Preparation of High-Purity Calcium Carbonate by Mineral Carbonation Using Concrete Sludge

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ABSTRACT: A new type of mineral carbonation process for concrete sludge, a waste of fresh concrete under hydration, was developed, and the carbonation performances of the process were examined by laboratory-scale experiments. The process is composed of two steps; filtration of concrete sludge and bubbling of CO_2 into the filtrate to form calcium carbonate. Model concrete sludge, a mixture of cement and water, was filtered through a cellulose filter after hydration for 24 h to obtain a solution containing dissolved calcium ions. Then, the model flue gas containing CO_2 (10%) was bubbled through the filtrate solution, and calcium carbonate was precipitated by the carbonation reaction. About 3% of calcium in the concrete sludge could be extracted into the filtrate in a single filtration step, and more than

95% of dissolved calcium was recovered as calcium carbonate by the bubbling of CO_2. The obtained calcium carbonate was calcite with a high purity (>95%) and 5−10 μm. The solid residue (concrete sludge) after filtration was mixed with fresh water and filtered through a cellulose filter. Then, the model flue gas was bubbled into the filtrate solution for carbonation. This filtration−bubbling step was repeated 5 times, and 10.8% of calcium in the feed cement was extracted into the filtrates in total. More than 95% of the extracted calcium could be recovered as calcium carbonate with high purity (>97%), and the overall conversion of calcium in the feed cement to calcium carbonate was 10.1%. The purity of calcium carbonate and the calcium conversion were much higher than those for the direct bubbling method, where the model flue gas is bubbled into concrete sludge.
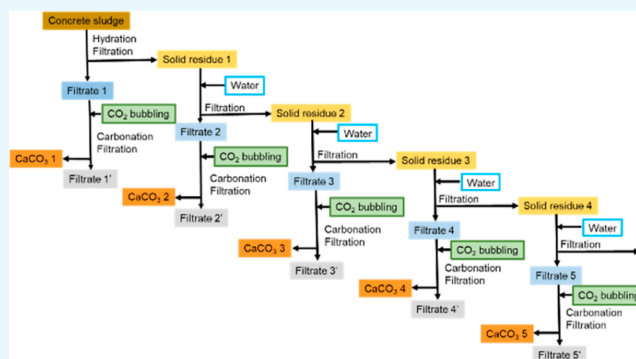
1. INTRODUCTION

An accelerated mineral carbonation process using alkaline solid waste has been recognized as one of the promising technologies to reduce the net emission of CO_2 as well as waste valorization. The global potential of CO_2 emission reduction is estimated to be 4.02 Gt/year, which corresponds to about 13% of the global energy-related CO_2 emission (33 Gt-CO_2).[1] A number of studies have been conducted on the mineral carbonation of solid alkaline wastes including iron/steel slags,[2−4] fly ash,[5−7] mining wastes,[8,9] and concrete/cement wastes.[10−15] Among them, the mineralization of waste cement/concrete has a huge potential for CO_2 emission reduction, estimated at about 50 Mt-CO_2/year for direct reduction and about 2 Gt-CO_2 for indirect reduction due to utilization of products.[1] In addition, the carbonation of waste concrete/cement is a measure for stabilization through the neutralization of alkaline wastes.[10]

Two types of waste concrete/cement can be used as a calcium source for mineral carbonation: waste concrete from demolished buildings[11−16] and fresh concrete waste.[17−20] The former waste concrete is fully hydrated and less reactive with CO_2. The carbonation of such waste concrete should require an acceleration method such as using acids,[15,21,22] chemicals,[23]

