

Insights into the Dielectric-Heating-Enhanced Regeneration of CO₂-Rich Aqueous Amine Solutions

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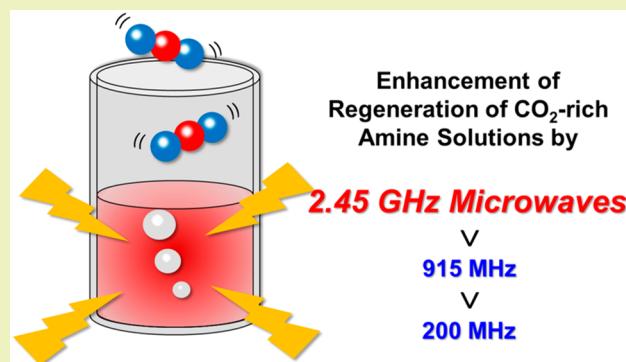
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ABSTRACT: Microwaves (MW) and radio frequency (RF) dielectric heating were used to facilitate the regeneration of CO₂-rich amine solutions, and the mechanism of the rate enhancement by dielectric heating was discussed. Dielectric measurements of aqueous solutions of monoethanolamine (primary amine), 2-(ethylamino)-ethanol (secondary amine), and N-methyl diethanolamine (tertiary amine) revealed that the formation of carbamate and bicarbonate ions by CO₂ absorption improves the dielectric loss tangent. Dielectric heating by MWs (2.45 GHz, 915 MHz) and RF (200 MHz) was compared to facilitate regeneration of the CO₂-rich amine solution at a constant power of 40 W. The CO₂ release rate was the highest at 2.45 GHz in all aqueous amine solutions, which was 1.47 to 1.74 times that of conventional heating by an oil bath operated at 120 °C. However, the CO₂ release rate decreased as the frequency decreased to 915 and 200 MHz. Electromagnetic field simulation suggested that CO₂ release was enhanced owing to the generation of a more intense electric field at 2.45 GHz than at lower frequencies.

KEYWORDS: *Microwaves, Radio frequency, Dielectric heating, Carbon capture and storage, CO₂, Chemical absorption process*



INTRODUCTION

The concentration of carbon dioxide (CO₂) in the atmosphere is increasing annually, thereby leading to serious climate changes. A significant reduction in CO₂ emissions is therefore required to realize a low-carbon society. Thus, CO₂ capture and storage (carbon capture and storage)^{1–5} as well as its effective utilization (carbon capture and utilization)⁶ are urgent research topics. Chemical absorption methods are one of the most promising methods that are being used in the actual CO₂ capture processes.^{1,7} In this process, CO₂ is chemically captured using absorbing media such as amine, ionic liquid,⁸ and amine-supported porous materials (e.g., SiO₂, SBA-15, and metal–organic frameworks (MOF)). The amine structure is designed to improve CO₂ absorption/desorption performance and consequently the process efficiency.¹² The amine solution absorbs CO₂ in the combustion exhaust gas by countercurrent contact in the absorption tower. Subsequently, the CO₂-rich amine solution is transferred to the regeneration tower and heated via steaming to dissociate CO₂. The regenerated amine solution returns to the absorption tower where the cycle repeats to continuously recover CO₂. The regeneration of the CO₂-rich amine solution by a conventional heating (CH) process involves high cost and consumes a considerable amount of energy and therefore needs to be improved.

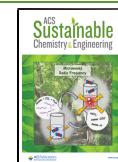
Dielectric heating with electromagnetic waves such as microwaves (MWs) and radio frequency (RF) is expected to reduce the energy consumption required for the regeneration of amines. Dielectric heating enables efficient heating using direct and selective material heating. MWs have been effectively applied, so far, for organic synthetic reactions,^{13–15} inorganic synthetic reactions,¹⁶ material processing,^{17,18} and catalytic reactions.^{19,20} Furthermore, the spread of renewable energy brings forth new chemical processes that produce chemicals using electricity generated by solar and wind power.²¹ Recently, a new MW chemical plant was implemented on a practical scale.²²

MW irradiation has been reported to be highly effective for CO₂ recovery systems using materials such as porous activated carbon,²³ MEA solution,²⁴ nonaqueous MEA solution,²⁵ and perfluorinated silica-stabilized dry alkanolamines.²⁶ Dielectric heating is expected to directly heat the CO₂-rich amine agents and release CO₂ in an energy-efficient way.²⁶ Understanding

