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Manuscript title: FTIR and Physicochemical Analysis of Carbon-rich and Regenerated Aqueous MEA

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Abstract

Carbon dioxide (CO₂) is the most significant greenhouse gases contribute 44% global warming using Coal combustion for electricity generation. The major goal is to reduce the CO₂ emissions by Carbon Capture and Storage (CCS) technique. Among various techniques amine-based post-combustion carbon capture plays a critical role in CCS Technology. Monoethanolamine (MEA) act as a benchmarking solvent in CCS process owing to high absorption capacity, lower cost, and high rate of reaction. Present investigation uses 30wt% MEA and animized flue-gas (15 vol% CO₂and resting N₂ gas) at 0.5psi inlet pressure for CO₂ absorption followed by 1 hour solvent regeneration (direct and indirect heating). Furthermore, physiochemical properties such as pH, carbon-loading, density, viscosity,alkalinity, surface-tension and FTIR spectroscopic analysis of unloaded, carbon-loaded and regeneratesamples. During CO₂ absorption rich loading of 7.775mol/Kg whereas after regeneration lean-loading of 3.099 and 3.937mol/Kg were accomplished.FTIR analysis of regenerated sample re-confirms carbamate and bicarbonate presence indicatingsample requires further regeneration. Increase in density, viscosity and surface tension was observed during CO₂loading due to higher inter-molecular forcesbetween the solvent and CO₂ molecules and decreases during solvent regeneration due to CO₂ stripping.

1. Introduction

Global warming is caused by the emissions of CO₂ from various sources such as power generation sector, industries and transportation (Li *et al.*, 2016). As per the Inter-governmental Panel on Climate Change (IPCC) report of about 79% CO₂ mainly emitted from fossil fuels and minerals for power generation & 60% CO₂ emitted from coal power plants. (Aghaie *et al.*, 2018; Dashtiet *et al.*, 2019). The CCS is the promising way to significantly cut down the CO₂ emissions. Several CO₂ capture technologies, involved are chemical-absorption, adsorption, membrane techniques, cryogenic separation etc had been reported in Lei *et al.*, (2013); Perry and O'Brien, (2011); Rochelle *et al.*, (2001). The advantages of membrane separation such as high mass transfer, low energy consumption, simple operation and high separation efficiency. The limitation of membrane process is long processing time, fouling, non-selectivity, chemical inconsistencies with process fluids. Another capture technique was adsorption. It is extensively used in many industrial processes namely natural gas and H₂ gas. Some of the adsorbent used in CO₂ capture process are Selexol, Purisol, Rectisol, Morphysorb etc. The cons of this adsorption process are high operating cost and difficult regeneration process. By considering the above barriers Absorption is the suitable mechanism for removing the CO₂ in gas stream using chemical or physical absorbents. (Dashti *et al.*, 2019). However, chemical-absorption is prominently used CO₂ separation technology by an aqueous ethanolamines (mono, di, tri, and N-methyldiethanolamine)(Pellegrini *etal.*, 2011). Among all alkanolamines monoethanolamine act as a potential solvent in CO₂ removal process due to high absorption rate, low solvent cost (Akhmetshina *et al.*, 2019; Stec *et al.*, 2014; Liu *et al.*, 2015; Moioli *et al.*, 2012; Dugas, 2006;

Moioli *et al.*, 2013; Aboudheir *et al.*, 2003). The reaction between carbon dioxide and amine solution was maintained at 40°C and desorption at maximum temperature as 120°C examined by Rochelle, (2009). Aboudheir *et al.*, (2003) looked into the reaction kinetics among CO₂ and aqueous MEA over a temperature range of 293 to 333K, carbon loading ~0:1 to 0.49 mol/mol and concentration 3 to 9M in a laminar-jet scrubber by varying contact time within gas and liquid. The obtained results developed a novel ter-molecular kinetics example between CO₂ and MEA.

Moioli *et al.*, (2012) studied CO₂ capture using monoethanolamine (MEA). The chemical absorption has been influenced by thermodynamics, mass transfer and kinetics. The mass and heat transfer phenomena were detailly analyzed by rate- based model approach. With the help of Aspen Plus, model has been proposed to predict the mass transfer coefficients. The experimental data of exhaust gas from the power plant had used to validate the model. The result shows better improvement in the absorption process. Pellegrini *et al.*, 2011 investigated the energy reduction in CO₂ capture using MEA by simulation software (Aspen Plus). With the use of Electrolyte -NRTL thermodynamic package, equilibrium solubility of CO₂ -MEA-H₂O had been implemented. They compared the energy reduction between reboiler duty and total equivalent work/time. Energy saving has been increased by recycling the double column. The CO₂ absorption and desorption processes are not well discussed by the previous researchers. Some of the existing literature that are related to the mechanism of CO₂ in amine using FTIR analysis and the physico-chemical characteristics of the 30wt% monoethanolamine has been discussed below.

Jackson *et al.*, (2009) investigated on FTIR analysis about the reactions involved between CO₂ aqueous amines such as MEA and AMP to study the forward and reverse reaction. Richner and Puxty, (2012) studied the thermodynamics behind the amine-based CO₂ absorption using infrared monitoring process. In addition the reaction mechanisms of amine-based CO₂ capture in an industrial CO₂ capture plant was observed through FT-IR online monitoring.

