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Operation of a Pilot-Scale CO_2 Capture Process with a New Energy-Efficient Polyamine Solvent

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Abstract: A new blending recipe of a polyamine-based solvent for capturing post-combustion CO_2 was proposed, and its performance and characteristics were investigated using a pilot-scale carbon capture process (PCCP). The proposed solvent is a blend of three types of amines and was designed to separate the solvent roles into those of a main amine, auxiliary amine, and reaction-rate-enhancing amine. Polyamine 3,3′-iminobis (N, N-dimethylpropylamine) was selected as the main amine given its ability to capture large amounts of CO_2. 2-Amino-2-methyl-1-propanol was used as the auxiliary amine, with piperazine added as the reaction-rate-enhancing amine. This solvent was tested in a PCCP that can handle 150 Nm^3/h of flue gas. The proposed solvent was found to operate stably while consuming substantially lower reboiler duty than the monoethanolamine (MEA) 30 mass% solvent.

Keywords: CO_2 capture; blending amine solvent; pilot carbon capture process

1. Introduction

Humankind faces an unprecedented challenge with regard to achieving climate objectives. Without carbon capture and storage (CCS), this challenge becomes infinitely greater [1]. CCS is a technology capable of capturing CO_2 from large-scale CO_2 emission sources and storing it in the ground. Post-combustion capture processes can be classified into three types: absorption, adsorption, and membrane technologies. The most commercially available technique is absorption, which typically uses various amine solvents [2–4]. The types of amines used in the solvents are divided into primary, secondary, and tertiary amines [5]. In general, CO_2 absorption is achieved with primary and secondary amines through a carbamate reaction and with tertiary amines through a bicarbonate reaction [6]. Primary and secondary amines can also form bicarbonate but only a small amount after the carbamate reaction is completed. In the past, a single amine such as monoethanolamine (MEA) and water were mixed and used commercially as a CO_2 capture solvent, but researchers have investigated the blending of various amines to improve the performance of the solvent. A typical example is the BASF's aMDEA®, a blend of methyl diethanolamine (MDEA) and piperazine (PZ), and research on the optimal mixing ratio of two amines is also being conducted through various methods [7].

In this study, we attempted to develop a solvent with low reboiler heat duty by blending several types of amines. For high CO_2 loading, we decided through a literature review and by reviewing existing experiments to use polyamine 3,3′-iminobis (N, N-dimethylpropylamine) (IBDMPA), a polyamine, as the main amine. Hereafter, we use IBD to denote IBDMPA for simplicity of presentation. As a triamine, one mole of IBD can react with up to two moles of CO_2 which is four times that of one mole of MEA reacting with 0.5 mole of CO_2 [8,9]. Like aMDEA®, PZ was selected as the reaction rate enhancer. Research on increasing the reaction rate by blending PZ with IBD was conducted by Aronu et al. [10] and Kim et al. [11]. The use of a high concentration of IBD leads to high viscosity of the solvent. Therefore, supplementing IBD and PZ with a non-high viscosity auxiliary amine was considered. MEA, 2-(butylamino)ethanol (BAE), and 2-amino-2-methyl-1-propanol (AMP) were chosen as auxiliary amine candidates.

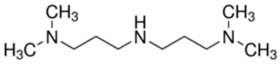
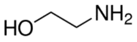
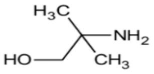
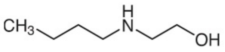
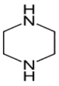
Comparing CO_2 solubility, absorption rate, and viscosity measurements in the laboratory, we finally chose a blend, IBD-AMP-PZ of 40:6:6 mass%, as a new recipe, and test operations were conducted in a pilot-scale carbon capture process (PCCP) that can normally handle 150 Nm^3/h of flue gas. These tests sought to investigate the stability of operation, the reboiler heat duty, the CO_2 loading, and the temperature profile in the absorber column at a constant flue gas flow rate. To compare the performance of the proposed solvent, a baseline evaluation was conducted first using MEA 30 mass% solvent.

