New insights in CO₂ post-combustion at Dürnrohr – An important step towards full-scale plants

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Kurzfassung

Neue Erkenntnisse aus der CO₂-Post-Combustion Versuchsanlage in Dürnrohr – Ein wichtiger Schritt hin zu Großanlagen

Ohne Subventionen ist der Betrieb von PCC-Anlagen (Post-Combustion Capture) und des gesamten CCS-Prozesses (Carbon Capture and Storage) unrentabel. Die Verwendung von Edele-
stählen verursacht hohe Investitionskosten. Der Lösungsmittelstrom bestimmt die Größe der Anlagenkomponenten sowie die notwendige Kühlleistung. Diese ist in der Auslegung von Kraftwerken of ein limitierender Faktor. Weite-
re Kosten entstehen durch die Entfernung von unerwünschten Komponenten aus dem CO₂- armen Rauchgas. Die hohen Betriebskosten werden vorwiegend durch den Energiebedarf zur Lösungsmittelregeneration verursacht. All die Punkte können wesentlich durch das CO₂-absorbierende Lösungsmittel beeinflusst werden. Aufgrund langjähriger Erfahrung in der Gasreinigung ist die CO₂-Abscheidung mittels Monoethanolamin (MEA) eine ausge-
reifte Technologie. Nun der Einsatz neuerartiger Lösungsmittel ermöglicht weitere Energieein-
sparungen.

Am Kraftwerk Dürnrohr (Österreich) wurde eine PCC-Versuchsanlage (CO₂SEPPL) in Betrieb genommen. Durch die industrielle Höhe der Absorberkolonne und der Verwendung von Kraftwerkstrauchgas können realitätsnahe Mes-
sungen durchgeführt werden. Die Amine Piper-
razin (P2), Ethylenlamin (EDA) und 2-Ami-
no-2-methyl-1-propanol (AMP) ermöglichen erhebliche Energieeinsparungen. Versuche mit Aminosäuren, deren Dampfdruck nahezu null ist, wurden durchgeführt, um schädliche Emissions zu reduzieren. Aufgrund ihrer schlech-
ten Regenerierbarkeit und langsamen Kinistik haben die getesteten Aminosäuren einen enorm hohen Energiebedarf.

Introduction

Carbon capture and storage (CCS) is one of the technologies to tackle climate change. While comprehensive renewable energy is the long-term objective, CCS can deliver an import contribution until then. How-
ever, CCS has a major influence on the con-
ventional energy industry. A considerable loss of efficiency must be accepted for CO₂ capture. The following work deals with the post-combustion capture (PCC) technol-
ogy which has the highest potential for retrofitting existing power and industrial plant. The investigated method is based on the already proved absorption/desorption process with chemical solvents.

Expansion of CCS will be a difficult jour-
ney. One must find a solution that will make CCS more competitive, and not place them in a more difficult situation. Selection, authorisation process and operation of CO₂ storages in the European Union are legally fixed by the directive 2009/31/EG [4]. Further sanctions such as the manda-
tory use of CCS in new and the retrofitting of existing power plants were discussed, but are not included in this directive [5]. The EU directive is not directly applicable to the member states. The member states have to convert the directive into national law. While geological CO₂ storage is pro-
hibited by law in Austria [1], a maximum storage amount of 4 million tonnes of carbon dioxide per year is legally fixed in Ger-
many [2]. Moreover, the low carbon tax complicates the expansion of CCS. Addi-
tional subsidies of renewable energies will create little incentive to expand CCS. The future of CO₂ storage primarily depends on long-term studies and political decisions. In contrast, the CO₂ capture process has intensive research potential with respect to efficiency enhancement.

The pilot plant studies in Dürnrohr (Aus-
tria) can make a valuable contribution to the scientific community in this area of research. Six years of intensive research were carried out together with EVN AG and ANDRITZ Energy & Environment GmbH. Separated CO₂ can be compressed and stored in cylinder bundles in order to demon-
strate the full CCS chain. Geological storage was not considered. A substantial utilisation of the separated CO₂ for the pro-
duction of poly-hydroxybutyric acid (bio-
plastics) is currently in operation. Essential factors that influence the energy demand are investigated by means of experimental parameter studies. Individual contribu-
tions of regeneration energy demand can be identified due to energy balance of the entire system. Approaches of system optimisation can be obtained as a consequence. These findings can be used for developing selection criteria for novel solvents.