high pressure, or high temperature conditions.[15,20] On the other hand, fresh concrete waste or concrete sludge is under-hydrated and highly reactive with CO_2, and it can be carbonated by direct contact with CO_2 gas even under ambient conditions.[10,17,24] In our previous study,[25] we reported a carbonation process of model concrete sludge with direct bubbling of CO_2-containing gas into model concrete sludge. The carbonation reaction of model concrete sludge was found to proceed quickly, and dissolved calcium ions from hydrated cement (as calcium hydroxide) can be almost completely transformed and precipitated as calcium carbonate. The product of calcium carbonate can be used as a raw material for several industries, which may indirectly reduce the emission of CO_2 by the reduction of mining of fresh limestone.[1] However, the calcium carbonate obtained by the
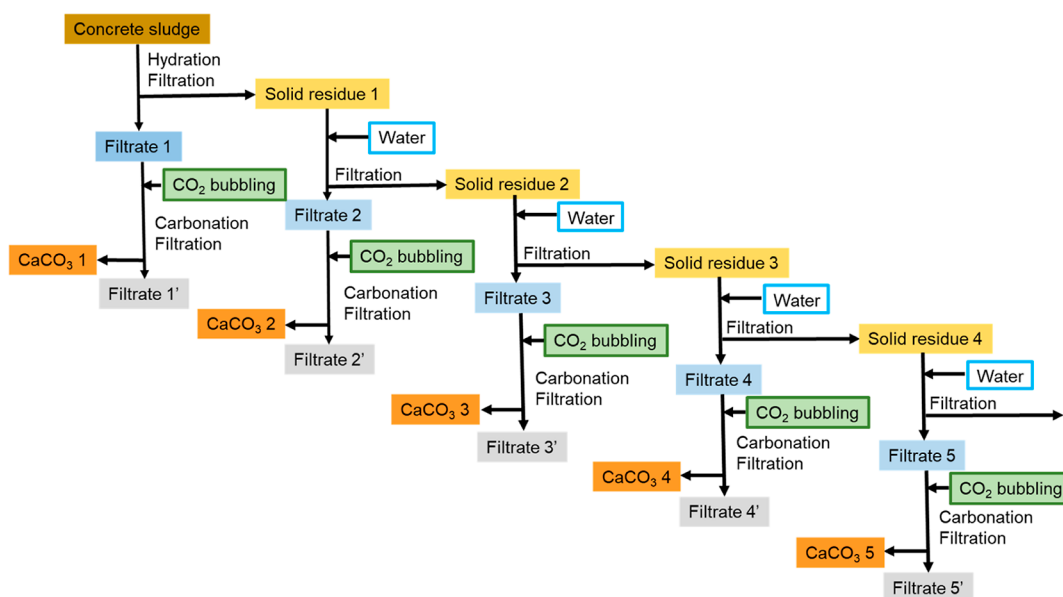
Figure 1. Schematic of the mineral carbonation process using concrete sludge coupled with filtration.

direct bubbling method is mixed with hydrated cement, which is composed of various calcium−silica−hydrate (C−S−H) compounds, and the purity of calcium carbonate in the solid product is 6.5 wt %. For the industrial uses of calcium carbonate as a substitute of limestone, the purity should be higher than 95%.

We here propose a new type of mineral carbonation process for concrete sludge comprising filtration and bubbling to obtain high-purity calcium carbonate. We examined the carbonation performances of the new method and compared the performances and the properties of calcium carbonate with the direct bubbling method. The principle of the present method is schematically shown in Figure 1. Concrete sludge is unused and returned concrete from construction sites. Normally, concrete sludge is almost fully hydrated because it takes a long time (−10 h) to form after being freshly made at a concrete-making industry. In the process, concrete sludge is filtered, the flue gas containing CO_2 is bubbled into the filtrate, and calcium carbonate is precipitated as a product. The solid residue of the concrete sludge is then mixed with fresh water and filtered again. The flue gas containing CO_2 was bubbled into the filtrate to form calcium carbonate. The residue is then mixed with fresh water and filtered, and so on. This filtration−bubbling operation is performed several times, and calcium carbonate is obtained in each step. By filtration, the dissolved calcium ions from the concrete sludge can be separated from insoluble hydrated cement components, and the high-purity calcium carbonate can be obtained by bubbling compared with the one obtained by the direct bubbling method.[25] In addition, repeated filtration of the hydrated cement, the higher conversion of calcium, and consequently the fixation ratio of CO_2 can be expected by this method.