2. Three IBDMPA-Based Amine Blends

2.1. Materials

Table 1 shows four individual amines associated with this research. IBD is a polyamine containing one secondary and two tertiary amines, known to have a large working capacity and relatively small heat of reaction [11]. MEA is the most representative primary amine with the akanolamine structure. A relatively fast CO_2 absorption rate and low price are advantages. AMP is a sterically hindered amine that has high absorption capacity [12,13]. BAE has a molecular structure of an alkanolamine including a secondary amine; the working capacity is relatively high, and the heat of reaction is low compared to MEA [11]. PZ is a representative amine species that can increase the CO_2 absorption speed when mixed with other amine solvents [14].

Table 1. List of amines associated with this study.

Name	Structure Formulation	M.W. (g/mol)	BP (°C)	CAS No.
3,3′-Iminobis (N,N-dimethyl propylamine) (IBDMPA or IBD)		187.33	239.4	6711-48-4
Monoethanolamine (MEA)		61.08	170	141-43-5
2-Amino-2-methyl-1-propanol (AMP)		89.14	165.5	124-68-5
2-(Butylamino)ethanol (BAE)		117.19	199	111-75-1
Piperazine (PZ)		86.14	146	110-85-0

The main amine employed in the present work was IBD, supplied by Huntsman Co., with a purity rating of >98%. MEA, AMP, and PZ were purchased from Sigma Aldrich, all with purity levels of >99%. Carbon dioxide and oxygen were supplied by Daechung Co., Korea with purity levels of 99.9%.

2.2. Solvent Formulation

Amine types and concentrations of the new blends are shown in Table 2. The first consists of IBD, MEA, and PZ with a ratio of 30:20:6 mass% and was named IMP using the initials of the three constituent amines. In addition, purified water was used as the formulation solvent. The second and third ones include AMP and BAE instead of MEA and were named IAP and IBP, respectively. The amine concentrations of IAP and IBP are different from those of IMP. In formulating IAP and IBP, we attempted to increase the total amine concentration by increasing IBD first and then increasing the auxiliary amine using the guideline that the maximum viscosity should not exceed 30 cP at 40 °C.

Table 2. Amine types and concentrations of the investigated amine blends.

Name	Concentrations of Amines
IMP	IBD 30 mass% + MEA 20 mass% + PZ 6 mass%
IAP	IBD 40 mass% + AMP 6 mass% + PZ 6 mass%
IBP	IBD 40 mass% + BAE 6 mass% + PZ 6 mass%

For the solvent viscosity screen, the existing literature has proposed less than 20 cP at 40°C [15]. However, in this study, the viscosity criteria for solvent selection were determined to be less than 30 cP at rich load at 40°C. In fact, it was confirmed that operation is possible when it is less than 30 cP in the PCCP.

2.3. Characteristics of the Solvents

2.3.1. Experimental Methods

The blended solvents were investigated in the lab first. The probed properties were CO_2 solubility, CO_2 absorption rate, and solvent viscosity, and one between IAP and IBP was selected together with IMP on the basis of the probed properties for the next stage pilot process test.

The CO_2 solubility was measured using a continuous flow bubbling reactor (CFBR). Different experimental methods were used with the CFBR depending on the equilibrium CO_2 partial pressure above and below 1 kPa. More details on the CFBR method can be found in Kim et al. [11].

A modified wetted wall column (m-WWC) was used to measure both the CO_2 absorption rate and the solvent viscosity by continuously increasing the CO_2 loading of the solvent. More details on the m-WWC device are described in Kim et al. [16].

2.3.2. Solvent Selection

The CO_2 solubility data for the three amine blends are presented in Figure 1. The CO_2 solubility curves for IAP and IBP almost overlapped. The cyclic CO_2 loading capacity at 40 °C based on the equilibrium CO_2 partial pressures of 0.05 and 5 kPa is 65 g- CO_2/kg-solvent for IMP and about 54 g-CO_2/kg-solvent for IAP and IBP.