The applicability of novel solvents should be examined by detailed parameter studies on the CO₂SEPPL pilot plant. The solvent stability is a decisive factor for solvent loss by means of degradation. The costs for supply of fresh solvent depend on it. Solvent loss can also be caused by volatility. Harm-
ful components of the solvent and certain degradation products which are emitted to the atmosphere should be monitored. Sol-
vents with low vapour pressure are consider-
ed in more detail to keep emissions of undesirable components low.

The use of amines and amino acids are considered separately in [18] and [19]. The present work should provide an overall view of all investigated solvents. More-
ever, new results of the recently investigated aqueous blend of 2-amino-2-methyl-1-pro-
panol and piperezine are presented.

Design of the CO₂SEPPL pilot plant

For more than 6 years, a research campaign to CO₂ capture has been run at Dürnrohr. First, a small pilot plant was put in opera-
tion. The understanding and balancing of the absorption/desorption process was the objective of the first plant. Only one year later, a major pilot plant designated CO₂SEPPL (CO₂ SEParation Plant) was put in operation, based on the findings from the first study. Main difference to the first pilot plant is an increase in absorber height, flue gas flow rate and measurement instru-
mentation. The CO₂SEPPL pilot plant has already been described in [18] and [19]. The plant will be presented briefly in the following and reference will be made to relevant literature.

A slippstream of the flue gas from the power plant process is branched off to the pilot plant (Figure 1). A downstream gas blower increases the pressure in or-
der to reduce degradation rates of chemi-
solvents, a pre-scrubber decreases the
enabled by means of structured packings. The capture of CO$_2$ between the gaseous and the liquid phase, the enriched solvent occurs. Heat must be transferred to the main heat exchanger, where a temperature increase of the enriched solvent passes the main heat exchanger. On the way to the desorption unit, the CO$_2$ enriched solvent is pumped from the absorber column to the desorption unit. There the CO$_2$ enriched solvent is heated to its distillation point by an almost industrial absorber column.

Distorted measurements can be avoided by an almost industrial absorber column height of 12 m. High absorber columns enable prolonged contact time of gaseous and liquid phase resulting in low influence of kinetics on measurements. The CO$_2$ absorption capacity and the regenerability in the desorber are decisive for the specific energy demand. The specific energy for solvent regeneration used in the present work, refers to the separated amount of carbon dioxide and regeneration energy. Heat losses must not be taken into account when calculating the energy demand.

In order to illustrate the realistic measurement conditions on the CO$_2$SEPPL pilot plant, studies with the benchmark solvent monoethanolamine (MEA) were performed. Measurement results of the pilot plants in Esbjerg (Denmark) and Niederaußem (Germany), two of the largest plants in the world, served as comparison. There were only slight different results determined on the CO$_2$SEPPL pilot plant, which are caused by different operating conditions and system configurations (e.g. absorber intercooler) [18].

Selection of solvents

First and foremost aqueous amine solutions are the most popular group of solvents for CO$_2$ separation. They have a long history in gas sweetening applications. Amines arederivates from ammonia (NH$_3$) and can be subdivided into primary, secondary and tertiary amines, depending on the number of hydrogen atoms of the former ammonia molecule, which are replaced by functional organic compounds.

When choosing an amine for CO$_2$ capture, a trade-off between reactivity and CO$_2$ loading capacity has to be taken into account. Primary amines are characterised by high reaction rates but a small CO$_2$ loading capacity. Tertiary amines have a high CO$_2$ loading capacity. However, their slow kinetics leads to higher absorber columns. Both primary amines as also secondary amines have a considerable heat of CO$_2$ absorption. The heat of absorption is part of the energy required for solvent regeneration. It seems that a high heat of
absorption is unfavourable with respect to the energy demand. However, the overall heat demand for solvent regeneration can be minimised when using solvents with high heat of absorption [12]. This is due to the fact that high heat of absorption solvents (such as MEA) profit from a temperature swing between absorber and desorber column. An increase in desorber pressure leads to less water vapour at the desorber column. An increase in desorber pressure leads to less solvent loss. Furthermore they have better resistance to oxygen-rich flue gas streams [7]. Many amino acids occur naturally and they also have favourable biodegradation properties, factors which make the disposal of these solvents easier and with lower environmental impacts [22]. An amino acid is only reactive toward CO₂ after addition of a strong acid such as sodium of potassium hydroxide (NaOH, KOH) [10].