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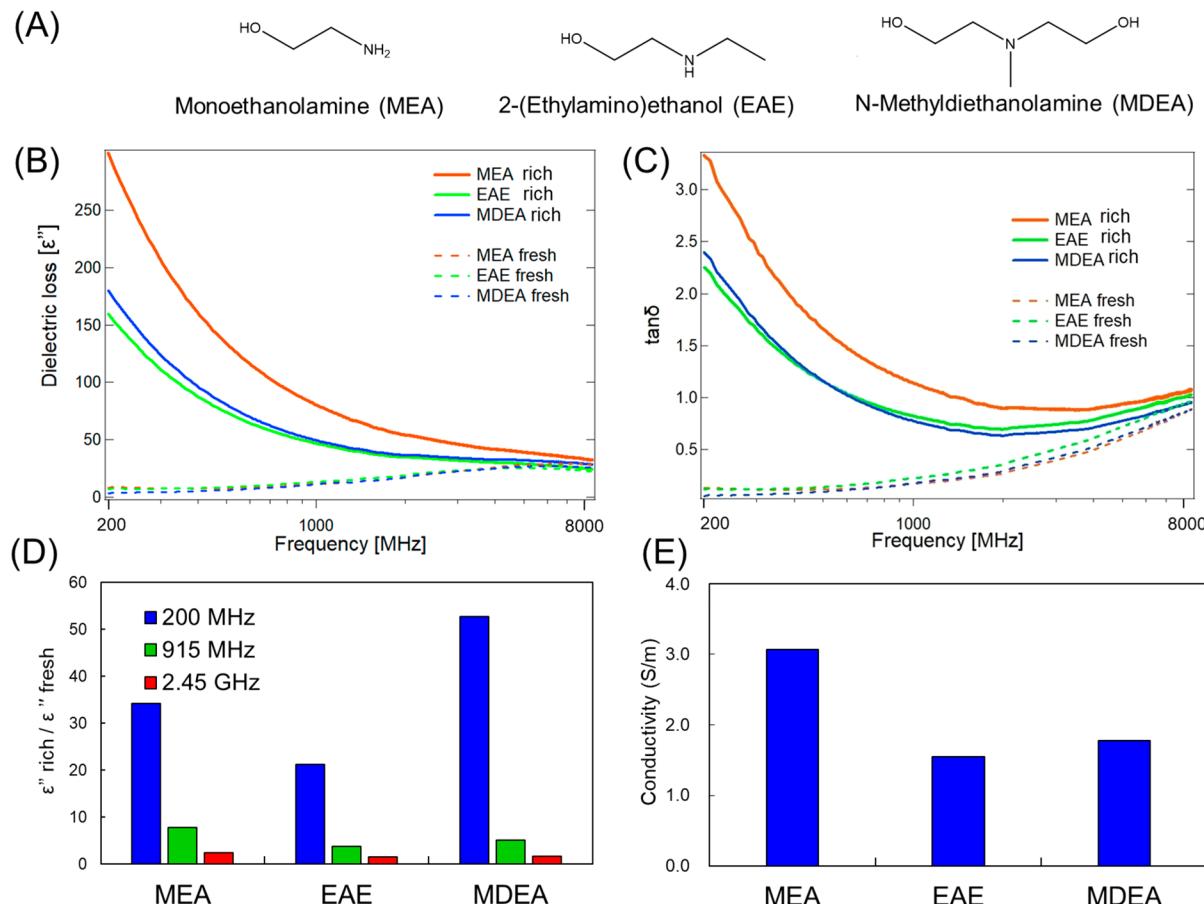


Figure 1. (A) Structures of the amine species. (B) Dielectric loss factor and (C) dielectric loss tangent of fresh and CO₂-rich amine aqueous solutions at 200 MHz to 8.5 GHz. (D) Ratio of the dielectric loss factor of each amine solution before and after CO₂ absorption. (E) Conductivity of the CO₂-rich amine solution.

the mechanism of the enhanced regeneration of an amine solution is relevant to improve the efficiency of the regeneration process by dielectric heating. In dielectric heating, heat is generated by the relaxation of the molecular orientation and vibration of ions.²⁷ Since the CO₂-rich amine solution contains carbamate and bicarbonate ions, MWs and RF can efficiently apply energy to these ion species selectively and enhance dissociation to release CO₂. Moreover, the different frequencies apply different intensities of the electromagnetic field at a unit volume. Lower frequency applies a weaker but more uniform electromagnetic field, while higher frequency applies a more intense electromagnetic field. Therefore, the frequency of the electromagnetic waves, intensity of the electromagnetic field, and dielectric properties of the materials are very important to achieve efficient dielectric heating. Moreover, the frequency dependence of the regeneration of the CO₂ amine is also important for scaling up of this system. Since the penetration depth of MWs at 2.45 GHz is short, the scaling up requires a lower frequency, such as 915 MHz and radio frequency below 300 MHz.