In this study, we examined the performances of the proposed mineral carbonation process based on the experimental results on a laboratory scale. The calcium leaching behaviors such as the extraction ratio of calcium in the cement as well as the extraction of impurities were investigated for the filtration step. In the bubbling step, we studied the conversion of leached calcium ions and CO_2 into calcium carbonate and the characteristics of the calcium carbonate such as purity, size,

and crystalline structure. The results of the carbonation performances were compared with those for the conventional method of carbonation of concrete sludge by the direct carbonation method.[25]

2. MATERIAL AND METHODS

Model concrete sludge was prepared by mixing commercially available Portland cement (Taiheiyo Cement Co., Japan) and deionized water with the weight ratio of solid/water at 0.05. The mixture was placed in a plastic bottle and stirred for 24 h on a roller mixer (40 rpm) to be fully hydrated.[25] The elemental composition of Portland cement is 46.4% Ca, 10.2% Si, 1.1% Fe, and 1.4% Al. The model concrete sludge after hydration for 24 h was filtered through a cellulose filter with a pore diameter of 20−25 μm. The filtration was completed within 1 h. The pH and the chemical composition of the filtrate were measured using a pH meter (HORIBA, Japan) and an inductively coupled plasma atomic emission spectroscope (Thermo Fisher Scientific, ICPA-6000, USA), respectively.

The model flue gas containing 10% CO_2 balanced with nitrogen (total pressure = 1.0 atm) was bubbled through the filtrate solution. Figure 2 shows the experimental apparatus for bubbling CO_2 into the filtrate. The flow rate of the model flue gas was 100 mL/min. On completion of the CO_2 addition, the
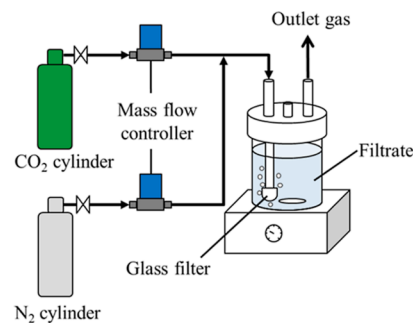
Figure 2. Schematic of the experimental apparatus for CO_2 bubbling.

mixture was taken out from the vessel and filtered. The solid residue was then dried in an oven at 100 °C for 24 h. The crystal structure of the solid residue was analyzed using an X-ray diffractometer (Rigaku, Ultima IV, Japan). The microstructure of the solid was observed using a scanning electron microscope (JEOL, JCM-7000 NeoScope, Japan). The calcium carbonate content was determined with a thermogravimetric analyzer (Shimadzu, DTG-60H, Japan) based on the weight decrease between 600 and 900 °C.

The solid residue of the filtration of the model concrete sludge and deionized water was mixed at the weight ratio of solid/water = 0.05, and the mixture was stirred with a roller mixer at 40 rpm for 1 h. The mixture was filtered with a cellulose filter. A model flue gas containing 10% CO_2 balanced with nitrogen was bubbled into each filtrate with the total flow rate at 100 mL/min and the total pressure at 1.0 atm. The time changes of pH and calcium concentration were measured using a pH meter and an inductively coupled plasma atomic emission spectroscope. All experiments were carried out at room temperature and atmospheric pressure. This filtration–bubbling step was repeated five times.

The CO_2 fixation performances of the process were evaluated by the following indices: extent of calcium extraction, calcium conversion, calcium recovery ratio, and purity of calcium carbonate defined as follows.

