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**DEVELOPMENT OF A MOBILE PILOT PLANT FOR THE
EVALUATION OF NOVEL SCRUBBING LIQUIDS FOR THE
ABSORPTION OF CO₂ FROM INDUSTRIAL GASES**

**MOBILNA INSTALACJA PILOTAŻOWA DO OCENY
NOWEGO PŁYNU DO ABSORPCJI CO₂ Z
PRZEMYSŁOWYCH GAZÓW ODLOTOWYCH**

Abstract

Most available scrubbing liquids suffer from either high heating duties for the regeneration or vulnerability towards gas components. In order to increase the efficiency of the absorption process a novel scrubbing liquid has been developed by thyssenkrupp Industrial Solutions AG. For verifying relevance and feasibility of long-term operation of the new fluid assumptions for installation were created – conceptual design and detailed simulation of the process without detailed thermodynamic information.

Keywords: CO₂ separation, process engineering, modular construction

Streszczenie

Na większość dostępnych cieczy absorpcyjnych oddziałują wysoka temperatura i są one wrażliwe na składniki gazów odlotowych. W celu zwiększenia wydajności procesu absorpcji firma thyssenkrupp Industrial Solutions AG przedstawiła nowy płyn do skrubierów. Dla weryfikacji przydatności i możliwości długoterminowej eksploatacji nowego płynu przedstawiono założenia dla instalacji – projekt koncepcyjny oraz szczegółową symulację procesu bez szczegółowych informacji termodynamicznych.

Słowa kluczowe: wydzielanie CO₂, projektowanie procesowe, konstrukcja modułowa

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1. Introduction

The importance of the removal of carbon dioxide (CO₂) from various industrial gases, such as coke oven gas, is of great scientific and industrial interest [1]. A common method for the removal of CO₂ is the application of reactive scrubbing fluids in an absorption process. Chemical absorption is advantageous for the application of gases with low partial pressures and a high required selectivity regarding the absorption of CO₂.

For this purpose, a novel scrubbing liquid has been developed by *thyssenkrupp Industrial solutions AG* in order to improve the process efficiency. Therefore, an application for the separation of CO₂ is discussed in this contribution to evaluate the applicability and the long-term robustness of the novel scrubbing liquid under industrial conditions.

The objective of the work is the operation of the pilot plant industrial conditions at a steel mill in Duisburg, Germany. In order to gain experience for the subsequent scale-up, a flexible, modular, and transportable pilot plant is designed and built at *Process Dynamics and Operations Group* at *Technische Universität Berlin* [4]. The pilot-plant itself mainly consists of the absorption section and desorption for the regeneration of the amine solution. Proceeding and following these, pre- and posterior treatment columns are installed to maintain sustainable operational conditions despite fluctuations in the gas feed.

The main goal of the operation of the plant and experiments is the evaluation of the novel scrubbing liquid under industrial conditions and the determination of viable operation conditions. Therefore, the minimal heat required for desorption is determined for each operating point and the scrubbing liquid is analysed in terms of selectivity, longevity, and applicability.

1.1. Proposed Workflow for the Plant Engineering

Towards the scaled up industrial application, experimental data is required to examine the removal of carbon dioxide from industrial gases and to investigate a conceptual design for a faster process development. For this purpose, the pilot plant is designed to gain information about the operability and the process itself to compensate for the lack of operational experience [2]. For this reason, workflow for the plant engineering of a pilot plant is given in this contribution.

2. Research and Process Concept

The objective of the plant is the evaluation of the scrubbing liquid under industrial conditions and the optimisation of the operation of the pilot plant to develop an efficient process concept, independent of the various industrial production sites. Due to the variety of possible applications, the plant needs to be designed with regard to mobility and consideration for a modular construction that offers increased flexibility.

The basic engineering of a pilot plant to examine the novel scrubbing liquids without operational experience or limited thermodynamic knowledge can be attempted by using a comparable medium. In this contribution, monoethanolamine (MEA) is used as a scrubbing liquid and considered as a typical representative for the separation of CO₂ [5]. Coke oven

gas from a steel mill is considered as the feed. In the following table, the possible compositions of the feed gas are shown.

