Microbial electrolysis desalination and chemical-production cell for CO₂ sequestration

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HIGHLIGHTS

• Acid and base solutions produced from renewable organic matter.
• Desalinated water produced at the same time.
• Acid solutions used to accelerate dissolution of the mineral serpentine.
• Magnesium/calcium carbonates formed that sequestered CO₂.

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ABSTRACT

Mineral carbonation can be used for CO₂ sequestration, but the reaction rate is slow. In order to accelerate mineral carbonation, acid generated in a microbial electrolysis desalination and chemical-production cell (MEDCC) was examined to dissolve natural minerals rich in magnesium/calcium silicates (serpentine), and the alkali generated by the same process was used to absorb CO₂ and precipitate magnesium/calcium carbonates. The concentrations of Mg²⁺ and Ca²⁺ dissolved from serpentine increased 20 and 145 times by using the acid solution. Under optimal conditions, 24 mg of CO₂ was absorbed into the alkaline solution and 13 mg of CO₂ was precipitated as magnesium/calcium carbonates over a fed-batch cycle (24 h). Additionally, the MEDCC removed 94% of the COD (initially 822 mg/L) and achieved 22% desalination (initially 35 g/L NaCl). These results demonstrate the viability of this process for effective CO₂ sequestration using renewable organic matter and natural minerals.

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1. Introduction

Climate change is largely driven by increasing CO₂ emissions due to the combustion of fossil fuels (Crowley, 2000; Hansen et al., 2000; Kumar et al., 2011). Many non-fossil fuel alternatives, such as nuclear, biomass, and solar energy, continue to be more expensive than fossil fuels, resulting in a need for CO₂ sequestration methods that are sustainable, renewable, and cost effective (Mikkelsen et al., 2010; Ramanan et al., 2010; Cheng et al., 2013). The weathering of silicate minerals (such as olivine, serpentine, and wollastonite) is an important natural geochemical phenomenon through which atmospheric CO₂ can be transformed to carbonates (Brady, 1991; Drever, 1994; West et al., 2005), through reactions such as:

Olivine:

\[ \text{Mg}_2\text{SiO}_4 + 2\text{CO}_2 \rightarrow 2\text{MgCO}_3 + \text{SiO}_2 \]  

Serpentine:

\[ \text{Mg}_3\text{Si}_2\text{O}_5(\text{OH})_4 + 3\text{CO}_2 \rightarrow 3\text{MgCO}_3 + 2\text{SiO}_2 + 2\text{H}_2\text{O} \]  

Wollastonite:

\[ \text{CaSiO}_3 + \text{CO}_2 \rightarrow \text{CaCO}_3 + \text{SiO}_2 \]  

The formation of carbonates through these reactions is a geologically stable and environmentally safe way to store carbon. Additionally, silicate minerals are abundant on the earth. Rocks exposed on the earth’s surface by plate tectonics and erosion can be used to capture and store billions of tons of CO₂ per year, but this can only be a practical approach if the rates for these natural processes can be accelerated (Kelemen and Matter, 2008; Matter and Kelemen, 2009). Natural mineral carbonation is extremely slow due to slow rates of mineral dissolution (=0.3 mm/year) (Lackner et al., 1995; Kelemen and Matter, 2008). Various attempts have been made to enhance the mineral dissolution including heat treatment, dry or wet attrition grinding, and the use of chemical...
additives such as NaHCO₃/NaCl, acidic and alkaline solutions (Park and Fan, 2004; Krevor and Lackner, 2011; Bonfils et al., 2012). Dissolution of silicate minerals can be promoted using acidic solutions, but production of the acid can be expensive.