Hartono *et al.*, (2014) investigated the physical properties such as density and viscosity of virgin and partially CO₂-loaded monoethanolamine solutions at the temperature range of (20 to 90°C) and up to 0.5 mol CO₂/mol MEA carbon-loading. The viscosities of aqueous monoethanolamine and triethanolamine were measured at a temperature range (25 to 80)°C and found to be decreased with increasing the temperature (Maham *et al.*, 2002). Recently, viscosities of an unloaded and CO₂-loaded MEA at 5 different CO₂ loading conditions were measured at a temperature range of 298.15 K to 373.15 K at every 5K increments. The obtained results revealed that the decreasing dynamic viscosity with an increasing the temperature was observed the reason being the high-temperature causes increased kinetic energy between the drifts between MEA and H₂O molecules. At higher temperatures, these molecules surpass intermolecular forces between the MEA and water resulting in lower viscosity (Idris *et al.*, 2017). The experimental investigation of density and surface tension were found to increase with an increasing the carbon loading, decreases with an increasing temperature was observed by Jayaratha *et al.*, (2013); Han *et al.*, (2012); Idris *et al.*, (2017). The effect of solvent concentration on surface tension were also discussed by Vazquez *et al.*, (1997) and it has been observed that the surface tension was high at a lower concentration

of solvent and found to be low at a higher concentration of the solvent.

Hence, the present study encompasses using FTIR spectroscopy, the CO₂ absorption and desorption using conventional regeneration (direct and indirect heating) were discussed to understand its mechanisms. In-addition the physicochemical characteristics such as carbon loading, viscosity, density, alkalinity, pH, and surface tension determined before and after absorption and desorption.

2. Materials and methods

2.1 Materials required

The chemicals, materials and equipments used in this experimental investigation are detailed explained in Nilavuckkarasiet *al.*, (2020)

2.2 Experimental procedure

2.2.1 CO₂ absorption

The schematic representation of gas absorption set-up is shown in Figure1 and the experimental design is shown in Table 1. 30wt% aqueous Monoethanolamine was used in this study. A gas mixture [15% CO₂ resting inert N₂] had been procured from the Bharath Oxygen Company, Chennai, India. The cylinder consists of 2 pressure gauge, one is to read the tank pressure and the other one is for process inlet pressure. The tank pressure gauge range is 0 - 3000 psi and tank outlet pressure gauge range is 0 – 15 psi. Present investigation 0.5 psi pressure was maintained during the CO₂ absorption process. Throughout the experiments the process pressure was maintained at desired pressure constantly with extra care. The detailed

experimental procedure reported in Nilavuckkarasiet *al.*, (2020) has been followed.

2.2.2 Solvent regeneration

The schematic drawing of solvent regeneration set-up is depicted in Figure 2a and 2b. In the direct heating, the heating element is direct contact with the glass reactor and for an in-direct heating, the glass reactor was placed in heating oil bath to reduce the skin temperature effect on the solvent. The experimental descriptions are discussed in Nilavuckkarasiet *al.*, (2020).

2.3 FTIR analysis

FTIR (Perkin Elmer model no 200) resolution was 400-4000 1/cm. The 30wt% MEA virgin, carbon loaded and conventionally regenerated (direct and in-direct heating) were subjected to FTIR spectroscopy investigation.

3. Results and discussion

In this work, at 0.5 psi pressure the absorption experiment was conducted followed by solvent regeneration, physicochemical analysis and FTIR characterization. The results are discussed in the following section.

3.1 Variation of reaction mixture temperature and CO₂ capture efficiency during carbon loading time

The reaction mixture temperature and CO₂ capture efficiency variation during carbon loading time was shown in Figure 3. Increasing the reaction mixture temperature while increasing the carbon loading time was observed. This is due to an exothermic reaction between

carbondioxide and MEA. The reaction mixture reached the maximum temperature of 46.3°C from 30.5°C initial reaction mixture temperature. Then the reaction mixture temperature diminishes slowly due to low-absorption capacity and heat loss to the surrounding. On the other hand, the capture efficiency decreases while increasing the carbon loading time. It was found that, first 80s of CO₂ charging, the capture efficiency is much greater than ninety percent due to unloaded aqueous MEA available for CO₂ absorption and high MEA concentration gradient, next 40s of loading, it came down to 65%. The solution becomes saturated at 88 minutes carbon loading with less than 1% capture efficiency due to a lesser extent of driving-force available for further CO₂ absorption process (Lvet *et al.*, 2015).

3.2 Temperature profile during solvent regeneration

Temperature profile during carbon-rich solvent regeneration using direct, in-direct heating are shown in Figure 4. The regeneration was conducted for about 1 hour. The first 16 minutes the temperature profile follows linearly increasing trend and then it stabilizes due to evaporative cooling. The observed maximum regeneration temperature during 1hr of direct and indirect heating were 108°C and 105.2°C. After solvent regeneration, the sample was cooled and subjected to physicochemical analysis. The results are discussed in the following section.

3.3 Physicochemical characteristics during CO₂ absorption and solvent regeneration

During CO₂ loading and solvent regeneration the physicochemical properties of solvents get changed. The change in physicochemical properties will affect the mass transfer kinetics. And it is significant to infer the properties of virgin MEA, carbon-rich and regenerated solvent for

further use. Hence the virgin, carbon rich and regenerated solvents was subjected to physicochemical properties such as pH, alkalinity, viscosity, carbon loading, density, and surface tension and are shown in Figure 5, 6 and 7. The detailed analysis of physicochemical properties of reproducibility has been carried out for 3 trials and the averaged data has been plotted with standard deviations.

