Proceedings of the 21st ICSMGE, Vienna, Austria, 14 – 19 June 2026. Pistrol, Adam & Schweiger (eds.)
Published by: ÖGG, Austrian Society for Geomechanics, Salzburg, Austria, ISBN 978-3-9503898-4-5
https://doi.org/10.53243/ICSMGE2026-2394

Experimental study on the ion concentration transitions in pore water of soot and dust specimens during CO_2 fixation reaction

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ABSTRACT: Industrial by-products are considered to be used to reduce net carbon dioxide (CO_2) emissions because of their CO_2 fixation capability. Quantitative and qualitative methods to evaluate the CO_2 fixation reaction have been studied, but few studies investigate the reactions occurring in the three phases (solid, liquid and gas) and its relationship with the CO_2 fixation. In order to investigate these reactions, the constant flow aeration type CO_2 fixation test was conducted for soot and dust (FC_K) changing testing period and the specimen was taken out at different times. The specimen was divided into half perpendicular to the air flow direction and for the liquid-solid phase water content, eluted calcium (Ca) amount and pH were measured, and for the solid phase the amount of CO_2 fixated amount was measured. The ions concentration in the pore water, which are considered to be involved in the CO_2 fixation reaction (e.g. carbonate ion (CO_3^{2-}), hydroxide ion (OH^-)), was calculated by chemical equilibrium using pH and Ca ion concentrations, which may represent the liquid phase reaction. It was found that the calcium ion (Ca^{2+}) concentrations rapidly decrease at the early stages, in which the minimum value was recorded at 12 h for the inlet side, and at 40 h at the outlet side. Additionally, when the Ca^{2+} concentrations were near the minimum, it was found that maximum values were reached for CO_3^{2-} concentrations, OH^- concentrations rapidly decreased and the increase rate of the amount of CO_2 fixated decreased. From the above findings, the ion concentration transition used in a new model which incorporates the mass balance and reaction rates in the three phases was proposed to evaluate the CO_2 fixation reaction.

KEYWORDS: CO_2 fixation, carbonation, industrial by-products, equilibrium, ion concentration

1 INTRODUCTION

To reduce net carbon dioxide (CO_2) emissions, the CO_2 fixation reaction, in which calcium carbonate ($CaCO_3$) is generated by CO_2 in the atmosphere air and Calcium ion (Ca^{2+}) leached from the particle, is considered. This reaction is one of the adaptation measures of global warming that can be implemented with low energy and low cost because the generated $CaCO_3$ is thermodynamically stable, and this reaction can occur at room temperature and atmosphere pressure. The target materials for this reaction include soot and dust, and steel slag. In earlier studies (Umino, *et al.*, 2017; Komine, *et al.*, 2023; Yokoi, *et al.*, 2023), the capabilities of these materials were confirmed with the constant flow aeration type CO_2 fixation test. Figure 1 shows the apparatus of the constant flow aeration type CO_2 fixation test. In this test, air flows through the sample at a constant flow rate while the CO_2 concentrations of inlet and outlet side are measured at each minute. From the result of this test, the change in the amount of CO_2 fixed by the sample over time is obtained, and the amount of CO_2 fixed can be calculated.

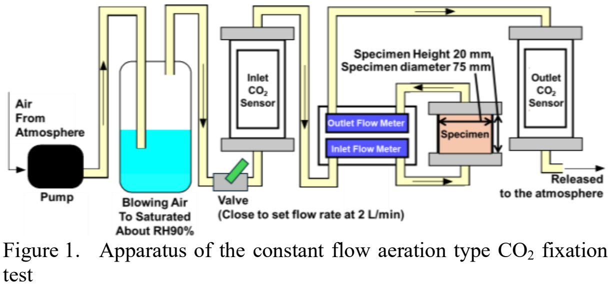


Figure 1. Apparatus of the constant flow aeration type CO_2 fixation test

In authors' former research, from evaluating the effect for CO_2 fixation reactions and fixation amounts by air pressure, the scatters of the reaction time were suggested to be increased in two stages (Kawabe, *et al.*, 2024a). In addition, the constant flow aeration type CO_2 fixation test using soot and dust mixed with a phenolphthalein solution was conducted, and color change of specimen were confirmed (Kawabe, *et al.*, 2024b). However, the cause of these scattering and color change have not been explained, and it is necessary to clarify the mechanism of CO_2 fixation reaction in more detail.

