

Sorbent Regeneration via Radio Frequency Assisted Dielectric Heating for Direct Air Capture of CO₂

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ABSTRACT: Direct air capture (DAC) of CO₂ is a negative emission technology that utilizes sorbents requiring a regeneration step for continued absorption-desorption cycles and reuse. Here, we report an unconventional approach to desorb captured CO₂ from a porous sorbent using radio frequency (RF) irradiation for dielectric heating and targeted energy transfer with the goal of lowering the regeneration energy requirement, enabling modularity, and promoting renewable energy input. Regeneration of composites of functional ionic liquid and metal organic framework (IL/MOF) with high CO₂ capacity, selectivity and enhanced transport are demonstrated under conditions relevant to DAC. RF-assisted dielectric heating at MHz frequency range shows effective absorption of the electromagnetic energy and consequently the rapid release of the captured CO₂. This study demonstrates a low frequency electromagnetic radiation as an alternative approach with multiple prospects of tunability for energy efficiency in DAC.

The constant rise in energy demand has driven atmospheric CO₂ concentrations to 410 ppm, primarily due to fossil fuel combustion and industrial processes.^{1,2} These emissions are major contributors to climate change and global warming, accompanied by an increase in the earth's average temperature.^{3,4} To mitigate these impacts and reverse the harm caused by excess CO₂, developing effective CO₂ capture materials and processes is crucial to remove approximately 10 gigatons of CO₂/year to limit global warming to 1.5 °C by 2050.^{5,6} However, for direct air capture (DAC), finding an energy-efficient and cost-effective sorbent remains a significant challenge particularly due to the very low concentration of CO₂ in air. While commercially available technologies like aqueous amines and alkali bases offer high CO₂ capture capacity, they rely on energy-intensive thermal regeneration.^{7,8} Thus, researchers are actively developing sorbents with high CO₂ capacity and ability to desorb the captured CO₂ via alternative regeneration methods such as dielectric heating,⁹⁻¹³ humidity swing,¹⁴⁻¹⁶ and electrochemical swing.¹⁷⁻¹⁹

Dielectric heating with electromagnetic (EM) radiations such as microwaves (MWs) is a promising approach for sorbent regeneration compared to conventional thermal heating methods.²⁰⁻²² This is because MW energy has the ability to directly and selectively heat the sorbent material primarily via reorientation of molecular dipoles, ionic conduction, or interfacial polarization in the presence of the rapidly oscillating electric field.^{23,24} As such, MW-assisted energy transfer achieves rapid

dielectric heating and faster CO₂ desorption rates by eliminating the heat transfer limitations associated with conventional conductive or convective heating, which involves gradual heating from the exterior of the adsorption column towards the sorbent. One of the earlier studies is by McGurk *et al.*²⁵ where they reported on the microwave-assisted regeneration of a monoethanolamine (MEA) solution, demonstrating its potential to rapidly regenerate the solvent post CO₂ saturation. Several other studies also suggest that MW based regeneration of solid sorbents including zeolites, activated carbon, and silica offers similar advantages of fast desorption rates.²⁶⁻²⁸

However, one recognized limitation of the MW-assisted regeneration technique is the formation of localized hot spots and large temperature gradients within the bulk sorbent due to the intense EM field.²⁹ This non-homogeneous heating leads to the partial thermal degradation of the material,³⁰ which impacts the sorbent performance and longevity. Thus, it is imperative to achieve a more uniform EM field and controllable heating during sorbent regeneration to reduce localized hot spots and temperature gradients. This can be achieved by air-sorbent contactor design in connection with the MW cavity design including the placement of the sorbent in the cavity. While there are operational parameters such as the way that the electromagnetic field is applied (e.g., power, pulsing,etc), there are also considerations that should be considered for the design sorbent. Up until now, sorbents have been developed

with the design feature of having a CO₂ affinity site for enhanced capacity and capacity as well as architectural features for enhanced transport.

Here, we report on the design consideration of sorbents for the RF based regeneration as well as demonstration of a stable absorption-desorption cycling with RF. To overcome the problem of localized overheating, we demonstrate the utility of lower range of EM radiation spectrum such as radio frequency (RF; 100–1000 MHz) compared to the much higher frequency of MW irradiation (2.54 GHz). By synergizing functional ionic liquids (ILs) that are susceptible to dielectric heating with structural supports that are not, sorbents with superior CO₂ capacities can be achieved. Studies have shown that the dielectric constant (ϵ') and dielectric loss factor (ϵ'') of ILs are high at lower frequencies (100–1000 MHz).^{31–33} Here, ϵ' describes materials ability to store MW energy, while ϵ'' indicates how much of that absorbed energy is converted to heat.