3.3.1 Variation of pH and alkalinity during CO₂ absorption and solvent regeneration

Figure 5 shows the pH and alkalinity of unloaded, carbon-loaded and regenerated solution. Bench-top pH meter with an accuracy of 0.01pH was used to measure the sample pH. The pH of unloaded aqueous 30wt% MEA was 12.20 and the carbon-rich was 8.30 suggesting that the reaction between 30wt% MEA and CO₂ are acid-forming reaction. The pH of regenerated sample was 10.48 and 10.22 for direct as well as an in-direct heating. During solvent regeneration the CO₂ was removed thereby an increase in pH. Further regeneration is needed to achieve the unloaded MEA pH. Consequently the alkalinity of unloaded and carbon loaded MEA was 3.60 and 3.04 mol/Kg due to high carbonate ions present in the fresh MEA. During CO₂ absorption between MEA and CO₂ the carbamate formed as an intermediate products followed by hydrolysis of MEA + CO₂ to form carbonate and bicarbonate. In case of solvent regeneration due to temperature rise the CO₂ and few water vapors stripped from the carbon rich solution it exhibit high MEA concentration. The alkalinity of conventionally regenerated sample using direct and indirect heating were 3.96 and 3.88mol/Kg due to loss of some water vapor.

3.3.2 Variation of viscosity and density during CO₂ absorption and solvent regeneration

The viscosity and density of unloaded, loaded and regenerated sample are shown in the Figure 6. The viscosity was measured in mPas using Cannon Fenske opaque routine viscometer with an accuracy of $\pm 0.3\%$. Gravimetry method was used to measure the density of the sample (weighing balance - Sartorius BSA 224 S, Max 220g, 0.1mg accuracy). The unloaded and carbon loaded sample viscosity are 2.05 and 4.42mPa.s while the viscosity of conventionally regenerated direct as well as indirect heating was 3.72 and 3.57mPa.s. It was observed that high viscosity was found in carbon loaded sample, while rejuvenating the solution undergoes CO₂ desorption thereby it decreases the viscosity. During CO₂ absorption process, an increase in intermolecular forces between MEA, H₂O and CO₂ leads to high viscosity in carbon rich sample (Kummamuru *et al.*, 2019). At high temperature, inter-molecular forces between molecules get exceeded results in lower viscosity in regenerated sample (Idris *et al.*, 2017). Subsequently the density of virgin and carbon loaded sample found to be 1.022 and 1.146 g/cc. The reason being carbon loading increases the density of the solution however while solvent regeneration, the decrease in density of the solution was observed due to stripping of CO₂.

3.3.3 Variation of carbon loading and surface-tension during CO₂ absorption and solvent regeneration

The carbon loading and surface tension of unloaded, CO₂ loaded and regenerated sample was shown in the Figure 7. Carbon loading was measured in mol CO₂/kg solution using Chittick apparatus. The surface tension was measured in dyne/cm using Tensiometer. The carbon

loading and surface tension are interdependent physico-chemical properties. The surface-tension of sample was found to increase with an increase of carbon loading. While regeneration, the solution moves towards the CO₂lean conditions hence carbon loading decreases thereby decreasing the surface-tension of the solution.

3.4 FTIR analysis

3.4.1 FTIR spectra of virgin 30wt % MEA

The FTIR measurements were executed in an aqueous solution. From the Table 2 it is observed that the O-H stretching of H₂O strongly absorbs at 3290 1/cm and this peak was particularly broad due to hydrogen bonding of molecules. The smaller peak at 2626 1/cm on account of C-H stretching of molecules (Einbuet *al.*, 2012). The primary amine shows the vibration characteristics modes at 1643 1/cm. Inadequately, there are C-O stretch at 1076 1/cm and the C-N stretch at 1028 1/cm. The peak 1319 1/cm is ascribed for CO₃²⁻ shown in Figure 8.

3.4.2 FTIR spectra of carbon loaded 30wt % MEA

The O-H stretching of H₂O strongly absorbs at 3294 1/cm. The primary amine at 1639 1/cm, at the same time with peaks of the –C-O stretch at 1159 1/cm and the –C-N stretch at 1018 1/cm showed in Figure9& Table 3. When CO₂ is absorbed by MEA it indicates the MEA – carbamate peaks and it was attributed to COO- symmetric and asymmetric elongating at 1564 1/cm and 1489 1/cm (Einbu *et al.*, 2012). These carbamate peaks are absent in Virgin MEA. At the end of the CO₂absorption of MEA, the sample contains mainly carbonate and bicarbonate this peak such as 1391 1/cm is assigned for CO₃²⁻ and 1320 1/cm for HCO₃⁻. From this result, it

showed that the CO₂ is efficiently absorbed by monoethanolamine.

3.4.3 FTIR spectra of regenerated solvent - direct heating

The FTIR spectra of direct heating of carbon rich solution was shown in Table 4 and Figure 10

It was observed that the O-H extending of H₂O absorbs at 3314 1/cm. The smaller peak at 2829 1/cm due to Carbon-Hydrogen atom stretching of molecules and this is similar to virgin MEA.

This reveals that carbon loaded MEA undergo regeneration (Jackson *et al.*, 2009). The

primary amine shows a peak at 1645 1/cm, unitedly with peaks of the –C-O stretch at 1074 1/cm and –C-N extend at 1163 1/cm. The presence of carbamate peaks was attributed to

COO- asymmetric and symmetric elongating at 1572 1/cm and 1492 1/cm and peak at 1315 1/cm ascribed for HCO₃⁻ because the carbon loaded MEA are not completely regenerated.

