5 min, ammonia solution (25 wt% NH<sub>3</sub>) with additive was added dropwise to the slurry under continuous stirring at 1000 rpm, and CO2 was continuously injected into the reactor at a predetermined flow rate. The CO<sub>2</sub> micropore disperser is made of a sand core with an average pore diameter of about 20 µm. To record calcium conversion during the carbonization process, approximately 10 mL of the slurry was regularly collected every 5 min. The effects of the main critical process parameters including carbonation temperature, dropping rate, the initial ammonia content and CO2 flow rate that affected the carbonation behavior, were investigated systematically. When the pH was below 8, the reaction was terminated, and the mixtures were filtered using filter paper. The filtered precipitate was washed using distilled water and then dried at 65 °C for 24 h and the filtrate ((NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>) was fed back to the



Fig. 1. (a) Particle size distribution, (b) Calcium distribution.

#### Table 1

Chemical components of waste carbide slag (wt%).

Sample	CaO	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	$SO_3$	SrO	Na <sub>2</sub> O	Fe <sub>2</sub> O <sub>3</sub>	MgO	Others
WCS	83.0	3.7	1.31	0.77	0.21	0.19	0.17	0.15	10.5

(2)

 $\times P_{CaSO4 \cdot 2H2O}$ 

 $\left(\frac{0.5814}{2} + 0.4186\right)$ 

leaching step to further reduce fresh  $(NH_4)_2SO_4$  consumption (see Fig. 2).

#### 2.3. Characterization

 $x_{CaSO4 \cdot 2H2O} =$ 

Cu K $\alpha$  radiation PANalytical X-ray diffractometer was used for XRD analysis, and data were collected with the scanning scope (2 $\theta$ ) from 5° to 90° under the speed of 0.01° s<sup>-1</sup> and a counting time of 0.1 s per step. The relative percentage content of each polymorph of CaCO<sub>3</sub> mixtures was calculated from their characteristic XRD peak intensities using the following equation (Jin et al., 2020)(Bo et al., 2019):

$$x_{\nu} = \frac{7.691(I_{\nu}^{110})}{I_{c}^{104} + 7.691(I_{\nu}^{110})}$$
(1)

$$x_c + x_v = 1$$

#### 2.4. Conversion ratio description

Since the purified product of  $CaSO_4 \cdot 2H_2O$  is the carbonation reactant, the calculation method of calcium conversion ratio was similar to that described by Tan et al. (2017) and Zhao et al. (2015). It is known that the weight loss of  $CaCO_3$  caused by  $CO_2$  emission, occurs in the range between 600 °C and 850 °C (B. Wang et al., 2020). The purity of  $CaCO_3$  was calculated by the following Eq. (3) (de Beer et al., 2015):

$$P_{CaCO3} = \frac{\Delta w\% \times M_{CaCO3}}{M_{CO2}}$$
(3)

Calcium conversion  $x_{CaSO4+2H2O}$  was calculated based on calcium carbonate purity *PCaCO3* (Tan et al., 2017). It was calculated by the following Eq. (4):

(4)





Fig. 2. Schematic process route of  $CO_2$  mineral sequestration with recyclable  $(NH_4)_2SO_4$ .

where *M* denotes molecular weight, and  $\Delta w$  represents the weight loss of the sample. The purity of calcium sulfate dihydrate (*PCaSO*<sub>4</sub>•2*H*<sub>2</sub>*O*) was determined by TGA analysis.

#### 2.5. Supersaturation ratio description

According to the classical nucleation theory (CNT), crystallization of  $CaCO_3$  started from nucleation and the initially formed nuclei grew into crystallites (Trushina et al., 2014). The higher nucleation ratios resulted in smaller crystals, which is in accordance with Eq (5) (Trushina et al., 2016):

$$r = 2\gamma \nu / KT lnS \tag{5}$$

where *r* is the radius of critical nucleus,  $\gamma$  is the interfacial free energy,  $\nu$  is the volume of a molecule inside the nucleus and *K* and *T* are Boltzmann constant and absolute temperature, respectively.