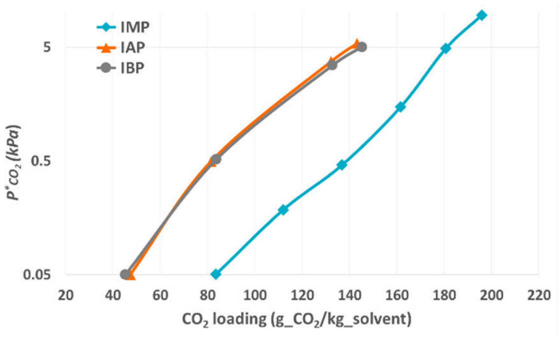
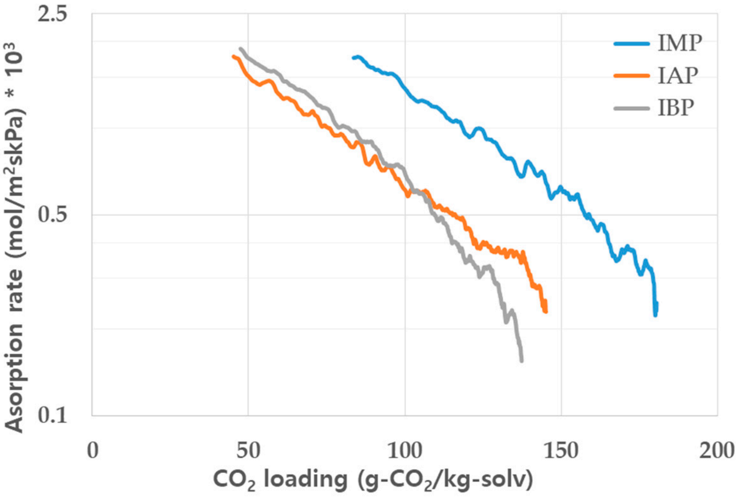
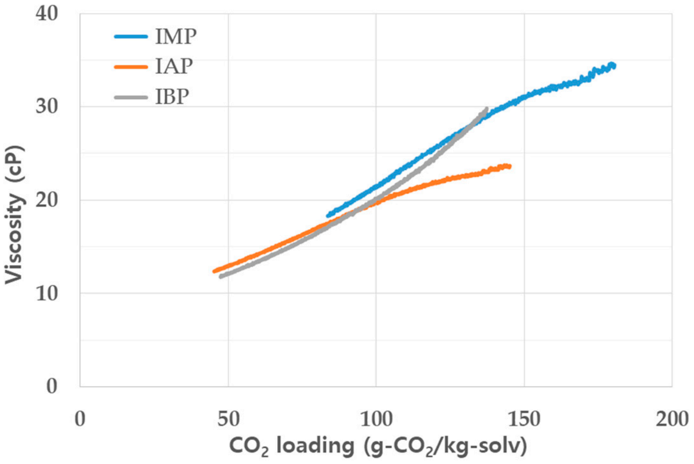


Figure 1. CO_2 solubility of the three amine blends at 40 °C.

Figure 2a shows the CO_2 absorption rate and Figure 2b shows viscosity of the three solvent candidates at 40 °C. All three solvent candidates show similar values and patterns of the overall mass transfer coefficient with CO_2 loading. However, IBP was found to have the slowest CO_2 absorption among the three.



(a)



(b)

Figure 2. (**a**) The CO_2 absorption rate in terms of the overall mass transfer coefficient. (**b**) Viscosity of the investigated amine blends.

The viscosity increased with increasing CO_2 loading. IMP showed a viscosity increase up to 34 cP at rich loading (corresponding to 5 kPa), while IAP and IBP exhibited lower viscosity values than IMP over the working CO_2 loading range. In the rich loading, IAP slowed the trend of increasing viscosity, showing 24 cP, while IBP accelerated the increasing trend and reached 30 cP.

Table 3 lists the average CO_2 absorption rates and rich loading viscosities at 40 °C of the three amine blends. Based on the selection criteria, the authors decided to operate with IAP, the solvent with the lowest viscosity and relatively fast reaction rate performance, in a pilot process.