Then, a new model named Three-Phase Element Model is created by the author for evaluating CO_2 fixation reaction. Figure 2 is a conceptual illustration of this model. This model considers the ion concentration, mass balance, and reaction rate in the three phases of gas, liquid, and solid in the microelements in the aeration direction in the constant flow aeration type CO_2 fixation test. Using this model, it is possible to interpret reaction details such as the type of reaction, the substances reacted, the reaction progress, and the amount of products in each part of the specimen.

In this study, the constant flow aeration type CO_2 fixation tests were conducted for soot and dust, which is a type of industrial by-products, with various experimental durations. After that, specimens were taken out and divided into two parts. Then, water content, pH, CO_2 fixation amount, and Ca^{2+} concentration of these two parts were measured. By these results, the concentration of the hydrogen ion (H^+), the hydroxide ion (OH^-), the carbonate ion (CO_3^{2-}), hydrogen carbonate (HCO_3^-), and carbonic acid (H_2CO_3), which were considered the factors affecting the CO_2 fixation reaction, were calculated. Finally, the mechanism of CO_2 fixation ration was explained by Three-Phase Element Model.

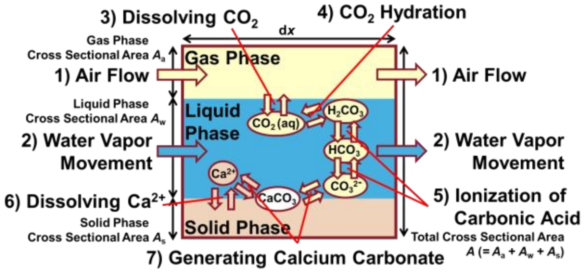


Figure 2. Conceptual illustration of the Three-Phase Element Model

2 FUNDAMENTAL PROPERTIES AND CO_2 FIXATION CAPABILITY OF SAMPLE

In this paper, a type of soot and dust named FC_K, which is a type of industrial by-products, was used. This ash is generated when waste materials (food packing bags, which include cellulose, polypropylene, and polyethylene) and wastewater treatment sludges (organic sludge and inorganic sludge) are incinerated in an incinerator with Refuse derived Paper and Plastics densified Fuel (RPF) as fixed fuel, and this ash is chelated with agent whose main component is potassium diethyldithiocarbamate. The sample was sieved to remove particles larger than 2 mm. Figure 3 is the appearance of the sample. The fundamental properties of the sample were measured and shown in Table 1.

Figure 3. Appearance of FC_K

Table 1. Fundamental properties of the sample, soot and dust FC_K

Water Content during Storage [%]	16.6
Particle Density [Mg/m^3]	2.84
Ca Content from XRF [mass%]	29.0
pH during Storage [-]	12.5

The CO_2 fixation capability of the sample was evaluated by the constant flow aeration type CO_2 fixation test mentioned in chapter 1 and shown in Figure 1. Table 2 is the test condition. In this paper, the distilled water was added to adjust the water content to 30%. Then, the sample was put into the column with an inner diameter of 75 mm and a height of 20 mm, and aerated with the air saturated to approximately relative humidity (RH) 90% at 2 L/min. The amount of CO_2 fixation was calculated from mass balance method (Umino, *et al*., 2017; Kawabe, *et al*.; 2024c) and carbonate content test method (Fukue, *et al*., 2000; Umino, *et al*., 2017; Kawabe, *et al*., 2024c). The mineral composition was determined by X-Ray Diffraction (XRD). The amount of cation leached was determined by Inductivity Coupled Plasma Optical Emission Spectrometer (ICP-OES) using test solutions prepared based on the Environmental Agency Notification No. 46 Leaching Test in Heisei 3rd.

Table 2. Test Condition of the CO_2 Fixation Test

Flow Meter [L/min]	2
Specimen Diameter [mm]	75
Specimen Height [mm]	20
Dry Density [Mg/m^3]	0.53
Void Ratio [-]	4.36