A microbial electrolysis desalination and chemical-production cell (MEDCC) is a bioelectrochemical system that has been used to desalinate salt water and produce acid and alkali using electricity generated by microorganisms at the anode and an external applied voltage (0.3 V–1 V) (Chen et al., 2012a,b). This system consists of an anode chamber, an acid-production chamber, a desalination chamber, and a cathode chamber (the alkali-production chamber). A bipolar membrane (BPM) is used to separate the anode chamber and the acid-production chamber. An anion exchange membrane (AEM) is placed between the acid-production chamber and the desalination chamber, with a cation exchange membrane (CEM) between the desalination chamber and the cathode chamber. With an electric field, water dissociation (H₂O → H⁺ + OH⁻) occurs in the BPM. H⁺ migrates through the cation exchange layer to the acid-production chamber to produce acid and OH⁻ migrates to the anode chamber through the anion exchange layer, neutralizing the H⁺ produced by microorganisms and maintaining near-neutral pH. Simultaneously, anions (e.g., Cl⁻) and cations (e.g., Na⁺) in the desalination chamber are driven through the AEM and CEM into the acid-production and cathode chambers, resulting in salt water desalination. In addition, an alkaline solution is produced in the cathode chamber through oxygen reduction (O₂ + 2H₂O + 4e⁻ → 4OH⁻).

The objective of this study was to investigate the feasibility of using a MEDCC for CO₂ sequestration through the use of the acid and alkali solutions to accelerate mineral carbonation in three steps (Fig. 1). First, acid produced by the MEDCC was used to dissolve natural minerals rich in magnesium/calcium silicates to obtain Mg²⁺ and Ca²⁺ ions. Next, part of the alkali solution produced by the MEDCC was used to raise the pH for CO₂ absorption, and then the rest was used to adjust the pH for carbonate precipitation.

2. Methods

2.1. MEDCC reactor construction and operation

The MEDCC reactors (duplicates) were made from four cylindrical chambers all 3 cm in diameter, consisting of an anode chamber (30 mL), an acid-production chamber (15 mL), a desalination chamber (15 mL), and an alkali-production chamber (30 mL) (Fig. 1). Chambers were made by drilling a hole in a solid block of Lexan. A BPM (Fumasep-FBM) was used to separate the anode and acid-production chambers, an AEM (Selemion AMV) was placed between the acid-production and the desalination chambers, and a CEM (Selemion CMV) was used to separate the desalination and alkali-production chambers. The anode was a heat treated graphite fibre brush (2.5 cm diameter/2.5 cm length) (Feng et al., 2010) placed horizontally in the centre of the anode chamber. The cathode with a projected surface area of 7 cm² was made from carbon cloth with four polytetrafluoroethylene (PTFE) diffusion layers and a 0.5 mg Pt/cm² catalyst layer (Cheng et al., 2006). Although Pt was used here, activated carbon can be used.

Fig. 1. Schematic representation of the MEDCC system and mineral carbonation processes.
instead of Pt to avoid the use of precious metals (Wei et al., 2011; Wang et al., 2013).

Anodes were preacclimated in microbial fuel cells (MFCs) inoculated with primary clarifier effluent collected from the Pennsylvania State University wastewater treatment plant. When stable cycles of voltage production were obtained using a 10 Ω external resistor after one month, anodes were transferred into MEDCC reactors. The anode solution contained (per L): 1 g sodium acetate, 4.28 g Na₂HPO₄, 2.45 g NaH₂PO₄·H₂O, 0.31 g NH₄Cl, 0.13 g KCl, 12.5 mL minerals, and 5 mL vitamins (Cheng et al., 2009). The acid-production and alkali-production chambers were filled with 0.35 g/L NaCl solution (~0.72 mS/cm), and 35 g/L NaCl was used in the desalination chamber (~54 mS/cm). The solutions in all chambers were replaced after each fed-batch cycle (every 24 h).

A fixed voltage of 1 V was applied to the MEDCC, and a 10 Ω resistor was connected between the positive lead of the power supply and the MEDCC anode to measure the current using a multimeter (Model 2700, Keithley Instrument). Once stable currents were achieved, samples were taken from each chamber for chemical analysis at the beginning and end of every fed-batch cycle, and mineral dissolution and carbonation were performed using the generated acid and alkali.

The soluble chemical oxygen demand (COD) from substrate was determined according to standard methods (Hach Co.). The pH was measured using a meter (VWR SB70P) and probe (SympHony probe), and conductivity was measured using a different meter (VWR SB70P) and probe (SympHony pH probe), and conductivity was measured using a meter (VWR SB70P) and probe (SympHony pH probe). Coulombic efficiency (CE) was calculated based on the total recovered coulombs divided by the acid-production or alkali-production efficiency was calculated based on the acid (or alkali) production and the recovered coulombs (Chen et al., 2012a).