This study aims to demonstrate the enhanced regeneration of aqueous amine solutions by dielectric heating using various frequencies and understand the mechanism in which dielectric heating enhances the CO₂ release from amine solutions. We first characterized the dielectric properties of fresh and CO₂-rich amine solutions to clarify their susceptibility to electromagnetic waves. Then, we tested different electromagnetic wave frequencies (MWs and RF) to determine the effective

frequency for releasing CO₂. Finally, we discuss the mechanism of the enhancement of the CO₂ release from amine solutions by dielectric heating by focusing on the intensity of the applied frequency.

RESULTS AND DISCUSSION

The frequency dependence of the complex dielectric constants of fresh and CO₂-rich amine solutions was first investigated to clarify the MW absorption properties of aqueous amine solutions. The coaxial probe method was used to measure the complex dielectric constant of monoethanolamine (MEA), 2-(ethylamino) ethanol (EAE), and N-methyl diethanolamine (MDEA). Primary, secondary, and tertiary amines with simple structures were selected to evaluate their dielectric properties depending on the chemical structure of amine. Each aqueous solution contained 30 wt % of the amine, which is the most common composition for amine-based chemical absorption.²⁸

Figure 1B and C shows the values of the dielectric loss factor (ϵ'') and the dielectric loss tangent ($\tan\delta = \epsilon'/\epsilon''$) from 200 MHz to 8.5 GHz. The spectra of the fresh amine solutions exhibited a maximum ϵ'' at approximately 5–6 GHz, and this value decreased as the frequency decreased. The absorption of CO₂ increased the values of $\tan\delta$ at lower frequencies, which is attributable to the formation of ionic species by the two reactions that occur between the amine and CO₂ in aqueous solution.²⁹