Recently, we reported microwave-assisted regenerability of a CO₂ reactive porous composite, zeolitic imidazolate framework (ZIF-8) loaded with 1-ethyl-3-methylimidazolium 2-cyanopyrrolide ([EMIM][2-CNpyr]) – a functional IL.³⁴ The sorbents demonstrated significant CO₂ capacity and rapid desorption upon MW application. However, the IL/MOF composite lost considerable working capacity after 10 continuous sorption-desorption cycles with MW regeneration. Here, these composites were leveraged for RF based regeneration, whereby formation of hotspots and non-homogeneous dielectric heating were suppressed significantly, thus enabling an energy efficient and modular approach to the current regeneration challenge of DAC.

We developed a custom-built RF setup consisting of a fringing field applicator configuration with two copper strips on a block of Teflon connected to an RF source, an amplifier, and a forward-looking infrared (FLIR) camera for temperature measurements. To determine effective frequency to achieve dielectric heating using RF radiations, we investigated frequency dependence dielectric response of three bulk solvents: MEA (a benchmark CO₂ capture solvent), ethyl-3-methylimidazolium tetrafluoroborate, [EMIM][BF₄] (a common non-functionalized IL), and [EMIM][2-CNpyr], a CO₂-reactive IL with a strong dipole moment.³⁵ By modulating both RF frequency and input power, we demonstrate the tunability of process parameters with respect to the specific solvent of choice based on their dielectric heating response. **Figure 1** shows the effect of frequency on temperature change measured by FLIR camera upon dielectric heating in response to the applied electric field and thermal images captured by FLIR camera at 60 seconds mark for bulk MEA, [EMIM][BF₄], and [EMIM][2-CNpyr] at four different EM wave frequencies (100, 200, 300, and 400 MHz) for 60 seconds at a constant input power of 50 W.

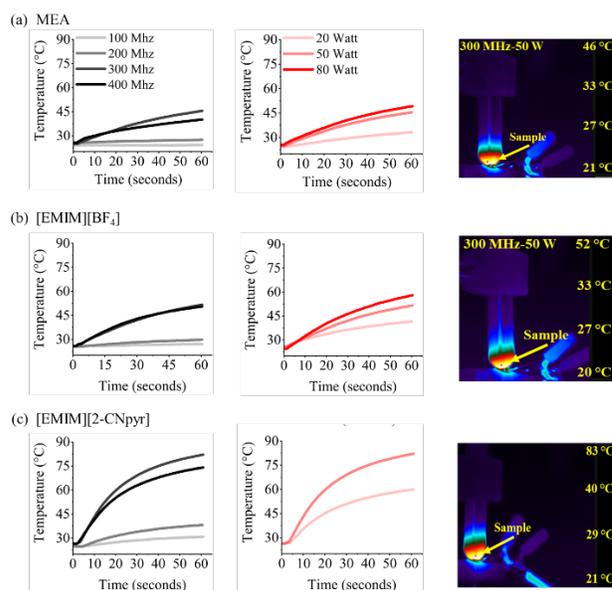


Figure 1. Left column: Temperature change over time upon dielectric heating at four different EM frequencies (100, 200, 300, and 400 MHz) for 60 seconds at a constant power of 50 W; Middle column: Temperature change over time upon dielectric heating at a constant frequency of 300 MHz and varied power for 60 seconds; Right column: Thermal images captured by FLIR camera at 60 second mark at a constant frequency of 300 MHz and 50 W power for (a) MEA (a), [EMIM][BF₄] (b), and [EMIM][2-CNpyr] (c).