3.4.4 FTIR spectra of regenerated solvent - indirect heating

From the Table 5 and Figure 11 it was observed the O-H adulterating of H₂O absorbs at 3308 1/cm. These peaks are broad due to H₂ bonding. Virgin MEA has a peak at 2626 1/cm was

formed due to stretching of Carbon-Hydrogen molecules and it is absent in carbon loaded MEA due to lack of C-H stretching. During regeneration process, stripping of CO₂ occurs

which promote the MEA and H₂O interactions thereby the regenerated sample delivers a carbon-Hydrogen stretching peaks. It indicates partial regeneration is accomplished. (Einbuet

al., 2012; Jackson *et al.*, 2009). The primary amine show characteristics oscillation fashions at

1645 1/cm. Furthermore, there were –C-O and –C-N elongatingfashions at 1073 1/cm and 1154 1/cm respectively (Jackson *et al.*, 2009). The carbamate peaks were attributed to COO-

asymmetric and symmetric elongating at 1588 $1/\text{cm}$ and 1482 $1/\text{cm}$ and peak assign for HCO_3^- as 1315 $1/\text{cm}$. The presence of carbamate and bicarbonate peaks reveal that the Carbon loaded MEA is not completely regenerated.

4. Conclusions

The physicochemical analysis and FTIR spectroscopic of unloaded, loaded and regenerated (direct and in-direct heating) 30wt% MEA solutions were experimentally measured and investigated to understand CO_2 absorption and stripping phenomena. First 80s of CO_2 loading, the absorption efficiency was much higher than 90% later it decreases due to less driving force available for further mass transfer.

During CO_2 absorption rich loading of 7.775mol/Kg whereas after regeneration lean loading of 3.099 and 3.937mol/Kg were accomplished. The pH of 30wt% MEA was 12.2, whereas pH of CO_2 loaded solution was 8.3. This is due to acid formation between CO_2 and MEA. But the pH was increased during solvent regeneration due to removal of CO_2 .

Increase in density, viscosity and surface tension was observed during CO_2 loading due to higher inter-molecular forces between the solvent and CO_2 molecules and decreases during solvent regeneration due to CO_2 stripping.

FTIR spectra analysis of virgin sample revealed presence of primary amine and carbonate ions whereas the carbon loaded sample showed carbamate, carbonate, bicarbonate and other primary amine. In the case of regenerated sample, the presence of carbamate and bicarbonate indicates that the carbon rich 30wt% MEA requires further regeneration.

Highlights

- CO₂ absorption and conventional regeneration using aqueous MEA.
- Detailed physicochemical analysis of virgin, carbon-rich and conventionally regenerated samples.
- Understanding the mechanisms of CO₂ absorption and regeneration through FTIR characterization.

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Table 1. Experimental design

S.No	Experimental run	Parameter	
1	CO ₂ absorption	15 vol%CO ₂ remaining N ₂ gas 0.5Psi Inlet gas pressure, 88minutes	-
2	Solvent regeneration	Direct Heating - 60minutes In-Direct Heating - 60minutes (K type thermocouple for temperature measurement)	-
3	Physicochemical analysis	pH (Bench-top pH meter, 0.01pH accuracy) Density (Gravimetric analysis using Sartorius weighing balance 0.1mg accuracy) Carbon loading (Chittick apparatus) Viscosity (Cannon Fenske Opaque Viscometer $\pm 0.3\%$ accuracy) Surface tension (Tensiometer)	Prior to absorption, after absorption and regeneration
4	FTIR characterization	Resolution 400-4000cm ⁻¹	Prior to absorption, after absorption and regeneration

Table 2. FTIR spectra of virgin 30wt % MEA

Sample	Frequency	Stretching
MEA	3290	O-H
	2626	C-H
	1643	Primary Amine
	1319	CO ₃ ²⁻
	1076	C-N
	1028	C-O

Table 3. FTIR spectra of carbon loaded 30wt % MEA

Sample	Frequency	Stretching
CL MEA	3294	O-H
	1639	Primary amine
	1564	Asymmetric stretching of COO ⁻
	1489	Symmetric stretching of COO ⁻
	1391	CO ₃ ²⁻
	1320	HCO ₃ ⁻
	1159	C-N
	1018	C-O

Table 4. FTIR spectra of regenerated carbon loaded 30wt % MEA - direct heating

Sample	Frequency	Stretching
Direct Heating	3314	O-H
	2829	C-H
	1645	Primary amine
	1572	Asymmetric stretching of COO ⁻
	1492	Symmetric stretching of COO ⁻
	1315	HCO ₃ ⁻
	1163	C-N
	1074	C-O

Table 5. FTIR spectra of regenerated carbon loaded 30wt% MEA - indirect heating

Sample	Frequency	Stretching
In-Direct heating	3308	O-H
	2874	C-H
	1645	Primary amine
	1588	Asymmetric stretching of COO ⁻
	1482	Symmetric stretching of COO ⁻
	1315	HCO ₃ ⁻
	1154	C-N
	1073	C-O