Supersaturation ratios (S) in the interface were expressed as in Eq (6) (Fernandez-Martinez et al., 2013):

$$S = \sqrt{\frac{\alpha_{Ca+}\alpha_{CO3}}{K_{sp}}} \tag{6}$$

where  $\alpha_{Ca}$  and  $\alpha_{CO3}$  are the activity of  $Ca^{2+}$  and  $CO_3^{2-}$  ions, respectively at the interface area.  $K_{sp}$  is the solubility product of calcium and carbonate ions, a constant at a fixed temperature.

## 3. Results and discussion

### 3.1. Waste carbide slag leaching

The leaching of WCS in (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution is a non-catalytic liquidsolid reaction. Calcium hydroxide and CaO in WCS are the main components with high leaching reactivity at ambient conditions (Figs. 1b and 3a). As shown in Fig. 3a, calcium components are enriched in



Fig. 3. XRD and TGA analysis of the carbide slag and leaching products (a) XRD (b) TGA.

leaching solid phase, owing to the formation of CaSO<sub>4</sub>·2H<sub>2</sub>O, which is relatively insoluble. Furthermore, CaSO<sub>4</sub>·2H<sub>2</sub>O with a purity of 95.3% was obtained after the separation of the impurities (Fig. 3b). In addition, the effect of temperature, solid-to-solution ratio and (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> concentration on the calcium conversion ratio was fully investigated, and the results are depicted in the Supplementary Material (Fig. S1.).

### 3.2. $CO_2$ mineralization

## 3.2.1. Preparation of the CCNP

The mineralization process was conducted in a three-phase system. The possible mechanism underlying the formation of CCNP can be described as follows: first,  $CO_2$  is diffused from the gas phase to the bulk solution phase, and then carbonate ions are generated probably by the dropwise addition of ammonia solution. Moreover, the dissolution of



Fig. 4. Proposed reaction pathway for the preparation of CCNP by  $\mbox{CO}_2$  mineralization.



Fig. 5. Effect of additives and the dropping rate on the particle size distribution.

CaSO<sub>4</sub>·2H<sub>2</sub>O is strengthened with the change of the pH, and the dissolved Ca<sup>2+</sup> and SO<sub>4</sub><sup>2-</sup> traverse the liquid film into the bulk phase solution. Therefore, high supersaturation of Ca<sup>2+</sup> and CO<sub>3</sub><sup>2-</sup> is achieved in the bulk solution. Based on the CNT, it is advantageous for the nucleation of nanoparticles of amorphous calcium carbonate (ACC), and then the ACC particles crystallize into nano-sized polymorph (vaterite) in the presence of dispersant (see Fig. 4).

The results depicted in Fig. 5 show the correlation between CaCO<sub>3</sub> particle size and additives. The optimized average particle sizes of the prepared CaCO<sub>3</sub> were 108, 500 and 120 nm with the addition of ASDP, glycerol (Gly) and sodium tripolyphosphate (STP), respectively. It was possible that with the addition of Gly, the surface tension decreases, reducing the interfacial free energy  $(\gamma)$ , thereby resulting in the enhancement of nucleation. Nevertheless, an increase in Gly also increased the viscosity of the reaction mixture, slowing down the ion diffusion. Compared with Gly and STP, ASDP is more effective at reducing the CaCO<sub>3</sub> particle size. Zhou et al. (2019)reported that EDTA accelerated the leaching of soluble  $Ca^{2+}$  in the solution, thereby increasing the supersaturation ratio (S) in the interface zone to enhance the nucleation of CaCO<sub>3</sub> particles. Additionally, the presence of STP would prevent the growth of CaCO<sub>3</sub> grains to a certain degree and reinforce the uniformity of the particle size of CaCO<sub>3</sub> microspheres (Yang et al., 2019). This may probably be due to chelation by soluble



Fig. 6. Effect of critical process parameters on the particle size distribution (a) Temperature (b)  $CO_2$  flow rate (c) Mole ratio of  $NH_4^+/Ca^{2+}$ .