Table 3 is the result of the CO_2 fixation capability of the sample. Where, the fixation time means the elapsed time when the amount of inlet and outlet CO_2 substance became equal first time. The amount of CO_2 fixation by the mass balance method was calculated from the mass balance until the fixation time and dry mass before the CO_2 fixation test. As a result, the treated sample was suggested to be able to fix approximately 15% CO_2 based on the dry mass before the CO_2 fixation test. The result in the carbonate content test, which means the amount of CO_2 fixation base on the dry mass after the CO_2 fixation test, was approximately 12%. In addition, comparing before and after the CO_2 fixation test that pH decreased, calcium hydroxide ($Ca(OH)_2$) was consumed, and $CaCO_3$ was produced was confirmed. The leached amount of sodium ion (Na^+), magnesium ion (Mg^{2+}), potassium ion (K^+), and calcium ion (Ca^{2+}) were confirmed both before and after the test except Mg before the test, then it is necessary to consider these ions in Three-Phase Element Model. Aluminum (Al), zinc (Zn), chlorine (Cl), chromium (Cr), Arsenic (As), Selenium (Se), Cadmium (Cd), and lead (Pb) were also measured by the Inductivity Coupled Plasma Mass Spectrometer (ICP-MS), but they were not measured in later tests and included in Three-Phase Element Model because the results were very small, either below the detection limit or less than 0.1 mg/L.

Table 3. Result of the CO_2 fixation capability

		Result
Fixation Time [h]		16.6
Amount of CO_2 Fixation per Unit Dry Mass from Mass Balance Method [g-CO_2/g-sample]		0.148
Amount of CO_2 Fixation per Unit Dry Mass from Carbonate Content Test Method [g-CO_2/g-sample]		0.119
	Before Test	After Test
Water Content [%]	29.2	31.4
pH [-]	12.5	8.5
Mineral Composition from XRD	$Ca(OH)_2$ $Ca_2Al[AlSiO_7]$	$CaCO_3$ $Ca_2Al[AlSiO_7]$
Leached Na^+ [mg/L]	654	889
Leached Mg^{2+} [mg/L]	373	215
Leached K^+ [mg/L]	Below Limit	13
Leached Ca^{2+} [mg/L]	421	376

Al, Zn, Cl, Cr, As, Se, Cd, and Pb leaching were below the detection limit or less than 0.1 mg/L.

3 OBTAINING PARAMETERS DURING THE CO_2 FIXATION TEST

For the Three-Phase Element Model, it is necessary to gain data from different parts of a specimen. Two samples were obtained from two layers perpendicular to the aeration direction of specimen for the constant flow aeration type CO_2 fixation test and were named inlet sample and outlet sample shown in Figure 4. The CO_2 fixation test was conducted in the same manner as in chapter 2. To get change of parameters over time, the CO_2 fixation tests were stopped at various times and the inlet sample and outlet sample were obtained. After the sampling, the samples were mixed enough and then subjected to water content tests, pH tests, carbonate content tests, and measurements of the leaching amounts of Na^+, Mg^{2+}, K^+, and Ca^{2+}.

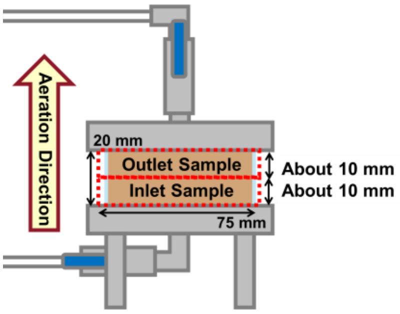
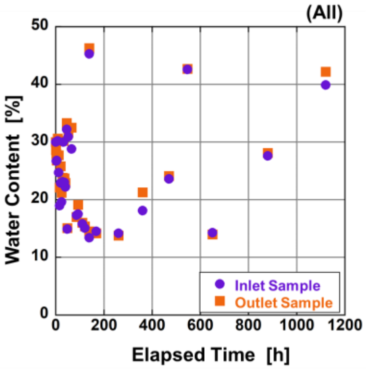


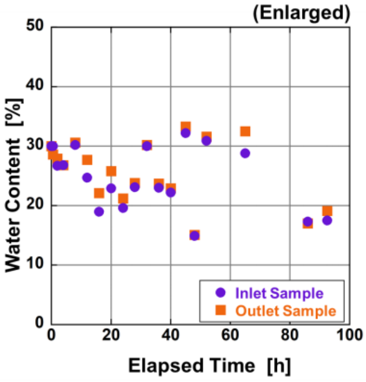
Figure 4. Conceptual illustration of two-layer sampling

The pH test was performed according to the pH test method for soil suspensions (JGS 0211-2020), because the amount of sample was small, a suspension was prepared using 3 g of sample (dry mass) and 15 g of water (including pore water) to ensure that the liquid-solid ratio was within the standard range. In addition, the amount of Na, Mg, K, and Ca dissolution was measured by ICP-OES using solutions made of 3 g of sample (dry mass) according to the method for the soil water-soluble material content test (JGS 0241-2020).