The selection of solvents based on an intensive literature study and preliminary laboratory experiments. Solvents which were investigated in the CO₂-SEPP pilot plant are listed in Table 1.

Review of the process

The absorption/desorption process is most influenced by the solvent flow rate. In the present work, the solvent flow rate is indicated as the ratio of the solvent volume flow and the flue gas volume flow when entering the bottom of the absorber column (L/G-ratio). Thereby, operating modes with different flue gas flow rates can be compared easily. Operating parameters of the measurement series with varying solvent flow rate are listed in Table 2. The default values from literature were used. Only in the case of the flue gas flow rate, which entails a drastic reduction of power plant efficiency for full scale CCS implementation [13]. Within operation MEA shows significant tendencies to degradation and foaming. The degradation of MEA occurs through oxidative reactions, salt formations, carbamate polymerisation and thermal influences [3].

In recent times solvents based on secondary and tertiary amines, polyamines as also sterically hindered amines gained in importance. By the mixture of amines attractive solvents can be produced. Piperazine is often used as promoter in such blends because of their fast kinetics. For a few years there has been a growing interest in the usage of amino acids for CO₂ capture. Due to the strong structure of amino acids they are non-volatile which leads to less solvent loss. Furthermore they have better resistance to oxygen-rich flue gas streams [7]. Many amino acids occur naturally and they also have favourable biodegradation properties, factors which make the disposal of these solvents easier and with lower environmental impacts [22]. An amino acid is only reactive toward CO₂ after addition of a strong acid such as sodium of potassium hydroxide (NaOH, KOH) [10].

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Because of vast experience in gas sweetening processes coupled with good availability and low price, MEA serves as benchmark solvent for CO₂ separation. MEA is a primary amine. Therefore, MEA has a high reactivity with respect to CO₂. The CO₂ loading capacity is limited to 0.5 mol CO₂/mol MEA. Besides the limited loading capacity, MEA is characterised by some other disadvantages with respect to CO₂ separation. First and foremost the regeneration duty is in the range between 3.5 and 4.0 GJ/t CO₂,

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maximum achievable CO$_2$ partial pressure in the absorber column at low solvent flow rates. The region of the maximum CO$_2$ partial pressure is characterised by the lowest optimal solvent flow rate (2 l/m$^3$). The fast kinetics of piperazine does not result in an increased solvents CO$_2$ loading because of the high absorber column. The low energy demand of 28/17 wt% AMP/PZ is also associated with the high CO$_2$ partial pressure in the desorber, resulting in small amounts of generated stripping steam. Moreover, the process with 28/17 wt% AMP/PZ is characterised by the lowest optimal solvent flow rate (2 l/m$^3$). The dissipated heat from power plants is a crucial factor in the design. A reduction of the solvent flow rate leads to a lowering of the heat to be dissipated in the heat exchanger of the regenerator solvent. Smaller dimensions of the entire piping and aggregates leads to a further reduction in operating and capital costs. 32 wt% EDA allows an energy saving of 9 % (3.34 GJ/tCO$_2$) in comparison to the process with 30 wt% MEA.

When considering Figure 2b it can be already observed that the investigated aqueous solutions of amino acids have a very high energy demand (>5 GJ/tCO$_2$) in comparison to the amine based solvents. In addition, extremely high solvent flow rates are required for reaching the optimal operating point. The high solvent flow rate totally negates the low specific heat capacity of the amino acid solvents. Decreasing amino acid concentrations shifts the optimum operating point to even higher solvent flow rates [17]. This series of measurements reduces the energy demand of aqueous NaGly solvents, which is often numerical determined in literature [9, 21]. Although mainly faster kinetics was published and faster CO$_2$ absorption was proven in laboratory scale, reduction in energy demand with aqueous KGly in comparison to aqueous NaGly is impossible [20].

**Solvent CO$_2$ loading**

Due to the limited CO$_2$ loading capacity of each solvent, a high CO$_2$ loading difference is coupled with a low lean CO$_2$ loading [18]. Figure 3 shows the CO$_2$ loading of 28/17 wt% AMP/PZ across the absorber column by varying L/G-ratio. The enriched solvent (0 m absorber height) is almost equilibrium loaded. The number of separation plates in the absorber column is sufficient. The rich loading decreases slightly by increasing the L/G-ratio. This is due to the higher temperature in the absorber column (Figure 4). Moreover, a high temperature reduces the equilibrium CO$_2$ loading [18]. The majority of CO$_2$ is absorbed in the upper part of the absorber column at low solvent flow rates. The region of the maximum

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### Tab. 3: Reboiler duty and L/G-ratio in the optimum operating point for different solvents [14, 16, 17, 20].