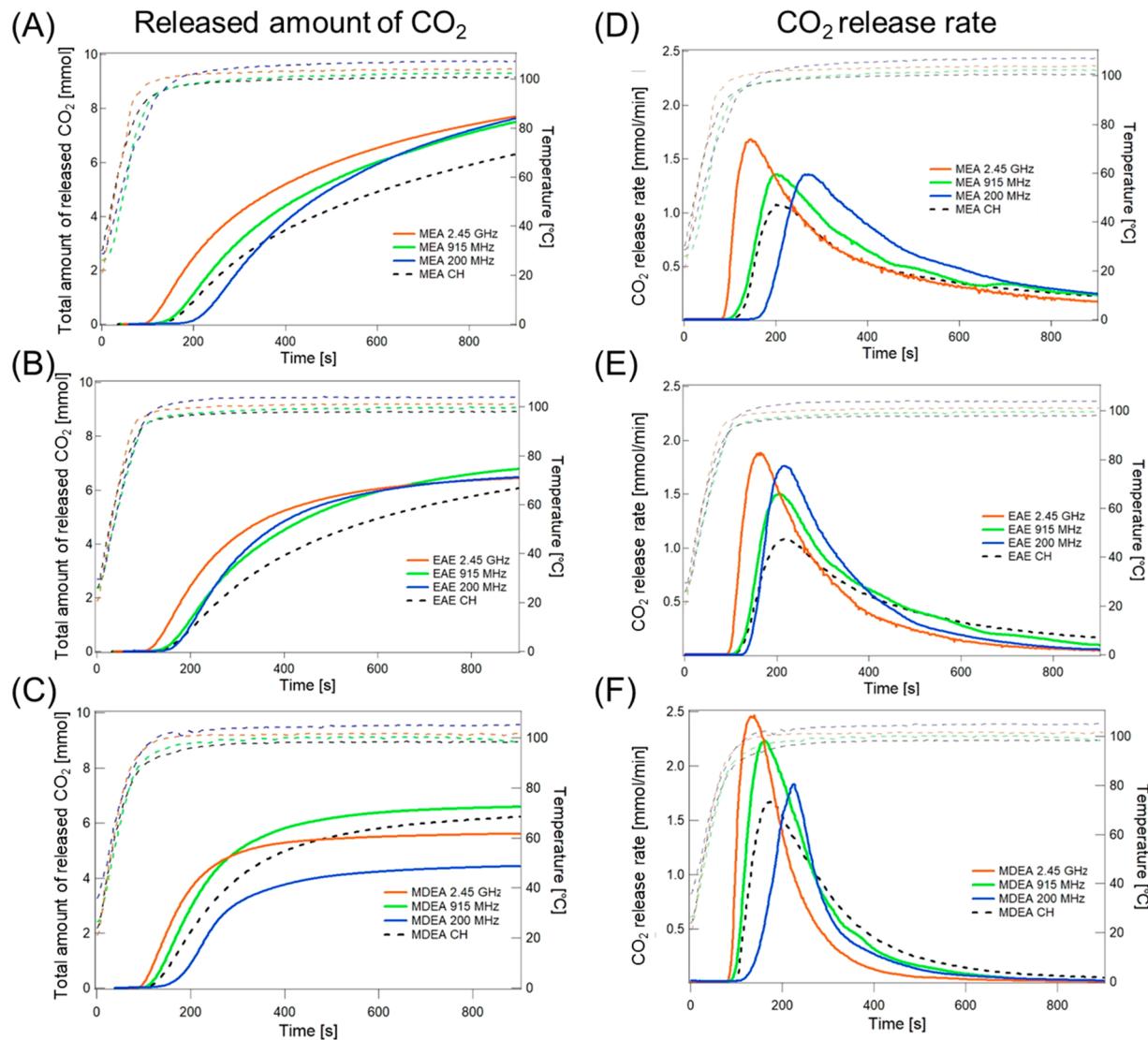


Figure 2. Total amount of released CO₂ at 200 MHz, 915 MHz, and 2.45 GHz for (A) MEA, (B) EAE, and (C) MDEA. The solid lines show the released amount, and the dotted lines show the temperature of the amine solution. CO₂ release rate at 200 MHz, 915 MHz, and 2.45 GHz for (D) MEA, (E) EAE, and (F) MDEA. The solid lines show the release rate, and the dotted lines show the temperature of the amine solution.

Table 1. Maximum CO₂ Release Rates from MEA, EAE, and MDEA at Different Frequencies^a

| Amine solution | Maximum CO ₂ release rate [mmol/min] | | | |
|----------------|---|---------|---------|----------|
| | CH | 200 MHz | 915 MHz | 2.45 GHz |
| MEA | 1.075 | 1.360 | 1.356 | 1.685 |
| EAE | 1.082 | 1.760 | 1.514 | 1.885 |
| MDEA | 1.669 | 1.830 | 2.220 | 2.457 |

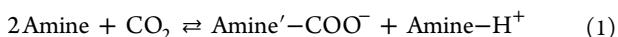
^aConstant power of 40 W.

Table 2. Heating Rates during CO₂ Regeneration^a

| Amine solution | Heating rate [°C/s] | | | |
|----------------|---------------------|---------|---------|----------|
| | CH | 200 MHz | 915 MHz | 2.45 GHz |
| MEA | 0.7947 | 0.5976 | 0.5476 | 0.8161 |
| EAE | 0.7919 | 0.7944 | 0.4263 | 0.8168 |
| MDEA | 0.8008 | 0.8018 | 0.3867 | 0.8448 |

^aConstant power of 40 W.

Carbamate formation:



Bicarbonate formation:



Both **reactions 1** and **2** occur with the primary and secondary amine species, whereas the tertiary amine participates only in **reaction 2**. The ionic species do not exist in the fresh amine solution and are generated when CO₂ is absorbed in the solution. The formation of the ionic species causes a conduction loss and improves the electromagnetic wave absorption characteristics.²⁷ Electromagnetic waves interact more strongly with the CO₂-rich amine solution due to high dielectric loss of the formed carbamate and bicarbonate ions. Thus, electromagnetic waves will promote CO₂ release. MEA has a larger dielectric loss than EAE and MDEA. This is attributed to the larger molar concentration of MEA that produces more ionic species in the solution (**Table S1**). **Figure 1D** and **E**, respectively, shows the dielectric loss factors before