All three solvents examined displayed an increased temperature response to increasing RF frequency up to 300 MHz. However, further increasing the frequency to 400 MHz led to a decrease in maximum achievable temperature for each solvent (**Figure 1, left**). At a fixed frequency of 300 MHz, MEA achieved the lowest temperature (46 °C) after 60 seconds, while bulk [EMIM][BF₄] and [EMIM][2-CNpyr] reached temperatures of 52 °C and 83 °C, respectively. Higher temperatures achieved by the ILs are due to their high polarity, which leads to a stronger overall dielectric response compared to the neutral MEA molecules. Furthermore, the anion ([2-CNpyr]⁻) has a larger dipole moment, resulting in a rapid dielectric response as compared to [BF₄]⁻. [EMIM][2-CNpyr] is also the highest viscosity solvent among the three examined. Additionally, increasing RF input power from 20 to 80 W at a fixed frequency of 300 MHz resulted in improved heating rates for all samples (**Figure 1, center**). Here, we also note that CO₂-saturated [EMIM][2-CNpyr] can be fully regenerated at 60 °C, as we have previously reported.³⁶ Therefore, to avoid exceeding the desired regeneration temperature, we limited the exposure of [EMIM][2-CNpyr] to 50 W, which effectively achieved 60 °C within the first 30 seconds of applying RF.

To assess the influence of CO₂ presence on solvent dielectric heating behavior, further experiments were performed at a fixed frequency of 300 MHz and 50 W

power. Since bulk [EMIM][BF₄] does not have CO₂ capacity at DAC conditions, we studied the dielectric heating response of CO₂-saturated MEA and [EMIM][2-CNpyr].

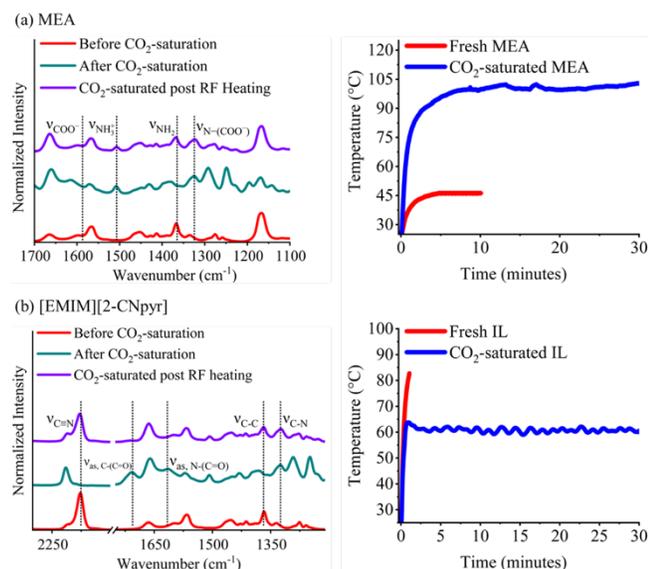


Figure 2. Left panel: FTIR spectra of fresh solvents (red), after CO₂ saturation (green), and post 30 minutes of RF application (blue); Right panel: Temperature change over time upon dielectric heating of fresh and CO₂-saturated solvents MEA (a) and [EMIM][2-CNpyr] (b).

FTIR spectra of fresh and CO₂-saturated MEA and [EMIM][2-CNpyr] confirm the presence of chemisorbed CO₂ when exposed to 1 bar of CO₂ (Figure 3, left). Both solvents increase in viscosity with CO₂ saturation. Subsequent to CO₂ sorption, samples were exposed to EM radiation using an RF frequency of 300 MHz at 50 W power. The corresponding temperature profiles of bulk MEA and [EMIM][2-CNpyr] before and after CO₂ saturation are also presented in Figure 3, right. While fresh MEA reached a temperature of 45 °C after 10 minutes of RF heating at 300 MHz, CO₂-saturated MEA reached to 90 °C after 10 minutes at the same frequency and power. This improvement in the dielectric response of MEA is due to the formation of ionic species such as carbamate and bicarbonate ions upon CO₂ saturation, which improved its ability to absorb EM irradiation.¹⁰ On the other hand, [EMIM][2-CNpyr] showed no observable difference in dielectric response with or without CO₂, and reached to 60 °C within 30 seconds of same RF application. The dielectric heating of CO₂-saturated MEA and [EMIM][2-CNpyr] for 30 minutes resulted in desorption of the captured CO₂, which was confirmed by the disappearance of CO₂-related peaks (carbamate and carbonate species),^{37,38} in the IR spectra obtained post-RF heating as seen Figure 3, left. These findings demonstrate that [EMIM][2-CNpyr] has excellent

dielectric response at low EM wave frequencies such that CO₂ desorption can be effectively achieved at much lower EM field strengths, compared to MEA.