Extraction ratio of calcium [%]

$$= \frac{\text{mass of calcium leached in filtrate [g]}}{\text{mass of calcium in feed cement [g]}} \times 100 \, [\%] \tag{1}$$

Overall conversion of calcium [%]

$$= \frac{\text{mass of calcium in precipitated calcium carbonate [g]}}{\text{mass of calcium in feed cement [g]}} \times 100 \, [\%] \tag{2}$$

Recovery ratio of calcium [%]

$$= \frac{\text{mass of calcium in precipitated calcium carbonate [g]}}{\text{mass of calcium dissolved in filtrate [g]}} \times 100 \, [\%] \tag{3}$$

Purity of calcium carbonate [%]

$$= \frac{\text{mass of calcium carbonate in precipitated solid [g]}}{\text{mass of precipitated solid [g]}} \times 100 \, [\%] \tag{4}$$

3. RESULTS AND DISCUSSION

3.1. Calcium Extraction from Cement. Figure 3 shows the calcium concentration and pH of the filtrate for each filtration step. The calcium concentration in the first filtrate was 934 mg-Ca/L, and it decreased step by step to reach 306 mg/L in the fifth filtrate. The pH of filtrate decreased from 12.44 (first) to 11.98 (fifth). Impurities such as silicon, aluminum, and iron were detected in the filtrate, the concentrations of which were 5.78, 0.34, and 0.02 mg/L, respectively, for the first filtrate, and the fraction of the calcium ion was about 99%. Figure 4 shows the cumulative extraction ratio of calcium against the total volume of water used. The extraction ratio of calcium increased with the filtration step,
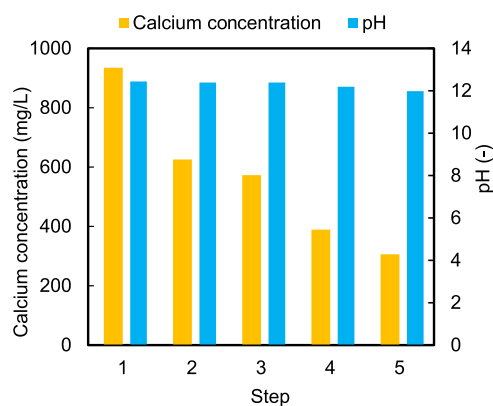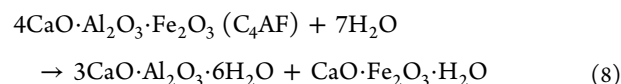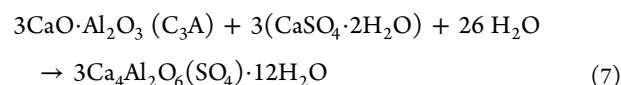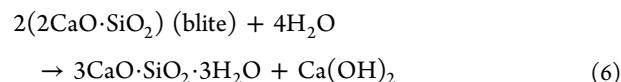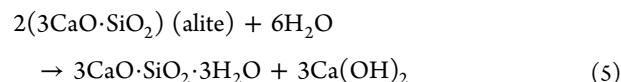

Figure 3. Calcium concentration and pH of the filtrate from each filtration step.
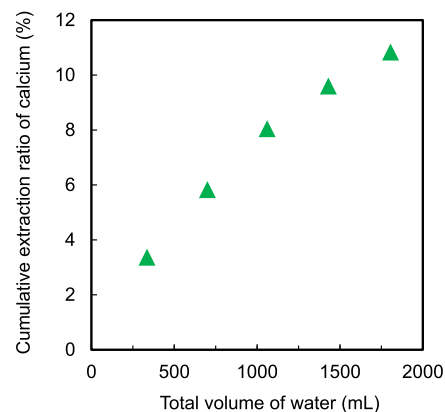
Figure 4. Cumulative extraction ratio of calcium against water used.

and 10.8% of calcium in the feed cement was extracted after five filtration steps.