Figure 1. Schematic representation of CO₂ absorption experimental set up

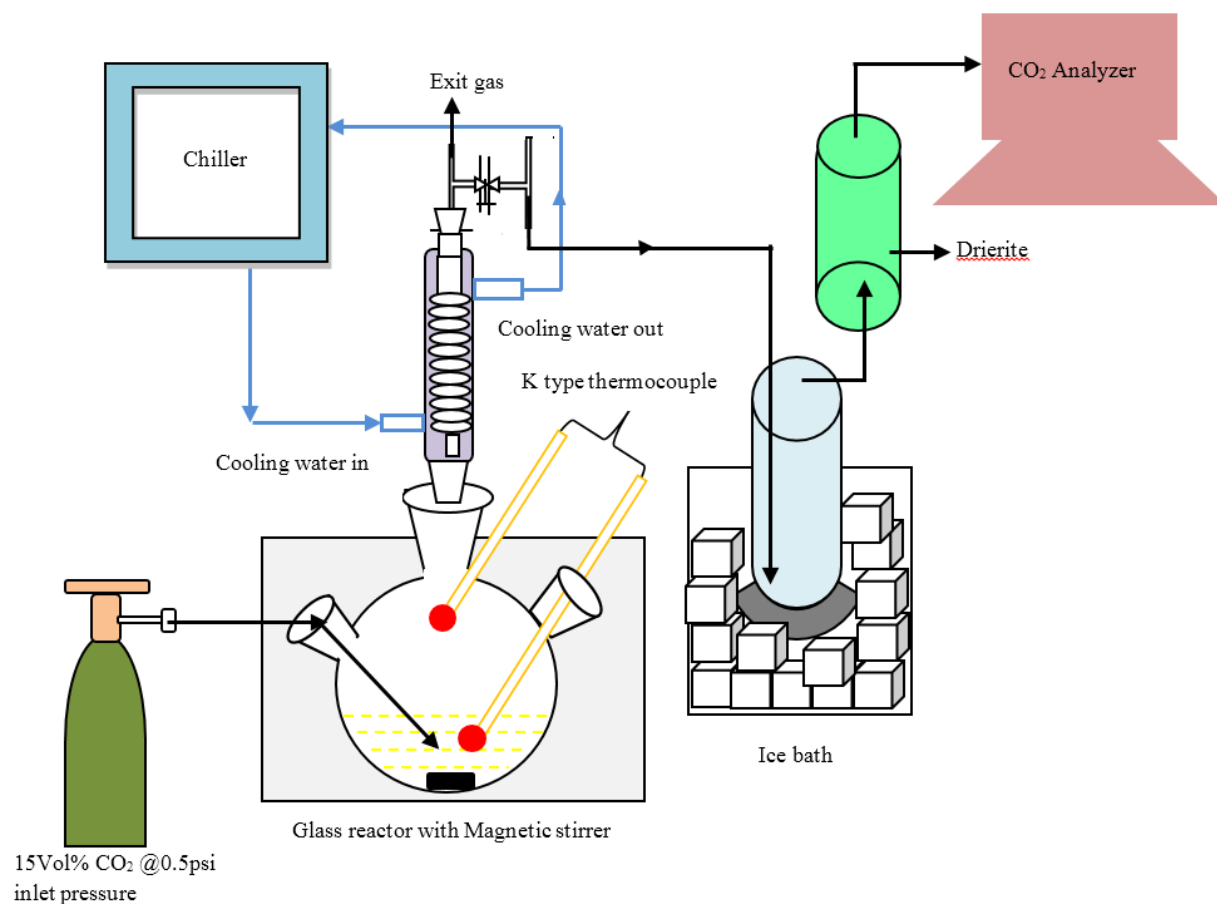
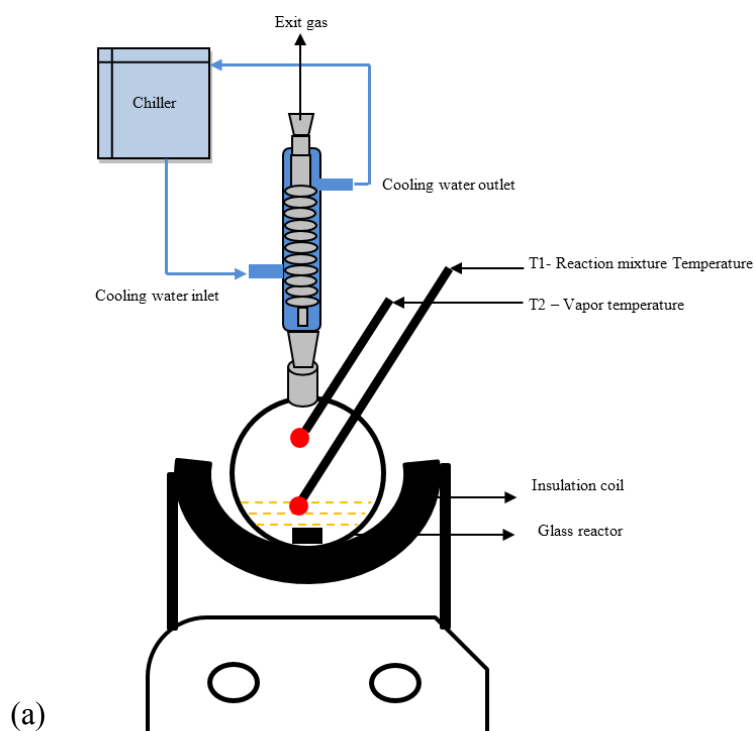
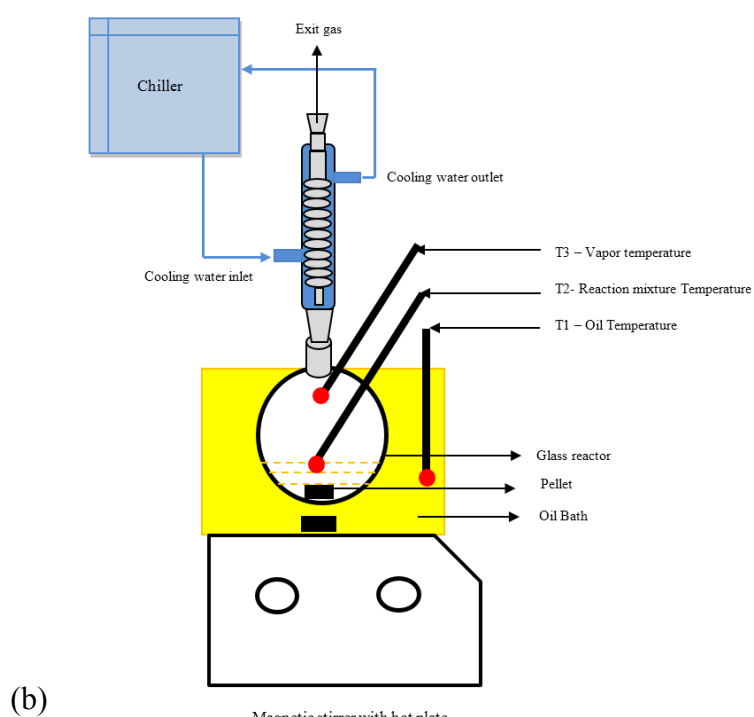