The data obtained was arranged over time and interpreted as the values at each elapsed time for inlet sample and outlet sample. Figure 5 is water content at each elapsed time. It was confirmed that the water content of the inlet sample and outlet sample were always kept close. Figure 6 is pH at each elapsed time. The pH of inlet sample mainly decreased before 20 h, that of outlet sample decreased from 24 h, and both of them decreased becoming closer in value between 24 h and 48 h. Figure 7 is the amount of CO_2 fixation at each elapsed time. The amount of CO_2 fixation of inlet sample increased rapidly until 8 h, that of outlet sample increased from 12 h, and both were closer from about 86 h. Figure 8 is the Ca^{2+} concentration of solution of samples and $n_{\mathrm{out}}/n_{\mathrm{in}}$ which is the ratio of the amount of CO_2 substance calculated from the CO_2 concentration and temperature measured inlet and outlet CO_2 sensor and indicates the proportion of CO_2 flowed out without reacting. The Ca^{2+} concentration decreased immediately after the start of the reaction in both samples. Inlet sample had a minimum value at 12 h, and outlet sample had a minimum value at 40 h. After that, it increased in both samples to about 500 h. It was also confirmed that the shape of the $n_{\mathrm{out}}/n_{\mathrm{in}}$ graph was similar to that of the Ca^{2+} concentration. However, the time at which the values changed was different. The $n_{\mathrm{out}}/n_{\mathrm{in}}$ increased significantly from 12 h, when the Ca^{2+} concentration in the inlet sample was at its minimum, to 40 h, when the Ca^{2+} concentration in the outlet sample was at its minimum, and the increase became smaller when the Ca^{2+} concentrations in both samples began to increase. Figure 9 is the concentrations of Na^+, Mg^{2+}, and K^+ at each elapsed time. As shown in Figure 9, the concentrations of Na^+ and K^+ decreased immediately after the start of the CO_2 fixation test and remained approximately constant. The concentration of Mg^{2+} increased slightly just before the end of the test but otherwise remained below the detection limit.

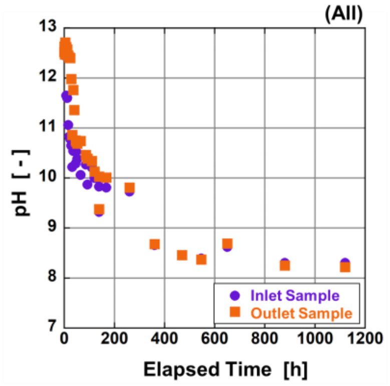


(a) All elapsed time



(b) Enlarged elapsed time

Figure 5. Water content at each elapsed time



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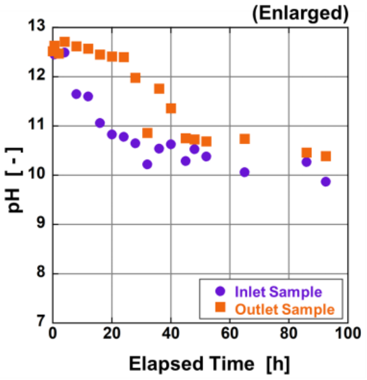
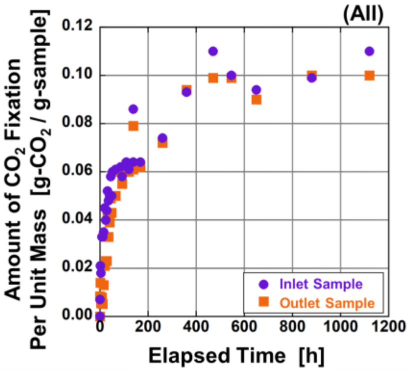
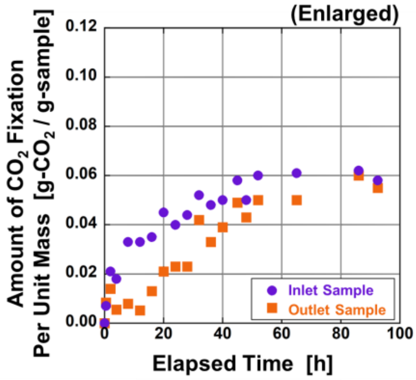