Ca<sup>2+</sup>, forming a larger steric hindrance, resulting in the increase of supersaturation in the system. The dropping rate of NH<sub>4</sub>OH solution combined with additives also had a remarkable effect on nucleation and crystallization. Smaller-sized particles, with a size range of 106–112 nm, were obtained at a dropping rate of 10–25 mL/min. The process of calcium sulfate dissolution and Ca<sup>2+</sup> ion diffusion away from the surface of calcium sulfate to the bulk phase was enhanced as the dropping rate increased. Furthermore, the excess ammonia accelerated the transfer rate of CO<sub>2</sub> from the gas phase to the liquid phase, resulting in a higher ions concentration, which leads to a higher nucleation rate.

Figure 6(a-c) shows the PSD of nano-CaCO<sub>3</sub> prepared with an ASDP additive under different conditions. PSD curves show a single peak in the range between 100 nm and 320 nm. Temperature is an important parameter for CaCO<sub>3</sub> crystallization as it allows for the control of the solubility and activity of calcium and carbonate ions (Trushina et al., 2014). The average particle size was increased from 106 to 225 nm with an increase in temperature (Fig. 6a), indicating that a higher temperature might accelerate crystal growth throughout the carbonation stage (Song et al., 2020). The nanoparticles tended to agglomerate and formed clusters of greater sizes when the carbonation temperature was increased; a similar tendency was also reported in a previous study (Jin et al., 2020).

In terms of CO<sub>2</sub> flow rate on particle size (Fig. 6b), the relatively small particle size of CaCO<sub>3</sub> was highly associated with the increase of CO<sub>2</sub> flow rate. The average particle size of CaCO<sub>3</sub> decreased to 106 nm when the CO<sub>2</sub> flow rate was increased from 150 to 500 mL/min. According to the CNT (Du and Amstad, 2020; Wu et al., 2022), the increase of CO<sub>2</sub> flow rate would increase the partial pressure of CO<sub>2</sub> on the interface, resulting in the elevation of the CO<sub>3</sub><sup>2</sup>-concentration and supersaturation in the interface zone, thus contributing to the CaCO<sub>3</sub> nucleation (El-Sheikh et al., 2013). Additionally, a relatively high  $CO_2$  concentration is suitable for transporting more  $CO_2$  across the gas/liquid interface and accelerating  $CO_2$  to diffuse in liquid bulk, increasing  $CO_3^{-1}$  ions concentration, which is beneficial to improve the supersaturation ratio (S) and the formation of nano-sized CaCO<sub>3</sub>.

In addition, the effect of ammonia content (the mole ratio of  $NH_4^+/Ca^{2+}$  in the stoichiometric range of 1.6–2.4) on the PSD is presented in Fig. 6c. It can be seen that carbonation products have a relatively narrow size distribution in the range of 100–180 nm, suggesting that ammonia content has a slight effect on PSD. However, from the reaction kinetics aspect, the dropwise addition of NH<sub>4</sub>OH solution increased the pH stepby-step, continuously increasing the mass transfer rate of CO<sub>2</sub> at an early nucleation stage (Zhang et al., 2020; Zhao et al., 2015). Meanwhile, maintaining a constant dropwise addition of NH<sub>4</sub>OH solution can also compensate the escape of the ammonia.

### 3.2.2. CCNP purity and calcium conversion ratio

Temperature is an important factor that affects carbonation reactions. As shown in Fig. 7a, a relatively high CaCO<sub>3</sub> purity of 92% was obtained at 25 °C, and the purity of CaCO<sub>3</sub> showed a downward trend with the increase in temperature, which may be ascribe to ammonia escape at a higher temperature. The diffusion of CO<sub>2</sub> and solubility of CaSO<sub>4</sub>·2H<sub>2</sub>O were changed at a higher temperature, resulting in more CO<sub>2</sub> dissolution into the mixture solution and more calcium ion dissociation from CaSO<sub>4</sub>·2H<sub>2</sub>O to be involved in the carbonation process (Bo et al., 2019; Zhou et al., 2019). The calcium conversion ratio increased significantly as the temperature increased between 25 and 80 °C within 10 min, which is consistent with the normal chemical reaction. After 15 min, all calcium conversion ratio curves were very close to 90%, regardless of the different reaction temperatures. Notably, the reaction



Fig. 7. Purity and conversion ratio of the products.

equilibrium is achieved within a short time (about 15 min) at room temperature and atmospheric pressure.