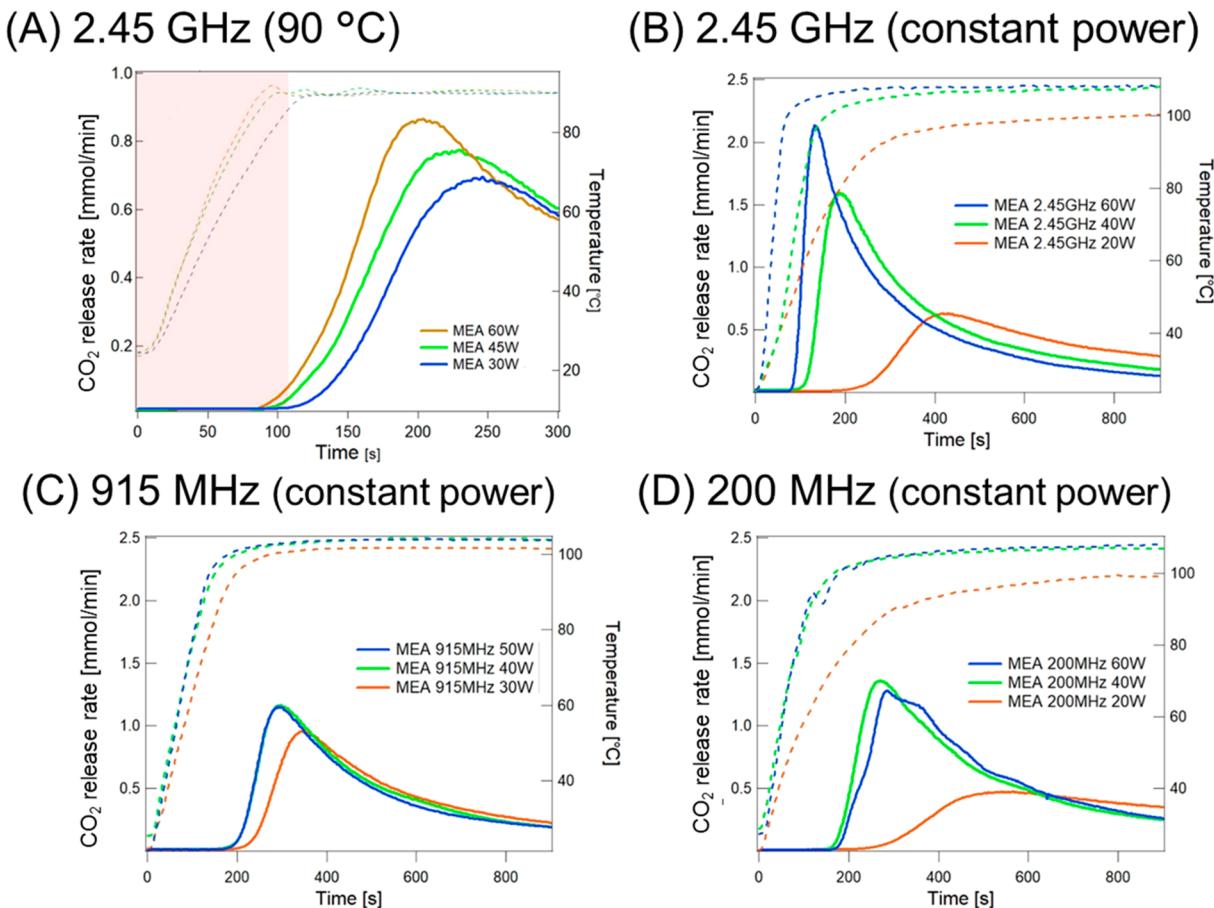


Figure 3. Dependence of the CO₂ release rate on the heating rate at different powers. (A) CO₂ release rate from MEA by constant MW power during temperature rise and held at 90 °C. CO₂ release rate from MEA at (B) 2.45 GHz, (C) 915 MHz, and (D) 200 MHz at rated powers of 20–60 W.

and after CO₂ absorption at each frequency (200 MHz, 915 MHz, and 2.45 GHz) and the conductivity calculated from the dielectric spectrum. The fresh amine solution does not exhibit conductivity, while the absorption of CO₂ significantly evolved that of the solution owing to the formation of the ionic species.

Figure S1 illustrates the depth of MW penetration in the solution calculated from the complex dielectric constant of the CO₂-rich amine solution. The dielectric loss factor and the penetration depth of the amine solution are important parameters for designing the scale-up of the process. Because the dielectric loss values and the penetration depth are inversely proportional to each other, the dielectric loss of MEA is larger than those of EAE and MDEA, while MEA has a smaller penetration depth. Moreover, the lower frequency was preferable for obtaining a larger penetration depth. Thus, they can be applied to a larger industrial process.