### 2.2. Mineral dissolution

Serpentine from the Cedar Hills quarry in southeastern Pennsylvania was donated by the Department of Energy and Geo-Environmental Engineering, Penn State University. Samples were ground in a ball mill and sieved to the following size fractions: <38 μm, 38–75 μm, 75–150 μm, and 150–300 μm. Analysis using X-ray diffraction (XRD, PANalytical MPD) indicated that lizardite was the primary mineral phase (supporting information Fig. S1).

Mineral dissolution experiments were carried out in a 50 mL continuously stirred bottle using the acid produced by the MEDCC (15 mL) with different solid concentrations, particle sizes, reaction times, and temperatures (Table 1). After this dissolution step, the leachate was filtered through a 0.45 μm pore diameter cellulose acetate membrane, and analysed for Mg and Ca concentrations using an inductively coupled plasma atomic emission spectrometer (ICP-AES; Perkin-Elmer Optima 5300DV). The percentage of the Mg/Ca extraction was determined by the Mg/Ca concentration in the leachate and in the solid. The chemical composition of solid was analysed by ICP-AES after dissolved using a lithium metaborate fusion technique (supporting information Table S1).

### 2.3. Mineral carbonation

Mineral carbonation was achieved through several steps. First, the mineral was dissolved using the MEDCC-generated acid under optimized conditions (50 g/L solid concentration, <38 μm particle size, 24 h reaction time, and 50 °C temperature). Next, the leachate was filtered (0.45 μm pore diameter, cellulose acetate) and the pH was adjusted to 9, 9.5, 10, 10.5, 11, or 12 for CO₂ absorption using part of the MEDCC-generated alkali. The solution was then sealed in a 125 mL bottle using a stopper, and the headspace was filled with 99.99% CO₂. The CO₂ concentration was then measured after 24 h to obtain the amount of absorbed CO₂. The remaining alkali solution was then added to the leachate for carbonate precipitation. The precipitates were removed by filtration (0.45 μm) and dried at room temperature for 24 h. The mass of produced solid was determined gravimetrically, and the crystals were analysed for composition using X-ray diffraction (XRD). The XRD was performed with a PANalytical MPD XRD system using Cu Kα radiation, with an operating voltage of 45 kV and a current of 40 mA.

### 3. Results and discussion

#### 3.1. MEDCC performance

The maximum current density (Iₘₐₓ) of the MEDCCs was 7.5 ± 0.5 A/m² (Fig. 2A) at an applied voltage of 1 V. This was slightly lower than that previously reported in a similar MEDCC (8.4 ± 0.5 A/m²) (Chen et al., 2012a) due to the larger acid-production and desalination chambers used here (each 15 mL compared to 10 mL previously). The amount of Cl⁻ transferred from the acid-production to the anode chamber was greatly reduced by using a BPM, with an increase of only ~0.03 g/L Cl⁻. Over the course of several cycles the maximum current density slightly decreased, likely due to the effects of this Cl⁻ concentrations on anodic exoelectrogenic bacteria.

COD removal, coulombic efficiency (Fig. 2B), desalination (Fig. 2C), and pH changes in each chamber (Fig. 2D) were very consistent over 18 consecutive cycles as the amount of charge transferred over each cycle was similar (see supporting information Fig. S2). COD removals were 94 ± 1% for an initial value of 822 ± 21 mg/L, with nearly 100% coulombic efficiencies. The extent

### Table 1

Mineral dissolution experimental design and results, where the control is conducted without acid addition.