As shown in Fig. 7b, the effect of  $\rm NH_4^+/Ca^{2+}$  on the calcium conversion ratio was not particularly significant. Except for the 80% ammonia content, nano-sized CaCO<sub>3</sub> with a purity of >90% was obtained. Meanwhile, nearly 90% of the calcium component in the leaching product was converted to CaCO<sub>3</sub>, which is consistent with previous studies (de Beer et al., 2014; Prajongtat et al., 2019), indicating that ammonia concentration is not the rate control factor. Theoretically, the molar ratio of  $\rm NH_4^+/Ca^{2+}$  is 2.0, but it was noted that a certain amount of ammonia escaped in the form of  $\rm NH_3$  due to the high volatility of ammonia. Furthermore, it was reported that carbonation efficiency significantly increased with the dosage of  $\rm NH_4OH$  solution (Ding et al., 2019a, 2019b). Thus, the ammonia solution should be added slightly in

excess.

The mass transfer in the bubbling reactor proceeds with the bubbles rising and growing, resulting in an increase of the gas-liquid mass-transfer area (Wang et al., 2016). The dissolution of  $CO_2$  in the solution is generally a rate-limiting step in the carbonation process, which can be enhanced significantly by increasing  $CO_2$  concentration (de Beer et al., 2014). The effect of  $CO_2$  flow rate (varying from 150 to 500 mL/min) on the CaCO<sub>3</sub> purity and calcium conversion ratio of the leaching product was investigated. Fig. 7c shows that nearly 90% of CaSO<sub>4</sub>·2H<sub>2</sub>O was converted to CaCO<sub>3</sub> within 10 min when the CO<sub>2</sub> flow rate was 500 mL/min, and the purity of CaCO<sub>3</sub> was up to 93%. However, since the solubility of  $CO_2$  was kept constant under the given conditions, and it is unreasonable to further increase the  $CO_2$  flow rate to achieve a high conversion ratio.

Interestingly, the gradual weight loss in the temperature range of 200–600 °C (blue shade marked) shown in TGA curves can be attributed to the weight loss of strongly bound water in the metastable CaCO<sub>3</sub> (Konrad et al., 2016; Zou et al., 2020). The main weight loss between 600 and 850 °C (red shade marked) can be considered as the decomposition of the stable CaCO<sub>3</sub>. As shown in the histogram, the content of the metastable CaCO<sub>3</sub> is <10%.

## 3.3. Characterization of the CCNP

The polymorphs of solid-phase products were observed and characterized by FTIR spectra and XRD patterns. Some typical bands for CaCO<sub>3</sub> polymorphs have been well reported in the literature (Vagenas et al., 2003). These absorption peaks corresponded to the asymmetric stretching vibration of the C–O band around 1447 cm<sup>-1</sup>, out-of-plane bending of the C-O bond at 877 and 712 cm<sup>-1</sup>, and doubly degenerated in-plane O–C–O deformation bending at 744 and 712 cm<sup>-1</sup> (Sun et al., 2017). In Fig. 8, strong peaks at 744  $\text{cm}^{-1}$  and 1087  $\text{cm}^{-1}$  in the CaCO<sub>3</sub> precipitate were corresponded to vaterite. Meanwhile, the weak spectrum peak at 849 cm<sup>-1</sup> was ascribed to ACC. More importantly, since the carbonates were absolutely dried, the absorption peak of water at 3400 cm<sup>-1</sup> was attributed to the formation of metastable calcium carbonate. Combined with the research results of Pair et al. (Pai and Pillai, 2008) it can be inferred that a certain amount of nanoparticles of ACC is produced at the initial stage of the reaction, which is consistent with the TGA analysis. Additionally, peaks at 877  $\text{cm}^{-1}$  attributed to calcite in the FTIR spectra of carbide slag, gypsum and vaterite/calcite were found, which might be due to the storage of the WCS in the open air, causing the formation of CaCO<sub>3</sub> by natural carbonation. Besides, the observed bands at 1146, 1116, 669 and 602  $\text{cm}^{-1}$  in the FTIR spectrum of gypsum were characteristic of those reported in the literature for the stretching and bending modes of sulfate (Böke et al., 2004). The stretching vibrations occurring at 3550, 3400, and 1688  $\text{cm}^{-1}$  were attributed to the bending movements of crystal water (Wang et al., 2017). Thus, it can be concluded that the leaching product (gypsum) contains a certain amount of CaSO<sub>4</sub>·2H<sub>2</sub>O.