(b) Enlarged elapsed time

Figure 6. pH at each elapsed time

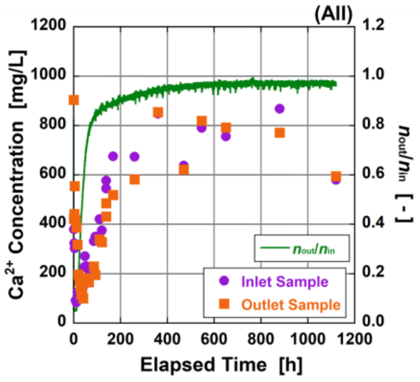


(a) All elapsed time

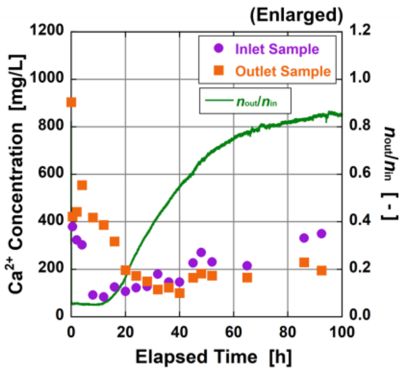


(b) Enlarged elapsed time

Figure 7. Amount of CO_2 fixation at each elapsed time



(a) All elapsed time



(b) Enlarged elapsed time

Figure 8. Ca^{2+} concentration and $n_{\mathrm{out}}/n_{\mathrm{in}}$ at each elapsed time

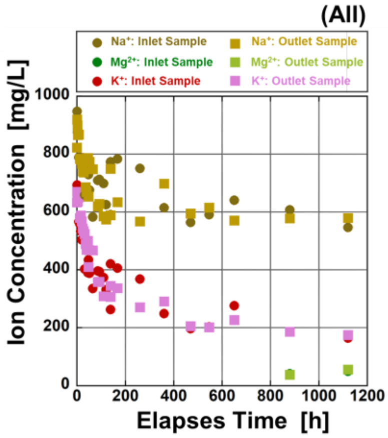


Figure 9. Concentration of Na^{+}, Mg^{2+}, and K^{+} at each elapsed time

As shown in Figure 5 to 9, it was experimentally confirmed that the CO$_2$ fixation reaction does not proceed simultaneously on the inlet side and outlet side of the specimen and the stage in which the reaction progresses significantly are visited in the order of the inlet side and the outlet side in the aeration. In addition, after 500 h, there was almost no change in the values except for scatter of the water content. Then, it is suggested that no change in the state of the pore water occurred after 500 h, and 500 h means the time the CO$_2$ fixation reaction had finished.

4 CULATION OF CHANGES IN THE VARIOUS IONS' CONCENTRATION IN PORE WATER

In order to obtain ion concentrations applicable to the Three-Phase Element Model, the ion concentrations in pore water are calculated using chemical equilibrium based on the previous study (Ishida, *et al.*, 2010). The results of pH and ion concentration were obtained by pH tests and leaching tests. In these tests, wet samples were added water and were left to stand. In other words, the pore water was diluted by adding water and was allowed to reach equilibrium before measurement pH and ion concentration. Therefore, in this paper, ion concentrations in the pore water were calculated by the water content, pH, and Ca^{2+} concentrations shown in Figures 5, 6, and 8 under the following assumptions below. Figure 10 is a conceptual illustration of the assumptions and calculation.

1. Water ionization equilibrium is always established.
2. CaCO$_3$ leaching reaches equilibrium in the test solution for the soil water-soluble material content test.
3. Carbonic acid ionization always gets equilibrium instantly (Ishida, *et al.*, 2010).
4. Other ions that are not directly related to the CO$_2$ fixation reaction (such as K^{+}) are not involved in the calculation.

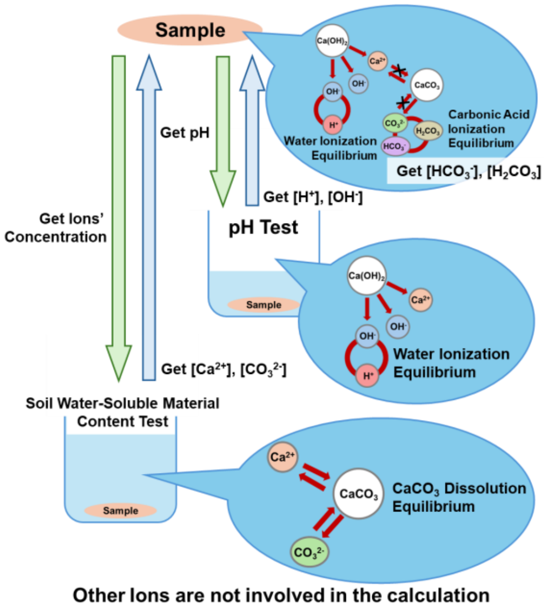


Figure 10. Conceptual illustration of assumptions and calculations

The constants used in the calculation, which is quoted from the previous study (Ishida, *et al.*, 2010), are as shown in Table 4. In addition, [X] in the formula means the concentration of X [mol/L].