Irradiation with different electromagnetic wave frequencies (915 MHz, 2.45 GHz, 200 MHz) was performed to investigate the effects of the applied frequency on the enhancement of the regeneration of the amine solution. CO₂-rich amine solutions (30 wt %, 5.0 mL) were added to a quartz test tube and placed in the maximum electric field (*E*-max) of the waveguide (915 MHz, 2.45 GHz) and parallel-plate-type applicator (Figure S3).^{30,31} Dielectric heating started after replacing the inside of the reaction system with Ar. A constant MW and RF power of 40 W was applied for 15 min. Stirring was performed by bubbling using the Ar carrier gas (30 mL/min), and therefore, no stirrer bars were needed. The amount of CO₂ released

during heating was analyzed using mass spectrometry (QMS 100, Stanford Research Systems). An optical fiber thermometer was used to measure the solution temperature during the reaction. A reflux condenser was connected above the quartz reactor to prevent the evaporation of water. The control experiment was conducted by CH using an oil bath (120 °C).

Figure 2A–C depicts the amounts of released CO₂ from MEA, EAE, and MDEA, respectively, by CH and MWs. After 15 min, there was almost no difference in the amounts of released CO₂ at any frequency for MEA and EAE. For MDEA, the highest amount of released CO₂ was observed at 915 MHz, followed by 2.45 GHz and 200 MHz. The earliest release of CO₂ occurred during heating at 2.45 GHz, followed by 915 and 200 MHz. Irradiation at 27 MHz (RF, parallel-plate-type) was also tested, although sufficient heating did not occur because of poor impedance matching owing to their large conduction losses. After 30 min of dielectric heating, residual CO₂ in the amine solutions was estimated from the conductivity calculated from the complex dielectric constant (Table S2). The regenerated amine solutions exhibited conductivity loss of 1.7742 S/m → 0 S/m (MDEA), 1.5471 S/m → 0.0317 S/m (EAE), and 3.0687 S/m → 0.2748 S/m (MEA), respectively. Assuming that the conductivity correlates linearly with the amount of carbamate and bicarbonate ions, approximately 8.95% of CO₂ is contained in the MEA solution after dielectric heating. In contrast, the complete regeneration was achieved by MDEA.