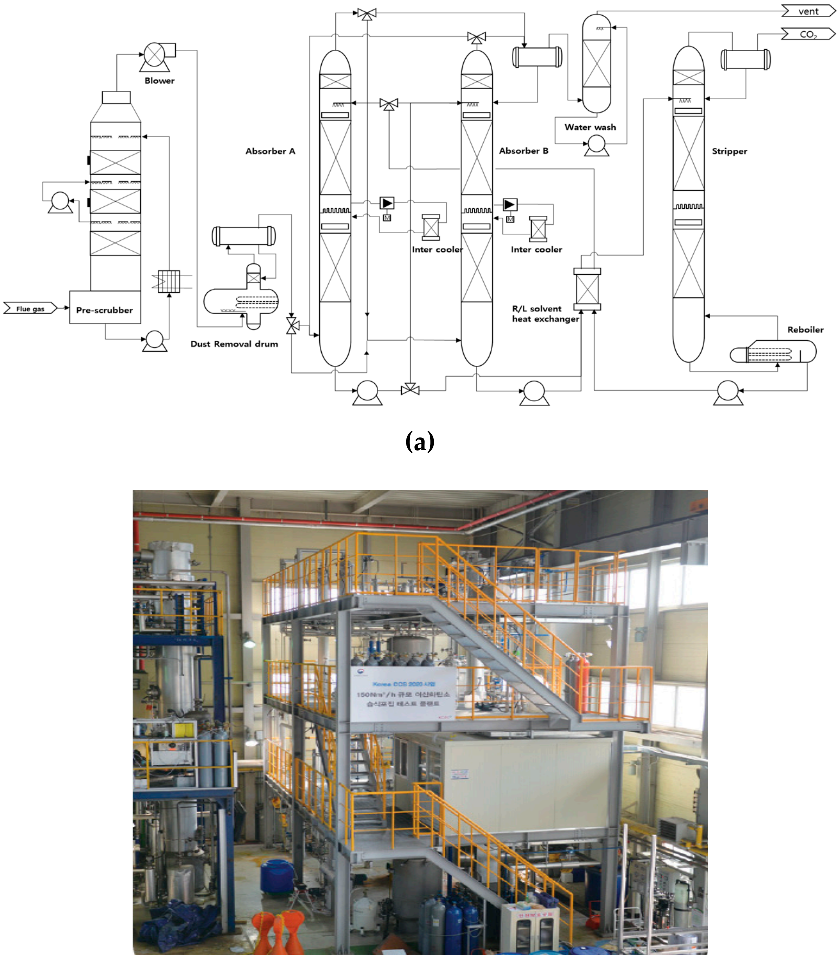
Table 3. Average overall mass transfer coefficient and rich loading viscosity values of the investigated amine blends at 40 °C.

Solvent	Average Overall Mass Transfer Coeff. at 40 °C (mol CO_2/m^2 s kPa)	Rich Loading Viscosity at 40 °C (cP)
IMP	5.45×10^{-4}	34.49
IAP	5.15×10^{-4}	23.68
IBP	4.46×10^{-4}	29.09

3. Test Operations in the Pilot CO_2 Capture Process

3.1. Description of the Pilot Process

The pilot CO_2 capture process (PCCP) was built at the site of the 2MW coal-fired power plant of the Korea Institute of Energy Research and could be tested with actual real flue gas or with a simulated gas. The PCCP was designed to normally treat 150 Nm^3/h of flue gas with a CO_2 capture rate exceeding 90% using the MEA 30 mass% solvent, though it can also be operated with various other solvents. In consideration of corrosion, all major equipment and piping were manufactured with stainless steel (SUS316). The stripper column and reboiler were designed to be able to operate at up to 7 barg, as some solvents can be regenerated under pressure. A schematic of the PCCP is shown in Figure 3, which also presents a photograph of the PCCP.



(a)

(b)

Figure 3. (**a**) Process flow diagram and (**b**) photograph of the PCCP(Pilot CO_2 Capture Process).