Table 4. Constants Used in the Calculation

Explanation	Variable	Value
Solubility Product of CaCO$_3$	K_{sp}	$3.6 \times 10^{-9}\ \mathrm{mol^2 \cdot L^{-2}}$
Ionic Product of Water	K_{w}	$1.0 \times 10^{-14}\ \mathrm{mol^2 \cdot L^{-2}}$
Equilibrium Constant for the First Steps of the Ionization of Carbonic Acid	K_1	4.45×10^{-7} mol/L
Equilibrium Constant for the Second Steps of the Ionization of Carbonic Acid	K_2	4.69×10^{-11} mol/L

The calculation method is as follows:

Step 1: Calculate the H^{+} concentration ($[\mathrm{H^+}]_{\mathrm{pH}}$) of the test solution for the pH test from the result of the pH test (pH) as in Equation (1).

$$[\mathrm{H^+}]_{\mathrm{pH}} = 10^{-\mathrm{pH}} \tag{1}$$

Step 2: Calculate the OH^{-} concentration ($[\mathrm{OH^-}]_{\mathrm{pH}}$) of the test solution for the pH test from the ionic product of water (K_{w}) as in Equation (2).

$$[\mathrm{OH^-}]_{\mathrm{pH}} = \frac{K_{\mathrm{w}}}{[\mathrm{H^+}]_{\mathrm{pH}}} \tag{2}$$

Step 3: Calculate the dilution ratio (D) of the pore water to the test solution for the pH test from the liquid-solid ratio (α) of the test solution for the pH test and water content (w) as in Equation (3).

$$D = \frac{\alpha}{(w/100)} \tag{3}$$

Step 4: Calculate the concentration of H^+ and OH^- of the pore water ($[\mathrm{H}^+]$ and $[\mathrm{OH}^-]$). From the first assumption, since the equilibrium of the ionization of water is established in the solution, it is assumed that the concentration of c [mol/L] increases due to the ionization of water to $[\mathrm{H}^+]$ and $[\mathrm{OH}^-]$. At that time, $[\mathrm{H}^+]$ and $[\mathrm{OH}^-]$ satisfy Equations (4) to (6). Find the c with the smallest absolute value that satisfies the simultaneous equations and substitute it into Equations (4) and (5) and calculate $[\mathrm{H}^+]$ and $[\mathrm{OH}^-]$ in the pore water.

$$[\mathrm{H}^+] = D \times [\mathrm{H}^+]_{\mathrm{pH}} + c \tag{4}$$

$$[\mathrm{OH}^-] = D \times [\mathrm{OH}^-]_{\mathrm{pH}} + c \tag{5}$$

$$K_{\mathrm{w}} = [\mathrm{H}^+][\mathrm{OH}^-] \tag{6}$$

Step 5: Convert the unit of concentration of Ca^{2+} from [mg/L] to [mol/L] and calculate the concentration of Ca^{2+} in the test solution for the soil water-soluble material content test ($[\mathrm{Ca}^{2+}]_{\mathrm{Ca}}$).

Step 6: Calculate the concentration of CO_3^{2-} in the test solution for the soil water-soluble material content test ($[\mathrm{CO}_3^{2-}]_{\mathrm{Ca}}$). From the second assumption, since the solution equilibrium of $CaCO_3$ is established in the solution for the soil water-soluble material content test, $[\mathrm{CO}_3^{2-}]_{\mathrm{Ca}}$ is calculated by Equation (7) using solubility product of $CaCO_3$ (K_{sp}).

$$[\mathrm{CO}_3^{2-}]_{\mathrm{Ca}} = \frac{K_{\mathrm{sp}}}{[\mathrm{Ca}^{2+}]_{\mathrm{Ca}}} \tag{7}$$

Step 7: Calculate the concentration of Ca^{2+} and CO_3^{2-} of the pore water ($[\mathrm{Ca}^{2+}]$ and $[\mathrm{CO}_3^{2-}]$) as in Equation (8) and (9). Where D_2 in Equation (8) and (9) is the dilution ratio of the pore water to the test solution for the soil water-soluble material content test.