The FTIR results were further supported by XRD analysis. All diffraction peaks in leaching product were associated with CaSO<sub>4</sub>·2H<sub>2</sub>O except for a very slight diffraction peak associated with calcite (104) at  $2\theta = 29.4^{\circ}$  (Fig. 9). In addition, both calcite and vaterite were detected in carbonation products, which was in agreement with the FTIR results. The content of vaterite and calcite was 82.19% and 17.81%, respectively, as calculated by Eqs. (1) and (2), indicating that the main crystalline phase was vaterite, and calcite might be attributed to the





Fig. 9. X-ray patterns of the solid products.

unreacted CaCO3 in the WCS.

SEM images show the morphological evolution of calcium components during the leaching and carbonation process (Fig. 10). The leaching product (CaSO<sub>4</sub>·2H<sub>2</sub>O) exhibits a smooth surface, and presents a rod-like shape with a length of up to 10  $\mu$ m (Fig. 10B). In addition, we can see that CaCO<sub>3</sub> formed on the surface of the CaSO<sub>4</sub>·2H<sub>2</sub>O particles. Combined with XRD analysis, Fig. 10A-E demonstrates that the calcium component undergoes multiple crystalline phase transformations of dissolution and recrystallization to finally form a nano-sized CaCO<sub>3</sub> in the presence of a dispersant. Morphologically, the CaCO<sub>3</sub> particle appears spherical with little aggregation (Fig. 10F). In addition, rhombic calcite was locally observed (Fig. 10F), which may be due to the unreacted CaCO<sub>3</sub> in feedstocks.

Fig. 11 shows the nitrogen adsorption-desorption curves for CaCO<sub>3</sub>, synthesized at 25 °C, the CO<sub>2</sub> flow rate of 500 mL/min and NH<sub>4</sub><sup>+</sup>/Ca<sup>2+</sup> of 2.4 with the addition of ASDP at a dropping rate of 10.0 mL/min. Based on the Barrett-Joyner-Halenda (BJH) analysis, the average pore diameter and Brunauer-Emmett-Teller (BET) surface area of carbonated products were about 7.86 nm and 29.31 m<sup>2</sup>/g, respectively, which was fully consistent with the application range of composite materials such as plastics, paint, ink, rubber and paper. Furthermore, the average particle size was 204.7 nm, which corresponds to the results of laser particle size analysis.

### 3.4. Evaluation of CO<sub>2</sub> uptake and industrial feasibility

Similar to estimates of the CO<sub>2</sub> consumption in mortars and concrete suggested by Steinour (Chang et al., 2012), the theoretical CO<sub>2</sub> captured capacity of the WCS was calculated using Eq. (7), based on XRF data in Table 1. Thus, the theoretical CO<sub>2</sub> uptake ratio of WCS ( $\xi_{co_2}^{Th C}$ ) was 62.88%. Additionally, the content of CaSO<sub>4</sub>·2H<sub>2</sub>O in the leaching product (gypsum) involved in the carbonation reaction was nearly 95%. Therefore, the theoretical CO<sub>2</sub> uptake ratio of gypsum ( $\xi_{co_2}^{Th G}$ ) was 26.93%, which can be expressed as the ratio of the molecular weight of CO<sub>2</sub> and CaSO<sub>4</sub>·2H<sub>2</sub>O.