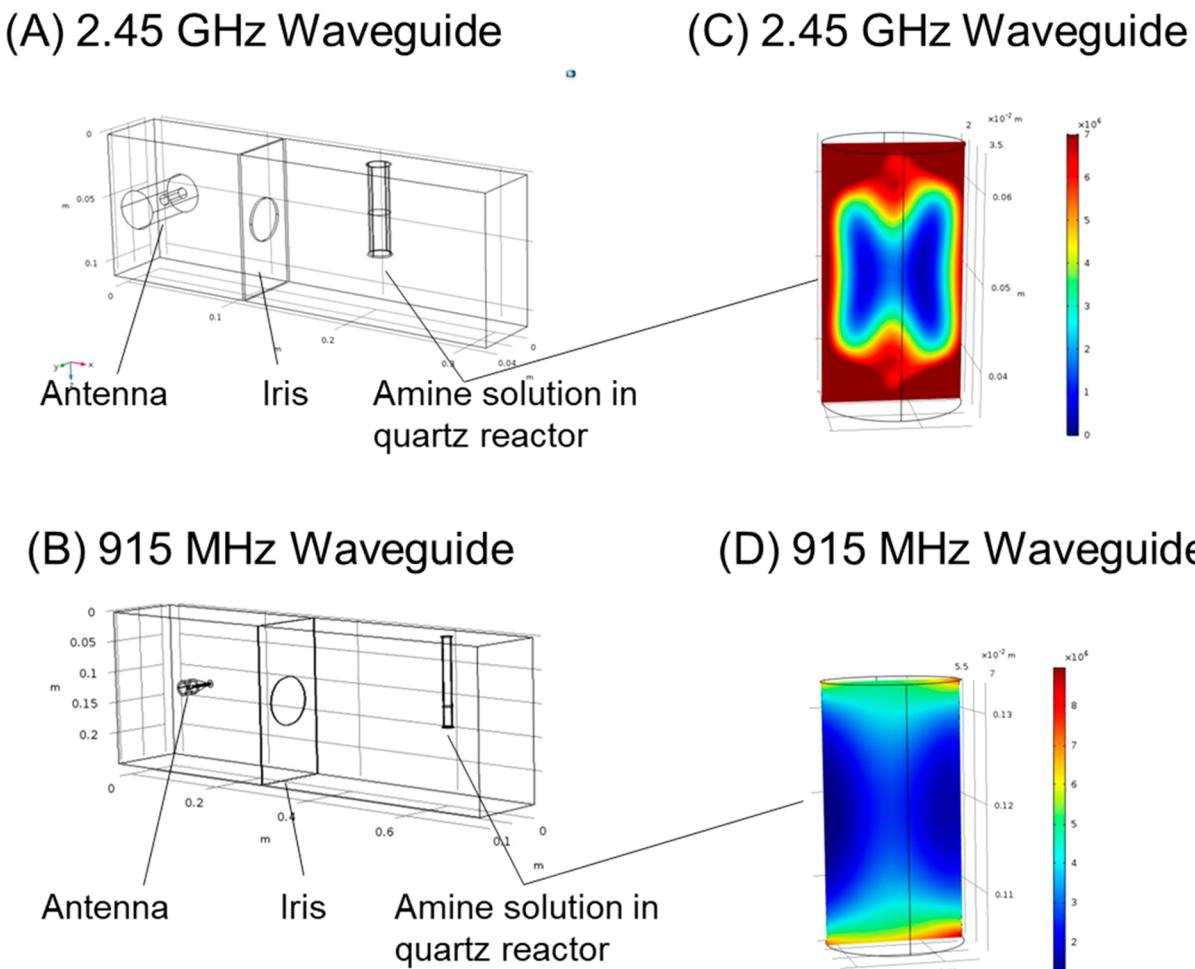


Figure 4. Waveguide geometry used for the simulation of the electromagnetic wave distribution and the electromagnetic field strength on the amine solution at 2.45 GHz and 915 MHz. Geometry of the (A) 2.45 GHz and (B) 915 MHz waveguides with the MEA solution in a quartz reactor. Simulated electric field strength (V/m) on the amine solution at (C) 2.45 GHz and (D) 915 MHz. The range of the color labels is from 0 to 1.2×10^6 V/m.

We focused on the CO₂ release rate in the initial stage of the regeneration owing to the large CO₂ release at this stage. Figure 2D–F illustrates the release rate of CO₂ at each frequency. The CO₂ release rate evolved when the temperature reached nearly 100 °C. After reaching 100 °C, the release rate gradually decreased. Table 1 presents the exact values of the maximum CO₂ release rate of each amine. For all the amine species, MW irradiation led to an increase in the CO₂ release rate compared to CH. Irradiation at 2.45 GHz produced the highest CO₂ release rate for all the species, attaining 1.47 to 1.74 times that of CH. However, the highest solution temperature was observed at 200 MHz. Therefore, the maximum solution temperature does not significantly affect the CO₂ release rate.

We verified the contribution of the heating rate to the release of CO₂. Table 2 presents the heating rate during CO₂ release. The heating rate was calculated from the slope of the linear temperature increase, from 40 to 80 °C. The increase in temperature due to CH was approximately 0.8 °C/s for all the amine species. In contrast, there was a significant difference in the heating rate depending on the frequency and amine species. At 200 MHz, a temperature rise of approximately 0.8 °C/s was observed for both EAE and MDEA, although this rate slowed to approximately 0.6 °C/s for MEA. At 915 MHz,

similar heating rates (approximately 0.4 °C/s) were observed for EAE and MDEA, while a rapid heating rate of 0.55 °C/s was observed for MEA. This is attributed to the high dielectric loss for MEA (Figure 1B, C). At 2.45 GHz, the heating rate was the same for all the amine solutions and was almost the same as that by CH because 2.45 GHz MWs interact more strongly with water than with ions, and therefore, no difference in the temperature profiles is observed depending on the amine species.

Then, the heating rate was changed by adjusting the MW power. The temperature was kept constant after it reached 90 °C by controlling the MW power. Figure 3A shows the CO₂ release rate of MEA at different MW powers during and shortly after the heating period. The shaded pink area indicates the length of the heating period. The heating rates changed substantially with different MW powers, which also improved the CO₂ release rate. Figure 3B–D shows the variations of the CO₂ release rates changing during the MW irradiation observed under the different applied constant powers. The heating rate and the CO₂ release rate were more significantly affected by the MW power at 2.45 GHz than at 915 and 200 MHz.