<table>
<thead>
<tr>
<th>Cycle #</th>
<th>Solid concentration (g/L)</th>
<th>Particle size (μm)</th>
<th>Reaction time (h)</th>
<th>Temperature (°C)</th>
<th>Mg²⁺ concentration (mg/L)</th>
<th>Ca²⁺ concentration (mg/L)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>5</td>
<td>&lt;38</td>
<td>24</td>
<td>25</td>
<td>311 ± 22</td>
<td>145 ± 2</td>
</tr>
<tr>
<td>2</td>
<td>25</td>
<td>&lt;38</td>
<td>24</td>
<td>25</td>
<td>738 ± 15</td>
<td>709 ± 48</td>
</tr>
<tr>
<td>3</td>
<td>50</td>
<td>&lt;38</td>
<td>24</td>
<td>25</td>
<td>892 ± 43</td>
<td>598 ± 60</td>
</tr>
<tr>
<td>4</td>
<td>100</td>
<td>&lt;38</td>
<td>24</td>
<td>25</td>
<td>1398 ± 87</td>
<td>77 ± 46</td>
</tr>
<tr>
<td>5</td>
<td>50</td>
<td>38–75</td>
<td>24</td>
<td>25</td>
<td>946 ± 68</td>
<td>1074 ± 101</td>
</tr>
<tr>
<td>6</td>
<td>50</td>
<td>75–150</td>
<td>24</td>
<td>25</td>
<td>913 ± 113</td>
<td>1016 ± 70</td>
</tr>
<tr>
<td>7</td>
<td>50</td>
<td>150–300</td>
<td>24</td>
<td>25</td>
<td>1254 ± 42</td>
<td>132 ± 8</td>
</tr>
<tr>
<td>8</td>
<td>50</td>
<td>&lt;38</td>
<td>6</td>
<td>25</td>
<td>932 ± 10</td>
<td>1072 ± 25</td>
</tr>
<tr>
<td>9</td>
<td>50</td>
<td>&lt;38</td>
<td>12</td>
<td>25</td>
<td>860 ± 32</td>
<td>774 ± 3</td>
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<tr>
<td>10</td>
<td>50</td>
<td>&lt;38</td>
<td>48</td>
<td>25</td>
<td>992 ± 53</td>
<td>724 ± 94</td>
</tr>
<tr>
<td>11</td>
<td>50</td>
<td>&lt;38</td>
<td>24</td>
<td>50</td>
<td>1025 ± 8</td>
<td>583 ± 37</td>
</tr>
<tr>
<td>12</td>
<td>50</td>
<td>&lt;38</td>
<td>24</td>
<td>100</td>
<td>1114 ± 63</td>
<td>278 ± 1</td>
</tr>
<tr>
<td>Control</td>
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<td>24</td>
<td>50</td>
<td>50 ± 4</td>
<td>4 ± 1</td>
</tr>
</tbody>
</table>
of desalination was 22 ± 3% for 35 g/L NaCl, which was lower than that previously reported for this system (86 ± 2%) (Chen et al., 2012a). However, a much higher concentration of NaCl was used here (35 g/L) than that previously tested (10 g/L NaCl), and the volume of the desalination chamber here (15 mL) was larger than that previously reported (10 mL). Therefore, the total amount of salt removed here was similar to that previously obtained.

The pH in the acid-production chamber decreased from 6.40 ± 0.22 to 1.20 ± 0.11 and the pH in alkali-production chamber increased to 12.75 ± 0.05 after a 24 h fed-batch cycle (Fig. 2D). In previous tests slightly better pH changes were obtained (0.68 and 12.9) as a result of the smaller acid-production and desalination chambers used in those tests (Chen et al., 2012a). A total of 0.84 ± 0.12 mmol of acid and 1.78 ± 0.13 mmol of alkali were produced for each cycle. Acid-production efficiencies (40 ± 6%) were lower than the alkali-production efficiencies (84 ± 6%) likely due to leakage of H⁺ into the desalination chamber. This proton leakage into the desalination chamber decreased the water pH from 6.65 ± 0.05 to 2.21 ± 0.05 (Fig. 2D). However, the anode pH was much more stable, with a final pH that was close to neutral (6.75 ± 0.05 V) due to the use of BPM (Fig. 2D), which was beneficial to the bacteria growth at the anode.

### 3.2. Mineral dissolution

As the solid concentrations were increased, Mg²⁺ concentrations in the leachate increased as expected, from 311 ± 22 mg/L to 1398 ± 87 mg/L (Table 1 and Fig. S3). Ca²⁺ concentrations initially increased from 145 ± 3 mg/L to 710 ± 48 mg/L, but then decreased to 77 ± 46 mg/L probably due to competitive Mg dissolution. The percentage of Mg extraction decreased from 30% to 7% with an increase in the initial mass concentration of the mineral. The percentage of Ca extraction declined from 100% to 3% as the mass of initial solid increased. Based on these results, the optimal initial mass concentration of the mineral was determined to be 50 g/L.