Key units in the PCCP are described in the following:

Pre-scrubber: This unit removes SO_2 together with dust in the flue gas. It was designed as a packed column with CMR(CASCADE MINI-RINGS)® random packing materials. An aqueous solution of NaOH is used to reduce the concentration of SO_2 to less than 2 ppm. The gas treated in the pre-scrubber passes through a dust removal drum for further removal of dust. The bed dimensions and packing materials of the packed columns are described in detail in Table 4.

Table 4. Bed dimensions and packing materials of the packed columns in the PCCP.

Column	Total Bed Height (cm)	Bed Diameter (cm)	Number of Bed Sections	Packing Types
Pre-scrubber	380	40	3	CMR, 1 inch
Absorber A	500	25	2	CMR, 1 inch
Absorber B	500	25	2	TPT 350X
Water wash	120	30	1	PALL ring, 1 inch
Stripper	410	20	2	CMR, 1 inch

Absorber columns: Two absorber columns were installed for a flexible configuration. The inner diameter of each column is 25 cm and the total height is 835 cm with two 250 cm packing beds. An intercooler was installed in the middle of each column. The two columns were each filled with different packing materials of TPT 350X structure packing, similar to Mellapak 350X and CMR random packing. The absorber columns can be operated in series by changing the order, or each absorber column can be operated independently.

Stripper column: The inner diameter of the stripper column is 20 cm, and the height is 790 cm, of which 410 cm is accounted for by the packing bed. The bed was filled with CMR random packing materials. The top of the stripper column was equipped with gauze-type structure packing to reduce the entrainment during feed flash. The exhausting vapor from the column is cooled to recover the solvent and water vapor by a shell and tube condenser. The reboiler was designed as a kettle-type.

Cross-heat exchanger: A plate heat exchanger was installed to efficiently recover the thermal energy in the hot lean loading solvent to the cold rich loading solvent.

Analysis: Table 5 shows on-line gas sensors installed in the PCCP. In addition to these sensors, gas and liquid samples were taken periodically and analyzed in the lab to measure CO_2 loading, solvent decomposition products, heat stable salts, and selected components in the absorber exhaust gas.

Table 5. Gas analyzers in the PCCP.

Stream	Species	Technique	Range
Absorber inlet	CO_2	NDIR	0~20 vol%
	SO_2	NDIR	0~500 ppm
	O_2	Zirconia sensor	0~100 vol%
	CO	NDIR	0~300 ppm
	NO_x	NDUV	0~550 ppm
	Moisture	Capacitance sensor	
Absorber outlet	CO_2	NDIR	0~20 vol%
	O_2	Paramagnetic sensor	0~21 vol%
Stripper outlet	CO_2	NDIR	0~100 vol%
	Moisture	Capacitance sensor	

3.2. Operating Conditions

To compare the performance of the new amine blends, a baseline evaluation was conducted first using MEA 30mass%. For simplicity, MEA 30mass% will be indicated hereafter as MEA when there is no possibility of confusion.

Table 6 presents the operating conditions covered during the test of the MEA and the new amine blend, IAP. In this operation, a simulation gas with the same CO_2 concentration in flue gas of a coal-fired power plant was used. Actual coal-fired flue gas is not used here in order to keep the concentration of CO_2 constant for reboiler heat duty measurement. The solvent circulation rate was varied over a range to find an optimum condition that required minimum reboiler heat duty. At each solvent circulation rate, the reboiler temperature was adjusted to achieve 88–92% of CO_2 capture.

Table 6. Operating conditions for the PCCP tests.

Variables	MEA 30 mass%	IAP
Flue gas flow rate	150 Nm^3/h	
Flue gas temperature	36–42 °C	
CO_2 in the flue gas	14 vol%	
O_2 in the flue gas	16.5 vol%	
H_2O in the flue gas	7 vol%	
Solvent flow rate	400–720 L/h	200~800 L/h
Lean solvent temperature entering the absorber column	40 °C	
Stripper pressure	100 kPag	
Target CO_2 capture rate	88–92%	88–92%

4. Results and Discussion

4.1. Time-Dependent Operation Trajectory

Figure 4 shows the trajectory of feed gas flow rate and CO_2 capture rate for 1000 h of continuous operation. During the entire operating period, the feed gas flow rate was stably maintained at 150 Nm^3/h. The CO_2 capture rate was relatively well controlled around 90% except around 200 h, when a sensor malfunction occurred. Overall, the average CO_2 capture rate was 89.6%.