<table>
<thead>
<tr>
<th>Concentration of active substance [wt%]</th>
<th>L/G-ratio [l/m$^3$]</th>
<th>Reboiler duty [GJ/t CO$_2$]</th>
</tr>
</thead>
<tbody>
<tr>
<td>MEA</td>
<td>3.67</td>
<td>3.67</td>
</tr>
<tr>
<td>EDA</td>
<td>3.34</td>
<td>3.49</td>
</tr>
<tr>
<td>PIP</td>
<td>3.17</td>
<td>5.73</td>
</tr>
<tr>
<td>AMP/PIP</td>
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<td>5.51</td>
</tr>
<tr>
<td>NaGly</td>
<td>8.00</td>
<td>7.00</td>
</tr>
<tr>
<td>KGly</td>
<td>9.00</td>
<td>5.16</td>
</tr>
</tbody>
</table>

a) The optimum operating point could not be achieved because of the high solvent flow rate.
b) A lower reboiler duty of 5.46 GJ/tCO$_2$ was measured at a L/G-ratio of 10.34 l/m$^3$.
c) A lower reboiler duty of 5.65 GJ/t CO$_2$ was measured at a L/G-ratio of 8.00 l/m$^3$.

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**Fig. 3. Solvent CO$_2$ loading across the absorber height when using 28/17 wt% AMP/PZ.**

**Fig. 4. Dependence of the absorber temperature profile on the L/G-ratio. Experimental data for 28/17 wt% AMP/PZ (dashed line) and 40 wt% KGly (solid line).**
CO₂ absorption moves downwards with increasing solvent flow rates. No CO₂ absorption occurs at the upper half of the absorber column starting from an L/G-ratio of 4 l/m³. In contrast, CO₂ is still absorbed over the entire absorber column at an L/G-ratio of 5 l/m³ when using 37.6 wt% PIP [14]. At high solvent flow rates only a part of the column height is effectively used for CO₂ absorption. CO₂ absorption in the lowest quarter of the column is very limited for all examined solvent flow rates.

Consideration of the measured CO₂ loadings of 37.6 wt% PZ shows that there is a risk of solidification over the entire studied range. The CO₂ loading of the regenerated solvent is at the edge of the solidification range at small solvent flow rates. In the case of a system failure, there is especially a risk of fast solidification in the line of the regenerated solvent between desorber and absorber. During a brief system failure, the entire line must be trace heated. A temperature above 35 to 40 °C (depending on the solvent flow rate) must be maintained at any point. In order to ensure a safe plant shutdown, the entire solvent inventory must circulate twice without regeneration to increase the CO₂ loading. In contrast, when using high concentrated amino acid solvents (>30 wt%) [20], there is a risk of precipitation of the CO₂ enriched solvent.

Absorption temperature

The temperature of the treated flue gas is measured at a height of 12 m. The lowest temperature sensor is located in the absorber sump and measures the temperature of the enriched solvent. The eleven intermediate temperature sensors measure a combination of the solvent and flue gas temperature, which is denoted as absorption temperature in the following.

Figure 4 shows the temperature profile of the absorber column for 28/17 wt% AMP/PZ and 40 wt% KGly. The highest absorption temperature is in the vicinity of the inlet of the regenerated solvent at low solvent flow rates. The large temperature change at the top of the column suggests an intense reaction of carbon dioxide with the solvent. The temperature decreases due to heat transfer from the liquid to the gaseous phase in the lower part of the absorber column. The higher heat capacity of the solvent relative to the flue gas tends to push the heat of reaction to the bottom of the column at higher L/G-ratios [8]. Consequently, the temperature across the lower absorber column increases. Thus, the CO₂ enriched solvent temperature rises, too. The temperature of the treated flue gas drops according to the total energy balance.

Furthermore, there is a balance in the enthalpy leaving with the liquid and the flue gas when the temperature bulge is located somewhere in the middle of the absorber column [8]. A further increase of the solvent flow rate when using 40 wt% KGly leads to reaching this critical value, which can be seen from a rapid change in the temperature of the liquid and flue gas leaving the absorber column. The rapid change in both the treated flue gas and CO₂ enriched solvent outlet temperature seems to appear where the maximal absorption temperature occurs at an absorber height of 10 m. This is consistent with the numerical simulation of [8] for 30 wt% MEA.