Figure 2. (a) Schematic diagram of conventional regeneration- direct heating. (b) Schematic diagram of conventional regeneration - indirect heating



Magnetic Stirrer with hot plate



Magnetic stirrer with hot plate

Figure 3. Variation of reaction mixture temperature and CO₂ capture efficiency during carbon loading time

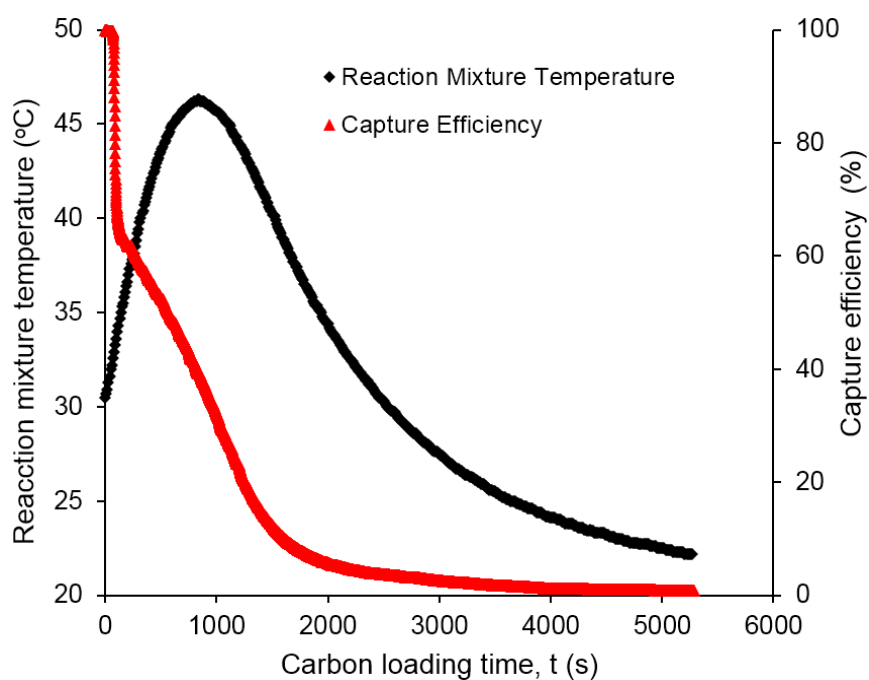


Figure 4. Temperature profile during carbon rich solvent regeneration