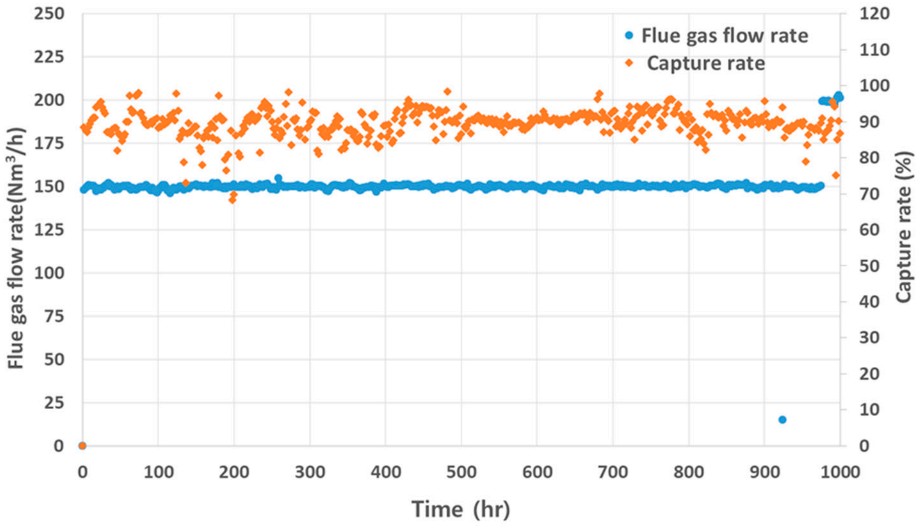


Figure 4. Feed gas flow rate and CO_2 capture rate from IAP operation over 1000 h.

4.2. CO_2 Capture Performance

Figure 5 shows the rich and lean loading outcomes for IAP at various solvent circulating flow rates. For the IAP, CO_2-rich loading was measured from 84 to as high as 133 g- CO_2/kg-solvent at a 150 Nm^3/h flue gas flow rate. The CO_2 working capacity at L/G = 1.7 L/Nm^3, the lowest energy operating condition, was analyzed to be 126 g- CO_2/kg-solvent. The working capacity of the IAP is calculated to be 268% greater than that of MEA 30 mass% at optimum operating conditions of each solvent. The IAP was measured to have a significantly lower CO_2 lean loading value of 4 g-CO_2/kg-solvent under optimum operating conditions.