Energy saving by increased flue gas flow rates and desorber pressures

The optimal L/G-ratios (Table 3) were set in these studies. The remaining fixed parameters are listed in Table 2. The separation performance of an absorber column is dependent on the gas load of the column. The gas load is expressed by the F-factor (F = u · v_p). At low flue gas flow rates, gas and liquid flow run with almost no mutual influence. This results in a low separation performance of the column. At high flue gas flow rates, the turbulence in the absorber column is higher. As a result, the separation efficiency increases until the flooding point of the column. The gas stream begins to entrain liquid at the flooding point. Consequently, the separation efficiency drops and the energy demand escalates.

The specific energy for solvent regeneration drops for all investigated amine based solvents (Figure 5) with increasing flue gas flow rate. The specific energy for solvent regeneration decreases to 3.0 GJ/tCO₂ for 37.6 wt% PZ, when the highest possible F-factor of 2√Pa is reached. This behaviour indicates the more than two times faster reaction rate of piperazine in comparison to MEA [6].

Figure 6 shows the influence of the desorber pressure on the process. The pressure in the desorber unit varies between 1.6 and 2.6 bar_{des}. In general, it can be seen that the specific energy for solvent regenera-
Post-combustion of carbon dioxide at Dürnhörf plant can be easily adapted.

The near-industrial height of the absorber vents in a realistic PPC-pilot plant. Due to the solvents in terms of PCC dimensioning can be achieved by adding of AMP to the piperoxase solution. Furthermore, piperazine based solvents have a large resistance to thermal and oxidative degradation, demonstrated by low concentrations of volatile components in the purified flue gas and separated CO₂ stream. Certain points have to be particularly observed for safe operation with piperazine based solvents. In order to prevent solidification, crystallisation and foaming during operation, additives have to be added and certain concentration ranges and operating areas must be avoided.

All amine based solvents show a decrease in regeneration energy with increasing flue gas flow rate. Especially 37.6 wt% PZ indicates a continuous decrease in energy demand up to the maximal flue gas flow rate of 120 m³/h. For MEA, there is even a stagnation of energy for flue gas flow rates up to 100 m³/h in contrast. Other amine-based solvents show only a small dependence on the absorber pressure compared to the benchmark solvent 30 wt% MEA. Substantial energy savings for higher desorber pressures (> 2 barₜₐₜ) are only recognizable for 37.6 wt% PZ.

In addition to amine based solvents, amino acids were tested in the pilot plant. Amino acids have a huge energy demand (> 5 GJ/t CO₂) in comparison to the tested amine based solvents. The increase in energy demand is caused by the poor solvent regeneration and the related slow kinetics of amino acids.

Conclusion

This paper provides a scientific investigation of amine and amino acid based solvents in a realistic PPC-pilot plant. Due to the ability to close the energy and mass balances meaningful results can be achieved. The near-industrial height of the absorber and desorber column and the operation with real flue gas from a hard coal-fired power plant enables a reliable characterisation of different absorption solvents. Different operating conditions of the pilot plant can be easily adapted.

An intensive literature review was carried out in order to find new attractive solvents for PCC. First, extensive laboratory tests, for study of the CO₂ absorption capacity, kinetics and solvent regenerability were carried out, in order to determine in advance the solvent behaviour in pilot plant operation. The amines ethylenediamine (EDA), piperazine (PIP) and 2-amino-2-methyl-1-propanol (AMP) were tested on pilot plant scale for their suitability as CO₂ absorption solvents.

Piperazine-based solvents show large saving potentials in terms of investment and operating costs because of their fast kinetics. For example, the energy requirement for solvent regeneration can be reduced to 15 % compared to the operation with the benchmark solvent 30 wt% MEA. Small solvent flow rates and thus significant savings in terms of PCC dimensioning can be achieved by adding of AMP to the piperazine solution. Furthermore, piperazine based solvents have a large resistance to thermal and oxidative degradation, demonstrated by low concentrations of volatile components in the purified flue gas and separated CO₂ stream. Certain points have to be particularly observed for safe operation with piperazine based solvents. In order to prevent solidification, crystallisation and foaming during operation, additives have to be added and certain concentration ranges and operating areas must be avoided.

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References

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