The dissolution of calcium into water can be attributed to calcium hydroxide generated by the hydration reactions of cement. The hydration reactions of cement components are as follows[26,27]

$$2(3CaO \cdot SiO_2) \, (\text{alite}) + 6H_2O$$
$$\rightarrow 3CaO \cdot SiO_2 \cdot 3H_2O + 3Ca(OH)_2 \tag{5}$$

$$2(2CaO \cdot SiO_2) \, (\text{blite}) + 4H_2O$$
$$\rightarrow 3CaO \cdot SiO_2 \cdot 3H_2O + Ca(OH)_2 \tag{6}$$

$$3CaO \cdot Al_2O_3 \, (C_3A) + 3(CaSO_4 \cdot 2H_2O) + 26 \, H_2O$$
$$\rightarrow 3Ca_4Al_2O_6(SO_4) \cdot 12H_2O \tag{7}$$

$$4CaO \cdot Al_2O_3 \cdot Fe_2O_3 \, (C_4AF) + 7H_2O$$
$$\rightarrow 3CaO \cdot Al_2O_3 \cdot 6H_2O + CaO \cdot Fe_2O_3 \cdot H_2O \tag{8}$$

The products of hydration reactions 5−8 are almost insoluble in water except for calcium hydroxide, $Ca(OH)_2$. The solubility of calcium in water is 919 mg-Ca/L based on the solubility of calcium hydroxide (0.17 g/L at 20 °C), and the equilibrium pH of the saturated solution is 12.19 based on the solubility product of calcium hydroxide [$K_{sp} = 5.50 \times 10^{-6}$ $(\text{mol/L})^3$, at 25 °C]. The observed calcium concentration for the first step of filtration was 934.6 mg/L and the pH was

12.44, both of which agreed well with the calculated values. The calcium concentration and pH in the subsequent filtration step decreased. The gradual decrease in the calcium concentration with increasing filtration step would be due to the gradual depletion of the hydrated calcium hydroxide in the feed cement.

3.2. Precipitation of Calcium Carbonate by CO_2 Bubbling. Figure 5 shows the time changes in the calcium
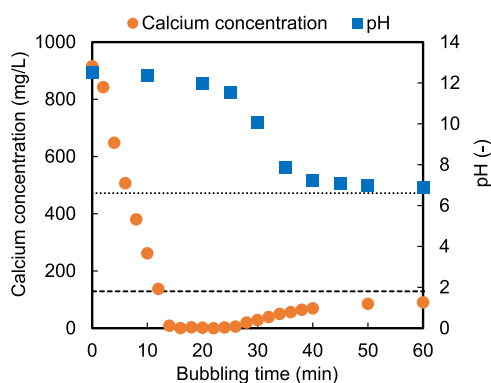

Figure 5. Changes in the calcium concentration and pH with time during the bubbling of CO_2 through the first filtrate (S/L ratio: 0.05 g/g; CO_2 concentration: 10%; gas flow rate: 100 mL/min, 1.0 atm). The dotted and dashed lines are the equilibrium pH and calcium concentration, respectively.

concentration and pH by CO_2 bubbling into the first filtrate. The calcium concentration decreased rapidly, and the precipitation of white solid particles was observed just after bubbling started. The calcium concentration decreased to almost 0 mg/L (below the detection limit) after 22 min after bubbling started, and after that, the calcium concentration increased to reach 90 mg/L after 60 min. The pH changed gradually from 12.5 (initial) to 11.5 (25 min) at the initial stage, and then it sharply decreased to pH 6.9 after 60 min. The equilibrium concentration of calcium ions and pH under $pCO_2 = 0.1$ atm are, respectively, 129 mg/L and 6.61, based on the thermodynamic analysis, which are shown by the dotted line and the dashed line in Figure 5.[25] The observed calcium concentration decreased below the equilibrium concentration after 12 min, reached the minimum value at 22 min, and gradually approached the equilibrium value. The observed pH monotonously approached the equilibrium value, which the observed pH reached after 60 min. The discrepancy of the calcium concentration between the observed value and the equilibrium value can be explained by the oversaturation of the calcium carbonate solution at the earlier stage.[25]

From the present results, the maximum conversion of calcium into calcium carbonate can be achieved when the bubbling is stopped at about pH = 11.5. Therefore, CO_2 bubbling for the subsequent filtrates was stopped when the pH reached 11.5.