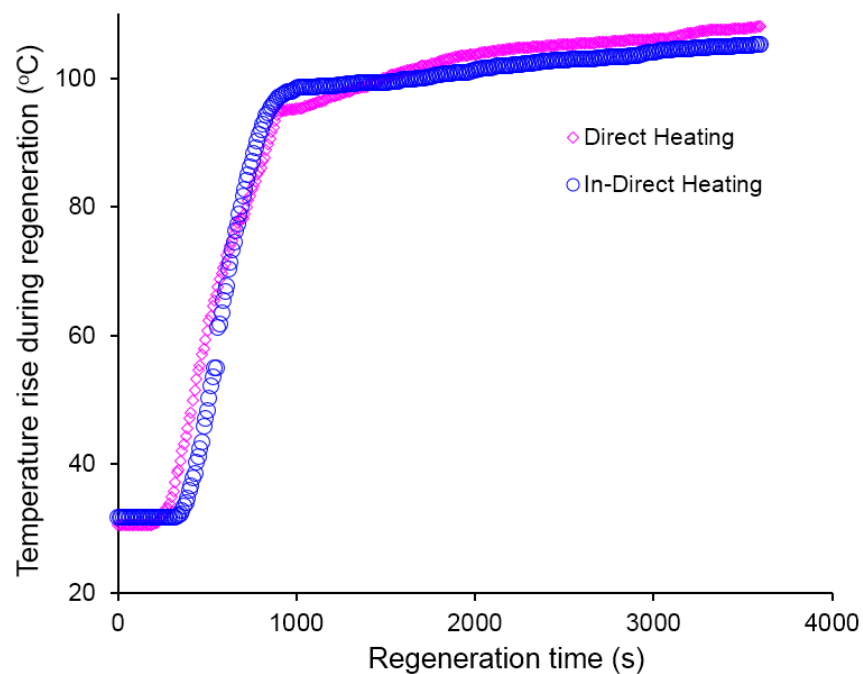


Figure 5. Variation of pH and alkalinity during CO₂ absorption and regeneration

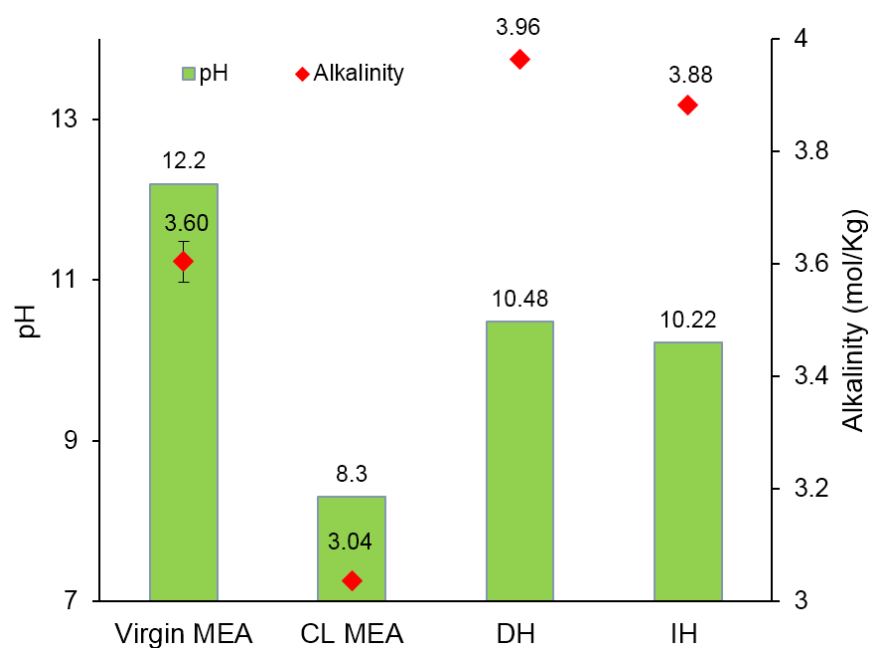


Figure 6. Variation of viscosity and density during CO₂ absorption and regeneration

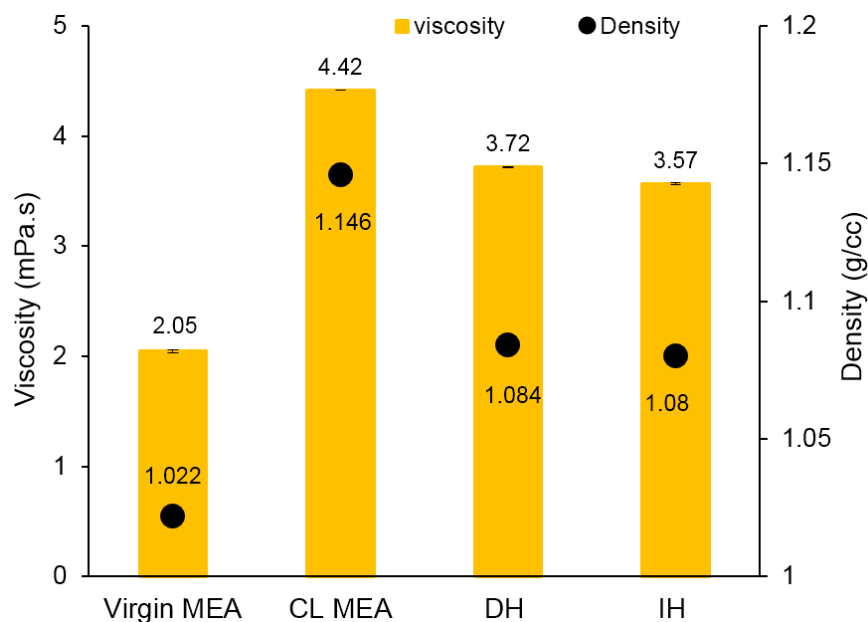


Figure 7. Variation of carbon loading and surface tension during CO₂ absorption and regeneration