A reduction in the initial particle size will generally improve mineral dissolution due to the increased surface area per mass of mineral. However, there was little improvement in the dissolution rate when the initial particle size was <150 μm (Table 1 and Fig. S3), this is in agreement with previous findings on mineral dissolution rates with acid addition (Alexander et al., 2007). Thus, the smallest particle size (<38 μm) was considered to be the most useful here.

Mg²⁺ and Ca²⁺ concentrations were similar at reaction times of 6 h, 12 h, 24 h, and 48 h for <38 μm particle size and 50 g/L solid concentration (Table 1 and Fig. S3). In order to be consistent with the 24 h fed-batch cycle of the MEDCC, 24 h was considered a suitable reaction time. When the temperature was increased from 25 °C to 50 °C, Mg²⁺ concentration increased due to the improved reaction rates, and there was little change in Ca²⁺ concentrations (Table 1 and Fig. S3). When the temperature was further increased to 100 °C, Mg²⁺ concentration slightly increased and the Ca²⁺ concentration substantially decreased probably due to the formation of a silica passivation layer at high temperature (Table 1 and Fig. S3). Therefore, 50 °C was considered to be the more useful temperature.

The combination of conditions that were used here for further tests on mineral carbonation are: initial conditions of 50 g/L mineral and <38 μm particle size, a total reaction time of 24 h, and an elevated temperature of 50 °C. Under these conditions, the Mg²⁺ concentration in the leachate was 1025 ± 8 mg/L and Ca²⁺ concentration was 583 ± 38 mg/L. These concentrations are 20 times (Mg²⁺) and 145 times (Ca²⁺) higher than those of the controls (51 ± 4 mg/L Mg²⁺ and 4 ± 1 mg/L Ca²⁺) conducted under the same conditions but with no acidic solution (only 0.35 g/L NaCl, Fig. 2. (A) Current generation, (B) COD removal, (C) desalination percentage, and (D) solution pHs in anode, acid-production, desalination and alkali-production chambers of MEDCCs for 18 consecutive cycles. (For interpretation of the references to colour in this figure legend, the reader is referred to the web version of this article.)
pH = 6.8 ± 0.1). These results therefore demonstrated that mineral dissolution could be accelerated by using acid produced in MEDCCs.

3.3. Mineral carbonation

Following mineral dissolution tests under the chosen conditions, part of the solution was used to adjust the leachate pH to several different values (9, 9.5, 10, 10.5, 11, and 12) in order to accomplish CO2 absorption. The remainder of the alkali was then added for magnesium/calcium carbonates precipitation, resulting in final pHs in the range of 11.6–6.8 (Fig. 3). With an increase in the initial pH, the amount of CO2 absorbed increased from 2 mL to 45 mL, but the solid mass decreased from 52 mg to 6 mg due to less remaining alkaline solution used for the final precipitation stage (Fig. 3).

The solids produced during precipitation were analysed by XRD (Fig. S4). At an initial pH of 9, the solid was approximately half calcite [(Mg,Ca)CO3] and half brucite [Mg(OH)2], because only 2 mL of CO2 was absorbed in this test. At initial pH 9.5 and pH 10, the percentages of brucite decreased to nearly 35% and 20% as more CO2 was absorbed, and other magnesium carbonates (such as hydro-magnesite, Mg5(CO3)4(OH)2·4H2O, and nesquehonite, MgCO3·3H2·O) appeared. At an initial pH of 10.5, about 10% calcite and other amorphous precipitates were obtained due to the decreased final pH. At the high initial pHs (11 and 12), there was only a little solid produced and it was 100% calcite due to the low final pHs (9.3 and 6.8). These results were in agreement with the literature (Teir et al., 2007; Hanchen et al., 2008).

Considering the amount of absorbed CO2 and the mass and composition of the precipitated solids, an initial pH of 10 was considered to be optimal. In this case, 13.0 ± 0.3 mL CO2 was absorbed and 37.3 ± 0.1 mg of solid was precipitated in the form of ca. 80% magnesium/calcium carbonates (such as calcite, hydromagnesite, and nesquehonite) and ca. 20% brucite. Because some CO2 was generated in the anode chamber (about 0.8–1 mL), the net absorbed CO2 of this system was nearly 12 mL (i.e., almost 24 mg CO2 based on the molar volume 22.4 L of an ideal gas) and the net CO2 fixed as carbonates was approximately 13 mg.