Figure 6 shows the cumulative conversion of calcium in the feed cement to calcium carbonate by bubbling. The calcium conversions increased with the increasing filtration−bubbling step almost linearly with a slight leveling off. The total conversions of calcium after the fifth step was 10.15%, which is more than double of the calcium conversion for the direct bubbling method at 4.8%.[25]

Figure 7 shows the recovery ratio of calcium as calcium carbonate by bubbling against the calcium content in the
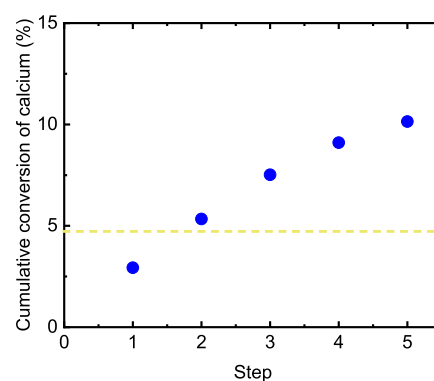

Figure 6. Cumulative conversion of calcium by bubbling. The dotted line indicates the conversion for the direct bubbling method into concrete sludge.
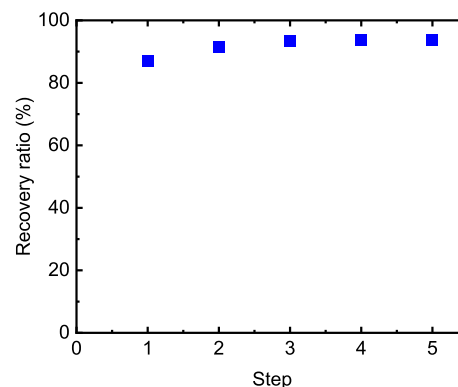
Figure 7. Recovery ratio of calcium in the filtrate to calcium in calcium carbonate.

filtrate for each step. The recovery ratio was 87% for the first filtrate and higher than 90% for all the steps after that. These results indicate that almost all the calcium ions extracted into the filtrate can be recovered by bubbling, except for the dissolved calcium ions corresponding to the solubility of calcium carbonate (129 mg/L for 0.1 atm CO_2).

Figure 8 shows the purity of calcium carbonate obtained in each filtration−bubbling step. The purity was lowest at 95.1% for the first step and the highest at 98.7% at the fifth step. This is because the major portions of the impurities in hydrated cement were extracted in the early stage of the filtration. The
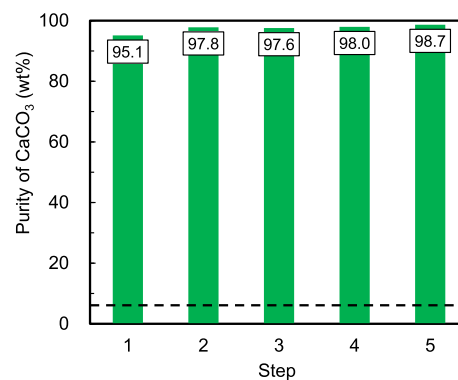
Figure 8. Purity of calcium carbonate from each filtrate achieved by bubbling of CO_2. The dashed line shows the result achieved by direct bubbling of CO_2 into the model concrete sludge.

purity of the obtained calcium carbonate was much higher than the one obtained for the direct bubbling method at 6.5 wt %.