The previous report on the [EMIM][2-CNpyr]/ZIF-8 composite, where regeneration via dielectric heating was achieved upon MW irradiation, a measurable decrease in CO₂ capacity was seen after the 10th cycle.³⁴ This loss in CO₂ working capacity was attributed to the possible degradation of the IL due to hot spot formation and uneven distribution of the EM field, despite the minimal exposure time. This issue is overcome with the lower frequency EM waves such as RF. The [EMIM][2-CNpyr]/ZIF-8 composites with 35 wt% IL loading were examined in a breakthrough measurement where absorption at 30 °C was followed by regeneration at 60 °C via dielectric heating due to RF irradiation. CO₂ breakthrough curves at 30 °C under 500 ppm CO₂ in N₂ under dry condition are shown in Figure 3a. The regeneration was performed by a no-contact fringing field applicator connected to an RF source. To maintain the regeneration temperature at 60 °C, RF field was kept at a frequency of 300 MHz with modulated RF power. The input power was 50 W initially and kept between 10 to 12 W after the temperature was reached to 60 °C. The composite temperature as measured by the FLIR camera is shown in Figure 3b. The CO₂ desorption was achieved rapidly within minutes as seen in Figure 3c. The full regeneration was completed in 30-min timeframe before the next sorption cycle was performed.

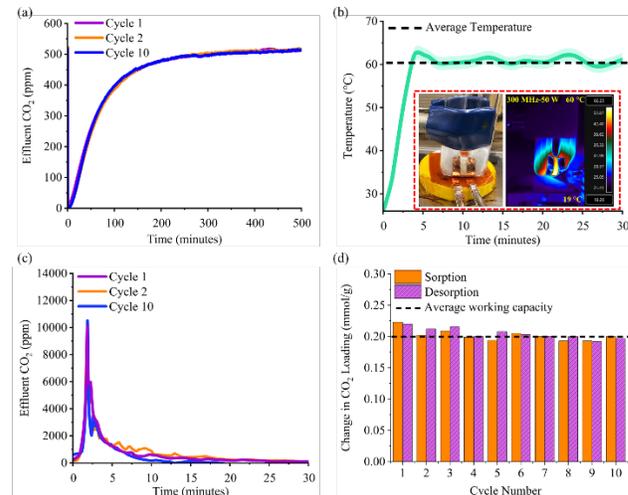


Figure 3. CO₂ breakthrough curves of [IL]₃₅/ZIF-8 under 500 ppm CO₂ in N₂ at 30 °C under dry conditions; (b) Smoothed temperature profile of [IL]₃₅/ZIF-8 composite during RF-assisted heating over time, shaded region indicates the error of ±2 °C. Inset shows photo of sample tube with [IL]₃₅/ZIF-8 composite (left) and thermal image of [IL]₃₅/ZIF-8 on copper strip applicator during RF-assisted CO₂ regeneration process (right) at a fixed frequency of 300 MHz; (c) RF-assisted CO₂ desorption under dry N₂ feed at 60 °C for 30 minutes; and (d) Dynamic CO₂ cyclic performance of [IL]₃₅/ZIF-8 under 500 ppm CO₂ in N₂ under dry condition.

Figure 3d shows the absorption-desorption behavior of the composites for 10-continuous cycles. Results show that both absorbed and desorbed amounts of CO₂ are consistent within the cycles, thus confirming the excellent cyclability of the composites under RF radiation. Accordingly, CO₂ capture capacity loss was minimal (~10%): 0.20 mmol CO₂/g (cycle 10) compared to 0.23 mmol CO₂/g (cycle 1). This performance surpasses MW-assisted regeneration, where the same composite demonstrated about 25% loss due to hotspot formation. This highlights the suitability of RF-based regeneration for composites of ILs, owing to the tunable ionic and dipolar polarization at the interface between the structural framework and the active liquid. Since captured CO₂ resides within the IL, EM energy directly heats the reactive component, thus enabling targeted heating and minimizing heat transfer to the ZIF-8 support structure.

This study showcases a low-energy approach to sorbent regeneration in DAC with the further ability to tune the dielectric properties of the sorbent with the choice of the active liquid such as a high-dielectric functional solvent (e.g. IL) and a structural support such as a low-susceptibility material to EM irradiation (e.g., MOF). The demonstrated approach suppresses the formation of hotspots in strong EM field, thus enabling cyclability in strategically designed composites for DAC technology powered by renewable energy.

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