3.4. Outlook

Mineral carbonation is a safe and relatively permanent method for CO2 disposal. Depending on the specific experimental conditions, it has been demonstrated here that CO2 could be chemically transformed to several different mineral substances (such as calcite, hydromagnesite, and nesquehonite). All of these mineral phases are considered to be stable on a time scale of millions of years (Lackner et al., 1995; Goff and Lackner, 1998). Moreover, calcium and magnesium-bearing minerals are abundant in nature and thus would not limit the application of this approach. The total accessible amounts of serpentine and peridotite have been estimated to significantly exceed worldwide reserves of coal (10,000 Gt), and could be used to bind about 1400–5550 Gt of CO2 (Lackner et al., 1995). However, this approach would not be practical unless rates of mineral carbonation could be increased, as shown here. By improving mineral dissolution by 20~145 times, it is estimated that 1~6 million tons of CO2 per year could be fixed using minerals in large and naturally occurring formations such as the Samail ophiolite, a thrust-bounded slice of oceanic crust and upper mantle (~350 km long, ~40 km wide, and ~5 km thick) (Kelemen and Matter, 2008). There are other ophiolites and calcium and magnesium-bearing minerals throughout the world, including the United States, Canada, and parts of Europe (Lackner et al., 1995; Goff and Lackner, 1998; Krevor et al., 2009). Therefore, it is estimated that most of the CO2 from anthropogenic emissions (about 4 billion tons per year) could be fixed by mineral carbonation assuming there was sufficient energy for this process.

The main operating costs of this system would be the mineral mining and grinding costs, and external power consumption. The digging and grinding costs for typical ores (e.g., copper) were estimated at about $4 per ton (Lackner et al., 1995). Thus, these costs are estimated to be $9.2 per ton CO2 based on 2.3 tons of serpentine required to bind one ton of CO2 (Lackner et al., 1995). This part of the costs may be avoided or reduced by using in situ mineral carbonation (Kelemen and Matter, 2008; Gislason et al., 2010; Lu et al., 2011). For a 24 h fed-batch cycle, approximately 5.72 × 10−3 kWh external electricity was consumed, with 24 mg of CO2 absorbed and 13 mg fixed in magnesium/calcium carbonates under optimal conditions. Assuming a cost of $0.065 kWh−1, this translates to $158 to absorb and $286 to fix one metric ton of CO2. This cost is similar to other current CO2 sequestration methods (Azar et al., 2006; Teir et al., 2007, 2009). However, it is possible to avoid the use of electrical grid energy by generating electrical power using salinity gradient energy rather than desalinating water (Post et al., 2008; Kim and Logan, 2011). Salinity gradient energy can be efficiently generated using a small number of anion and cation exchange membrane in a reverse electrodialysis stack (Post et al., 2008; Kim and Logan, 2011). Salinity gradients can be obtained using river and seawater sources, or generated from waste heat energy using thermolytic solutions (Cusick et al., 2012; Luo et al., 2013). Additionally, these estimates do not take into account possible additional economic benefits such as wastewater treatment and water desalination. Thus, the MEDCC could be a useful new approach for CO2 sequestration, although more research is needed to make it a practical technology. Additional studies should consider, using sustainable and green power sources to replace the external electrical power supply, modifying the MEDCC configuration to improve acid and alkali production efficiency, and investigating the feasibility of in situ applications.

4. Conclusions

A new approach for CO2 sequestration was demonstrated here using renewable organic matter and natural minerals. Over a fed-batch cycle (24 h), MEDCCs produced 0.84 mmol acid with a 40% production efficiency, and 1.78 mmol alkali with an 84% production efficiency. Simultaneously, the MEDCCs accomplished 94% COD removal and 22% desalination of the salt water (35 g/L NaCl).
By using the acid and alkali produced in the MEDCCs, mineral dissolution was improved by 20 times for Mg and 145 times for Ca, with 24 mg of CO₂ absorbed and 13 mg of CO₂ fixed as magnesium/calcium carbonates under optimal conditions.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at http://dx.doi.org/10.1016/j.biortech.2014.02.062.

References