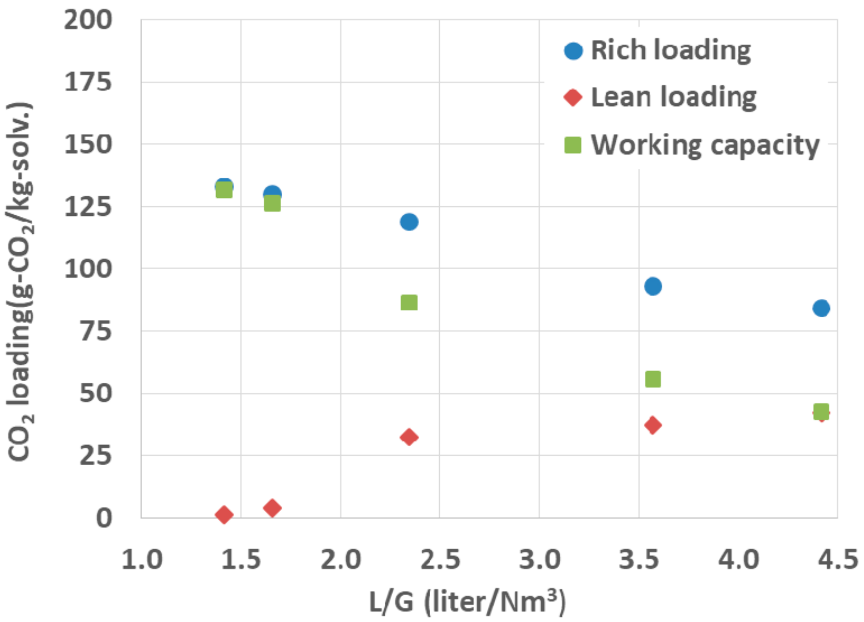


Figure 5. Rich/lean loading and working capacity results depending on L/G for IAP.

4.3. Reboiler Heat Duty

The reboiler heat duty of IAP was measured at various solvent flow rates, and the minimum reboiler heat duty was 3.3 GJ/t- CO_2 at L/G of 1.7 as shown in Figure 6. This is lower than the value for MEA by 0.7 GJ/t- CO_2. The optimum L/G of 1.7 for IAP indicates that the same amount of CO_2 could be captured with only about 40% of the solvent flow rate compared to the optimum L/G of 4.3 for MEA at a 150 Nm^3/h flue gas flow rate.

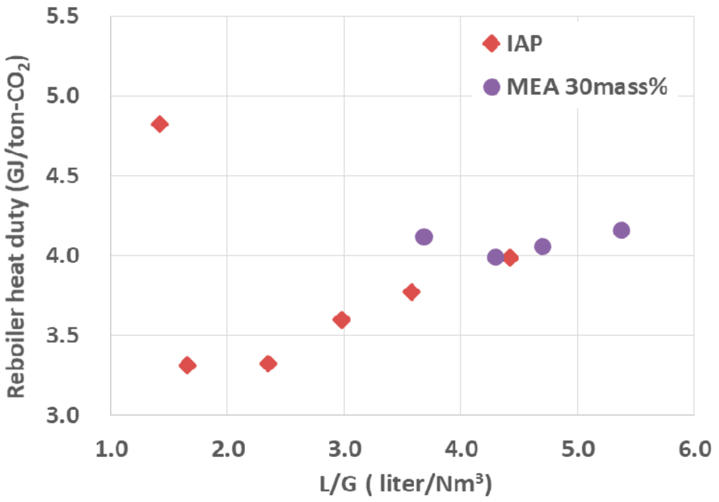


Figure 6. Reboiler heat duty outcomes for MEA 30mass% and IAP.

The PCCP has significant heat loss compared to commercial processes. In fact, during the operation of a 0.5 MW process employed by our research group, the minimum reboiler heat duty for MEA was measured as 3.6 GJ/t- CO_2, showing a gap of 0.4 GJ/t- CO_2 compared with the result in the PCCP. Based on this observation, IAP can be assessed to have minimum reboiler heat duty lower than 2.9 GJ/t- CO_2.

Table 7 compares CO_2 loading values of the lean state, mid-rich state (after the first column), and rich state (after the second column) at optimum operating conditions for MEA and IAP. It can be seen that IAP reaches 82.5% of the working capacity in the first column and achieves the remaining

17.5% in the second column. On the other hand, MEA attains 55% and 45% of the working capacity in the first and second columns, respectively. These results show that IAP possesses a faster CO_2 absorption rate than MEA.

Table 7. CO_2 loadings at different locations for minimum reboiler heat duty.

Solvent	L/G	Lean Loading	CO_2 Loading after the 1st Column	CO_2 Loading after the 2nd Column	Working Capacity	Reboiler Heat Duty
	L/Nm^3	g-CO_2/kg-Solvent				GJ/t-CO_2
MEA 30 mass%	4.3	51	75	95	44	4.0
IAP	1.7	4	108	130	126	3.3

4.4. Column Temperature Profiles

The temperature profiles in the absorber and the stripper columns were compared under the optimum operating conditions of IAP and MEA in Figure 7. For the case of IAP, a temperature peak appeared soon after the lean solvent entered the absorber column and temperature dropped and remained relatively flat. On the other hand, temperature for MEA along the absorber bed increased slowly and remained high throughout the entire bed compared to the case of IAP. The result is obviously a manifestation of the higher CO_2 absorption rate of IAP than MEA, which is shown in Table 7.