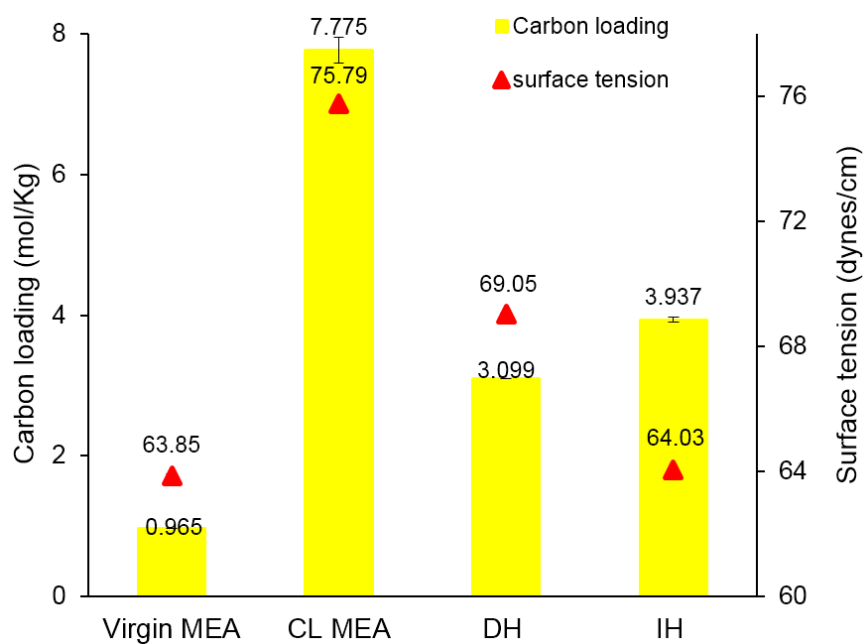


Figure 8. FTIR spectra of 30wt% MEA

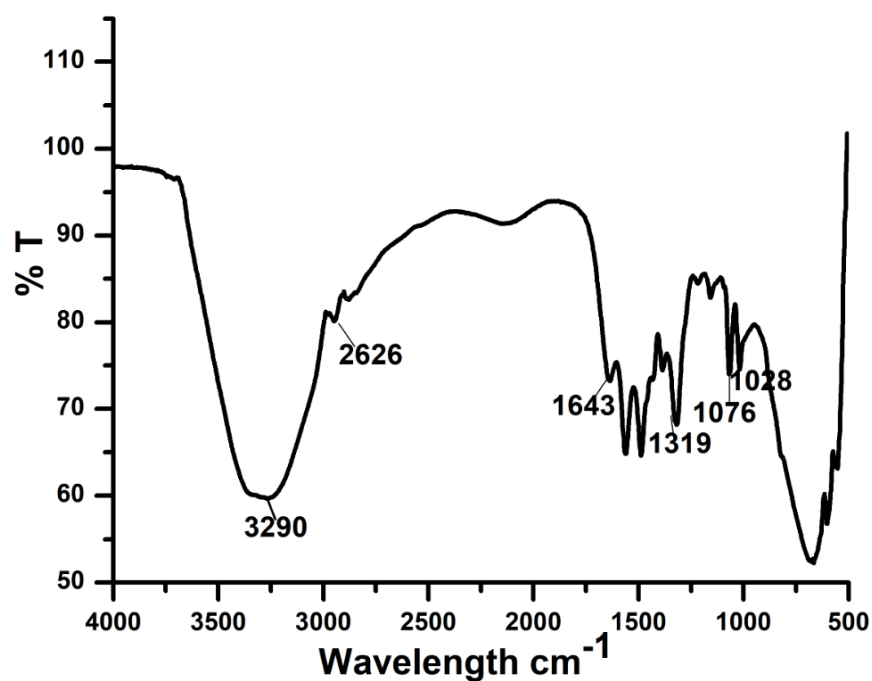


Figure 9. FTIR spectra of carbon loaded 30wt% MEA

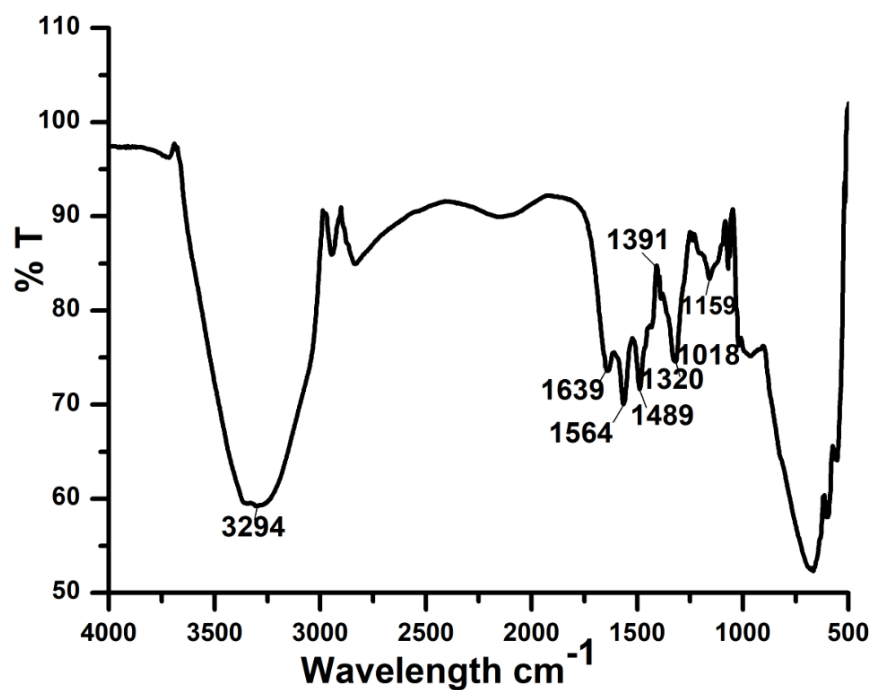


Figure 10. FTIR spectra of direct heating of carbon loaded 30wt% MEA

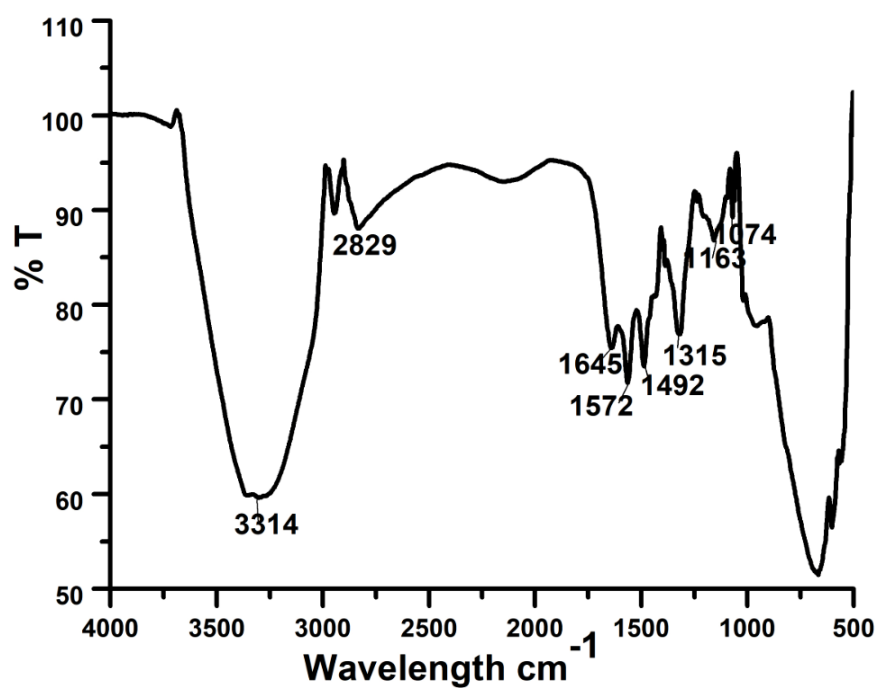


Figure 11. FTIR spectra of in-direct heating of carbon loaded 30wt% MEA