Figure 9 shows XRD patterns of the filtered solids from each step. Peaks assigned to calcite are observed, which is the most
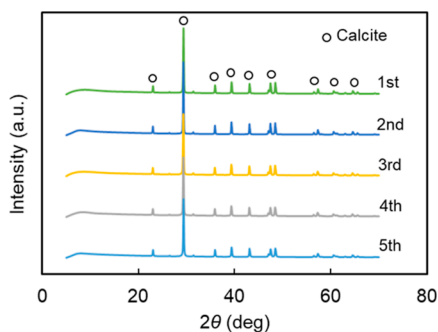

Figure 9. XRD patterns of solid residues of each filtrate after addition of CO_2.

stable crystalline form of calcium carbonate under the temperature condition of room temperature and atmospheric pressure. Figure 10 shows an SEM image of the precipitated
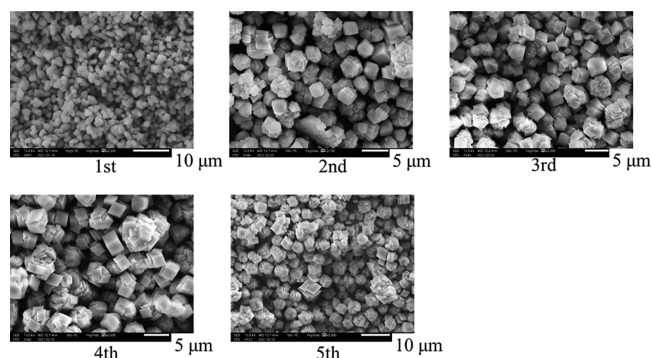

Figure 10. Scanning electron micrographs of solids precipitated by bubbling of CO_2 into each filtrate.

calcium carbonate. The crystalline size of the particles are in the range of 2−5 μm, which is categorized as the precipitated calcium carbonate and can be used for a wide range of industrial applications such as filler in plastics, paper, and an additive in food.

3.3. Process Evaluation. The purity of the calcium carbonate obtained with the present method is much higher (>95%) than the one obtained by the direct bubbling method (6.5%). The overall conversion of calcium in the feed cement to calcium carbonate was much higher (10.1%) for the present method, which is much higher than the one for the direct bubbling method (4.8%). By coupling of the simple filtration step with the bubbling of CO_2 gas, both the conversion of calcium and the purity of the product calcium carbonate were significantly improved. The filtration−bubbling process is composed of two separate operations, and the capital cost and the operation cost should be higher than the direct-bubbling process. Excess amount of water is required for the present process, but the water used for each step can be recovered and reused after filtration. From the viewpoint of cost, the product with high purity (>97%) has a wide range of applications, and the higher capital and operation cost could be compensated for by selling calcium carbonate at higher prices.

From the viewpoint of power consumption, the carbonation reaction by CO_2 bubbling requires no power supply because the reaction can proceed under atmospheric pressure and ambient temperature conditions. No additional chemicals are required for the acceleration of carbonation reaction. Thus, the net CO_2 emission reduction can be achieved when the process is applied to the anthropogenic CO_2 emission sources such as power plants and cement industries. However, when the flue gas contains SOx from the combustion of sulfur-containing fuels, the purity of calcium carbonate should be reduced due to the formation and contamination of calcium sulfate. In such cases, the desulfurization process before the CO_2 fixation should be necessary to obtain high-purity calcium carbonate.

This process can be applied for a variety of emission sources of fresh concrete sludge. In Japan, about 1% of fresh concrete is disposed of as concrete sludge.[28] The annual fresh concrete production in Japan is about 100 million m³, and the annual emission rate of concrete sludge can be estimated to be 5 million metric tons. The solid content in the concrete sludge is about 70%, and the potential amount of CO_2 fixation by the concrete sludge can be estimated to be 0.35 million metric tons based on 10% of the calcium content in concrete sludge.

4. CONCLUSIONS

The carbonation of concrete sludge by bubbling of CO_2-containing gas into a simply filtrated solution resulted in the formation of high-purity calcium carbonate (>95%). The conversion of calcium in the cement components in the concrete sludge increased with increasing the steps of filtration−bubbling. After five steps of filtration−bubbling, the conversion of calcium reached about double that of the direct bubbling method. The higher conversion of calcium means the higher conversion of CO_2. Thus, the performances of the present method are highly improved compared with the process of the direct bubbling method. This method can be applied for emission sources of CO_2 and concrete sludge.

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Notes

The authors declare no competing financial interest.

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