$$[\mathrm{Ca}^{2+}] = D_2 \times [\mathrm{Ca}^{2+}]_{\mathrm{Ca}} \tag{8}$$

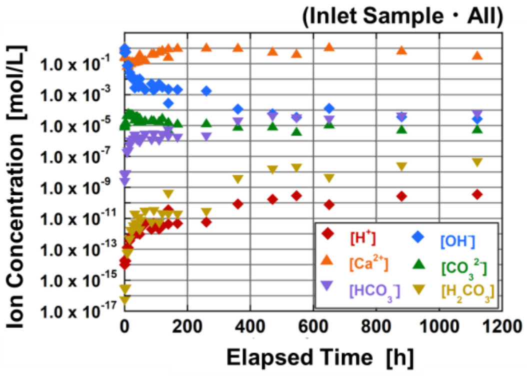
$$[\mathrm{CO}_3^{2-}] = D_2 \times [\mathrm{CO}_3^{2-}]_{\mathrm{Ca}} \tag{9}$$

Step 8: Calculate the concentration of HCO_3^- and H_2CO_3 of the pore water ($[\mathrm{HCO}_3^-]$ and $[\mathrm{H}_2\mathrm{CO}_3]$). From the third assumption, since the ionization of carbonic acid is established in the solution, $[\mathrm{HCO}_3^-]$ and $[\mathrm{H}_2\mathrm{CO}_3]$ are calculated as in Equation (10) and (11) using equilibrium constants for the first and second steps of the ionization of carbonic acid (K_1 and K_2).

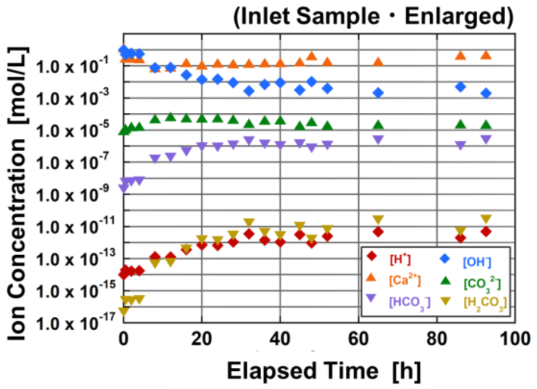
$$[\mathrm{HCO}_3^-] = \frac{[\mathrm{H}^+][\mathrm{CO}_3^{2-}]}{K_2} \tag{10}$$

$$[\mathrm{H}_2\mathrm{CO}_3] = \frac{[\mathrm{H}^+][\mathrm{HCO}_3^-]}{K_1} \tag{11}$$

Using the above calculations, the transition of various ion concentrations each of inlet sample and outlet sample were calculated from the pH and Ca^{2+} concentration obtained in chapter 3. Figures 11 and 12 are the transition of ion concentrations over elapsed time.