On the basis of the above-mentioned results, the mechanism by which the CO₂ release rate from the amine solution varies

depending on the applied MW frequency can be inferred. Different applicators are needed to change the applied frequency because the wavelength of the electromagnetic waves differs from one another. For example, 2.45 GHz has a wavelength of 122 mm, while 915 MHz has a wavelength of 328 mm. The longer the wavelength is, the larger the required waveguide is. Therefore, the electric field strength per unit volume can vary, even with the same power. Thus, the strength of the electromagnetic field exerted on the sample was calculated by finite element analysis using COMSOL Multiphysics. We reproduced the geometries of the waveguides at 915 MHz (TE_{102} -mode) and 2.45 GHz (TE_{103} -mode) and compared their electric field strengths. Figure S6 shows the electromagnetic field distribution when an electromagnetic wave of 40 W was applied to the empty waveguide. The electric field strength at the E-max position was 1.16×10^5 V/m at 2.45 GHz, while it was 0.797×10^5 V/m at 915 MHz. Figure 4 shows the electric field strength generated in the sample. The electromagnetic power loss, which indicates the actual MW power absorbed by the amine solution, of the MEA solution reached 30.9 W for 2.45 GHz, while that for 915 MHz was 20.3 W. Therefore, 2.45 GHz exerted a more intense electric field on the amine solution. Since the penetration depth of MWs at 2.45 GHz is short, the scaling up requires a lower frequency, such as 915 MHz and radio frequency below 300 MHz. However, our result indicated that the lower frequency was not as suitable as 2.45 GHz. The electromagnetic field simulation suggested that the concentrated electromagnetic field is relevant to enhance the CO_2 release.

An intense electromagnetic field can also occur at the microscopic level. De Bruyn et al. reported that the reactions are accelerated by local heating that occurs by concentrating the MW electric field near the interface of bubbles.³² Similar to this study, the present system uses Ar bubbling during electromagnetic wave irradiation. From this, a local high-temperature field could be formed near the bubbles by the MWs without changing the bulk temperature. Local heating of the bubbles can enhance the CO_2 release rate by accelerating the diffusion of CO_2 into the bubbles.

In addition, we can design a more dielectrically superior amine solution to enhance energy propagation efficiency by MWs. We can also stabilize these amines on mesoporous supports.^{9–11,23} Since most of the mesoporous supports (such as SiO_2) are transparent to MWs, the supported amine can be more selectively heated by MWs. The MW process bypasses the heat convection through the supports; therefore, the energy efficiency can be more improved than CH. The larger penetration depth of this system is also preferable for scaling up.

CONCLUSIONS

This study demonstrated the dielectric-heating-enhanced regeneration of CO_2 -rich primary (MEA), secondary (EAE), and tertiary (MDEA) amine aqueous solutions. The complex dielectric constants of the CO_2 -rich amine solutions were measured to evaluate their MW and RF susceptibilities. The fresh amine solutions showed a broad peak at 5–6 GHz. However, the CO_2 -rich amine solutions showed a clear increase in the dielectric loss in the low-frequency region. This is attributed to the conduction loss caused by the formation of carbamate and bicarbonate ionic species. Next, the CO_2 -rich amine solution was regenerated by dielectric heating (MW and RF). The release of CO_2 was enhanced at

2.45 GHz compared to the other frequencies. Furthermore, the CO_2 release rate from the amine solution increased as the heating rate increased. The enhancement of the CO_2 release rate by dielectric heating was attributed to the high-density electromagnetic field formed by the shorter wavelength electromagnetic waves of 2.45 GHz.

ASSOCIATED CONTENT

Supporting Information

The Supporting Information is available free of charge at <https://pubs.acs.org/doi/10.1021/acssuschemeng.0c05342>.

Detailed materials and methods, CO_2 release rate, electromagnetic field simulation for estimating the intensity of the electric field under 2.45 GHz and 915 MHz, and additional experimental data. (PDF)

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Notes

The authors declare no competing financial interest.

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ABBREVIATIONS

CH, conventional heating; EAE, 2-(ethylamino) ethanol; MDEA, *N*-methyl diethanolamine; MEA, monoethanolamine; MW, microwave; RF, radio frequency.

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