Considering the conversion ratio of leaching and carbonization process, the actual  $CO_2$  uptake ratio of gypsum and the raw material (WCS) at different times was estimated using Eqs. (8) and (9).

$$\xi_{co_2}^{Th} = 0.785 \left( w_{\text{CaO}} - 0.56 w_{\text{CaCO}_3} \right) \tag{7}$$

$$\xi_{co2}^{Gyp} = \xi_{co2}^{Th \ G} \cdot x_{CaSO4 \cdot 2H2O} \tag{8}$$

$$\xi_{co_{2}}^{WCS} = \xi_{co_{2}}^{Th C} \cdot x_{CaSO4 \cdot 2H2O} \cdot x_{CaO}$$
<sup>(9)</sup>



Fig. 10. SEM images of the raw materials and products (A) waste carbide slag (B) leaching product (C) carbonation for 5 min (D-E) carbonation for 15 min.



Fig. 11. Nitrogen adsorption-desorption curves for calcium carbonate.



Fig. 12. Evaluation of  $CO_2$  uptake of waste carbide slag and the leaching product.

where  $w_{CaO}$  and  $w_{CaCO3}$  are the content of CaO and CaCO<sub>3</sub> in WCS,  $x_{CaSO4.2H2O}$  and  $x_{CaO}$  are the calcium conversion ratio in carbonation and leaching process, respectively.

After 15 min, the actual  $CO_2$  uptake ratio of gypsum and WCS is 23.1% and 50.8%, respectively (Fig. 12), which are relatively considerable. Based on the above analyses, the coordinated treatment of WCS

and  $CO_2$  capture to produce  $CaCO_3$  is economically promising. The treatment of 1.0 ton of WCS consumes ~0.509 tons of  $CO_2$  and yields about 1.15 tons of nano-CaCO<sub>3</sub>. In addition, a comparison of nano-sized  $CaCO_3$  performance obtained from this study with those of commercial  $CaCO_3$  for rubber and plastics is given in Table S1. The index basically meets the requirements of third-grade industrial fine CaCO<sub>3</sub>. It is indicated that the CaCO<sub>3</sub> obtained from this study can be well used as a filler material for rubber and plastics products. The conversion of WCS to commercial-grade products not only saves the WCS disposal costs but also eliminates economic as well as potential environmental challenges caused by long-term storage of WCS.

# 4. Conclusions

In this paper, we have proposed a new approach for preparing highvalue CCNP through carbonation. The WCS was firstly leached using (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution. After impurity separation, a nano-sized CaCO<sub>3</sub> was prepared through a dropwise carbonation process. Vaterite crystals with a purity of about 93% and an average size of 106 nm were produced in the presence of ASDP at a dropping rate of 10.0 mL/min under the conditions of 25 °C, CO<sub>2</sub> flow rate of 500 mL/min, and NH<sub>4</sub><sup>+</sup>/Ca<sup>2+</sup> of 2.4. Additionally, the ability of WCS to capture CO<sub>2</sub> demonstrated that treatment with 1 ton of WCS consumed ~0.5 tons of CO<sub>2</sub>, indicating that the stepwise mild mineralization route was a promising strategy for large-scale CO<sub>2</sub> sequestration. Furthermore, the leaching agent, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub> solution, was recyclable which reduces the capital cost. The approach proposed in this work is useful in the treatment of WCS waste, while simultaneously achieving CO<sub>2</sub> mitigation.

#### CRediT authorship contribution statement

Wenxiu Li: Investigation, Formal analysis, Conceptualization, Methodology, Software, Data curation, Writing – original draft, Writing – review & editing. Yan Huang: Resources, Writing – review & editing. Tao Wang: Project administration, Supervision, Visualization, Writing – review & editing. Mengxiang Fang: Resources, Visualization, Writing – review & editing. Yan Li: <style name=>Resources and Editing</ style>.

# 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.

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## Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2022.132463.

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