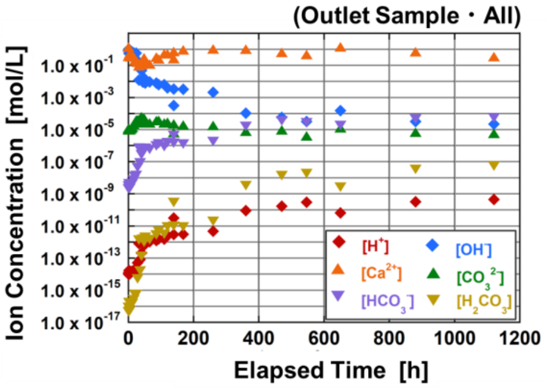


(a) All elapsed time

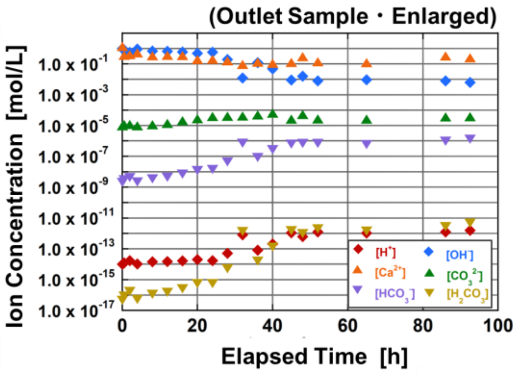


(b) Enlarged elapsed time

Figure 11. Transition of Ion Concentrations of inlet sample



(a) All elapsed time



(b) Enlarged elapsed time

Figure 12. Transition of Ion Concentrations of outlet sample

Comparing the enlarged versions of Figures 11 and 12, the various ion concentrations of inlet sample were changed significantly between 0 h and 24 h, and those of outlet sample were changed significantly between 24 and 48 hours. From this result, it is also suggested that the area where the CO_2 fixation reaction progresses actively moves from the outlet side to the inlet side in the Constant Flow Rate Aeration Type CO_2 Fixation Test. In addition, it is shown in Figure 8 that the Ca^{2+} concentration of inlet sample reached its minimum at 12 h and that of outlet sample reached at 40 h, and it was confirmed that the CO_3^{2-} concentration reached its maximum and the OH^- concentration rapidly decreased at this time.

One method to verify this calculation results is that the rate of $CaCO_3$ production is calculated from the concentrations of Ca^{2+} and CO_3^{2-}, and the amount of CO_2 fixation is calculated by integrating the rate and compared with Figure 7. According to previous research (Davies and Jones. 1955; Dawe and Zhang, 1997; Zhang and Dawe, 1998), the rate of $CaCO_3$ production ($r_{\mathrm{CaCO_3}}$) is shown in Equation (12).

$$r_{\mathrm{CaCO_3}} = k_{\mathrm{p}} \left(\left(\frac{[\mathrm{Ca^{2+}}][\mathrm{CO_3^{2-}}]}{K_{\mathrm{sp}}} \right)^{\frac{1}{2}} - 1 \right)^2 \qquad (12)$$

Where k_{p} is the precipitation rate constant. However, in the assumptions used in this paper, the product of $[\mathrm{Ca^{2+}}]$ and $[\mathrm{CO_3^{2-}}]$ was almost the same value because $[\mathrm{Ca^{2+}}]$ and $[\mathrm{CO_3^{2-}}]$ were calculated using the solubility product of $CaCO_3$ K_{sp} and the dilution ratio D, and the same reaction rate was calculated at both inlet sample and outlet sample over all times. Thus, this verification result does not match the result in Figure 7. Therefore, while the Three-Phase Element Model has the potential to be used as a new model to explain CO_2 fixation reaction, further improvements are required by changing the four assumptions and measuring the concentrations of other ions such as carbonate ions.

5 CONCLUSIONS

In this paper, the Three-Phase Element Model was proposed to interpret the CO_2 fixation reaction, and the water content, pH, the amount of CO_2 fixation, and Ca^{2+} concentration were obtained experimentally for use in the model. From the results, the transition of various ion concentrations in the pore water of inlet sample and outlet sample was investigated using chemical equilibrium calculations under four assumptions. The findings are as follows:

1. It was experimentally confirmed that the CO_2 fixation reaction does not proceed simultaneously on the inlet side and outlet side of the specimen.
2. It was suggested that the constant flow rate aeration type CO_2 fixation test has a stage in which the reaction progresses significantly, and that the stages are visited in the order of the inlet side and the outlet side in the aeration.
3. In the latter half of the CO_2 fixation reaction, the changes in ion concentrations became smaller, which suggested that the reaction was convergent.
4. It was confirmed that the changes in various ion concentrations in the pore water during the CO_2 fixation reaction can be calculated by calculating chemical equilibrium from pH and Ca^{2+} concentration.
5. It was suggested that when the reaction progresses significantly in the constant flow rate aeration type CO_2 fixation test, the Ca^{2+} concentration reaches a minimum value, the CO_3^{2-} concentration reaches a maximum value, and the OH^- concentration reaches a large decrease rate.
6. It was suggested that the Three-Phase Element Model has the potential to be used as a new model to explain CO_2 fixation reaction and further improvements are required by changing the four assumptions and measuring the concentrations of other ions such as carbonate ions.

6 ACKNOWLEDGEMENTS

This work was the result of using research equipment (RINT-Ultima III, ZSX Primus II: Material Characterization Central Laboratory) shared in MEXT Project for promoting public utilization of advanced research infrastructure (Program for supporting construction of core facilities) Grant Number JPMXS0440500024. Furthermore, the sample was given by MIDAC Holdings Co., Ltd., and generous advice on research and English writing was received from Prof. Dr. Eng. Komine, Dr. Eng. Ruan, Mr. Suzuki, and Mr. Urano in Waseda University. The authors would like to express their deepest gratitude to them.

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