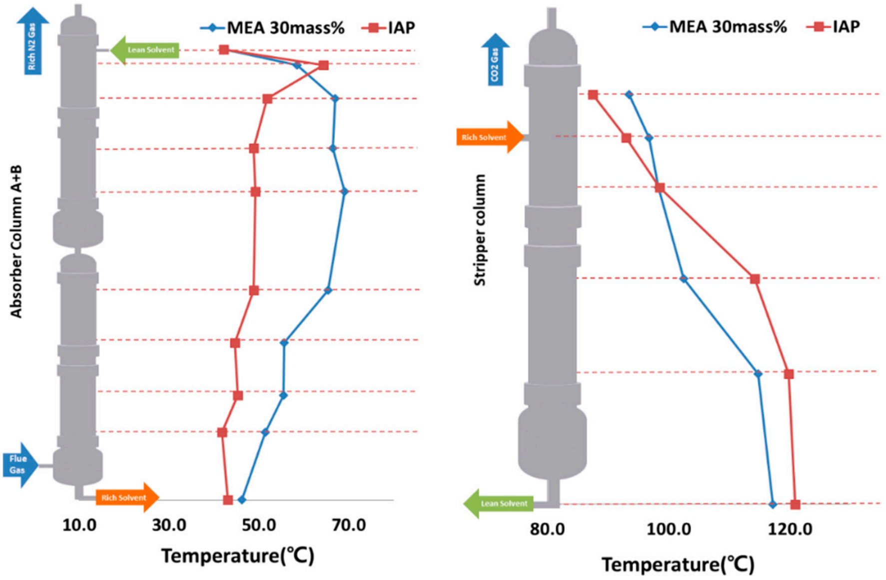


Figure 7. Temperature profiles in the absorber and stripper columns under the optimum operating conditions for MEA and IAP.

Temperature profiles in the stripper column indicate that IAP yielded higher temperature than MEA in the bottom region but lower temperature than MEA in the upper region. In the stripper, the mass transfer rate was quite high due to high temperature.

5. Conclusions

In this study, a new blending recipe of a polyamine-based solvent for the capturing of post-combustion CO_2 was proposed, and its performance and characteristics were investigated using a pilot-scale carbon capture process (PCCP). The proposed solvent was derived from three different IBD-based blends. All three blends contained PZ as a reaction-rate-enhancer and one of MEA, BAE, or AMP as an auxiliary amine to increase the total amine concentration while not increasing the viscosity substantially. Comparing the experimental measurements of CO_2 solubility, CO_2 absorption rate, and viscosity in the lab, IBD-AMP-PZ, abbreviated as IAP herein, was selected as the best solvent candidate and tested in a PCCP that can handle 150 Nm^3/h of flue gas. To determine the performance and characteristics of the PCCP, a baseline evaluation was conducted with the MEA 30 mass% solvent. The proposed IAP solvent demonstrated highly stable operation and a reboiler duty that was approximately 0.7 GJ/t-CO_2 lower than that of MEA 30 mass% solvent. Excluding the heat loss, the reboiler heat duty of IAP was estimated to be approximately 2.9 GJ/t-CO_2. The absorber temperature profile under the optimal operating conditions indicated that the proposed solvent utilized the column more efficiently than MEA 30mass%.

Of course, the optimization ratio for IBD, AMP, and PZ must be determined through additional laboratory tests and PCCP operations. Stability analysis of IAP degradation by SO_2 should also be performed with pilot long-term demonstrations. In addition, for the commercialization of IAP, direct comparative evaluation with commercial solvents such as aMDEA® is required. These comparative assessments should be done in a larger scale process as well as the PCCP used in this study. Nevertheless, the IAP contained in the polyamine showed its potential as a direct CO_2 capture solvent through pilot demonstration.

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Conflicts of Interest: The authors declare no conflict of interests.

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