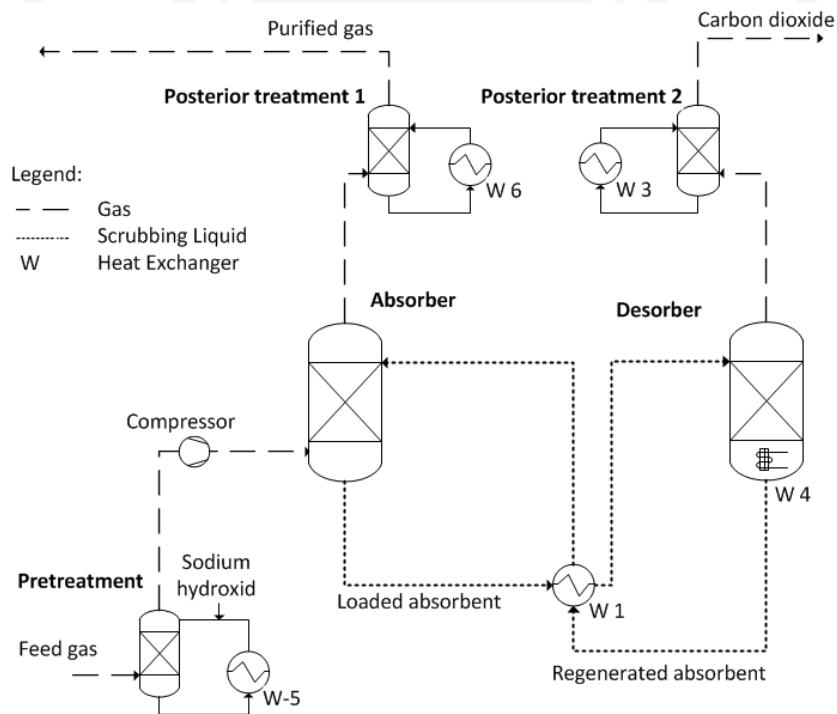
Table 1

Composition of the feed gas with pressure and temperature

	N ₂	H ₂	O ₂	CO	CH ₄	CO ₂	NH ₃	H ₂ S	p [bar]	T [°C]
Range [Vol.-%]	5-49	1-4	0-1	10-65	0-1	0-25	0-1	0-1	1- 1.4	30- 150

Regarding the compositions of the gases, a flexible operation of the process is required, such as a protection against corrosive or acidifying components. The operational pressure ranges from 1 to 1.4 bar in the absorption section, whereas in the desorption section, the pressure is increased to reduce the evaporation of water and amine at the required temperature for desorption (see Fig. 1). In the following section, the aforementioned plant design is detailed.

3. Plant Design

Fig. 1 Simplified flow diagram for the separation of CO₂

Based on the process concept a simplified flow diagram for the separation of CO₂ is shown in Fig. 1. The feed gas enters the plant in the pre-treatment column at almost ambient pressure, where cooling takes place and impurities or corrosive components, such as H₂S, are removed by using sodium hydroxide. The suction and transportation of the gases is realised by using a compressor. Upon entering the absorption section, carbon dioxide is separated from the gas and chemically dissolved in the scrubbing liquid as carbonate and carbamate ions. The gas leaving the absorber may contain smaller amounts of absorbent. Therefore, posterior treatment columns are installed in order to reduce the quantity of the absorbent and decrease the temperature of the purified gas. The loaded scrubbing liquid is pumped to the desorber, wherein, the regeneration of the scrubbing liquid takes place by electrical heating. The carbon dioxide is emitted to the gaseous phase and leaves the process through the second posterior treatment column. In turn, the regenerated liquid is reused and pumped to the absorber, thus closing the scrubbing liquid cycle.

3.1. Process Simulations and Assumptions

After the conceptual design of the process, a preliminary estimation of the operation conditions and design parameters with further investigations is needed. For this purpose, the process is modelled and simulated in Aspen Plus[®]. The rigorous simulation is required for the following sizing of the equipment and the estimation of the number of theoretical stages for both the absorption and desorption columns.

Fixed specifications for the estimation of the packing height is the separation of 90 Vol.-% of carbon dioxide. This is set by a *design spec* within the simulation. In order to keep the flexibility of the pilot plant, three different gas loads factors are considered, which are characterised by the F factor and three different gases with varying CO₂ concentrations. For the different values of the F factor, the operation points needed to be determined. Under the restriction of the separation of 90 n/n % of CO₂, the operation point is set by the minimal required electrical heating. The minimal required heating value is defined by the following equation:

$$q_{spez} = \frac{Q_{Desorber}}{m_{CO_2,seperated}} \quad (1)$$

where

- q_{spez} – specific amount of heating for the current operation conditions,
- $Q_{Desorber}$ – required amount of heating in the desorber,
- $m_{seperated}$ – mass flow of separated carbon dioxide in the scrubbing liquid.

The results for the operation points of the pilot plant are shown in the following table:

Table 2

**Operation conditions of the pilot plant for three gas load factors with following composition:
22.7 Vol.-% CO₂, 24.6 Vol.-% CO, 4 Vol.-% H₂, 48.6 Vol.-% N₂**

F factor [Pa ^{0.5}]	Q _{Desorber} [kW]	q _{spez} [MJ/kgCO ₂]
0.5	5.36	3.37
1.0	11.47	3.61
1.2	14.06	3.72

Changes of the gas load have an influence not only on the absorber, but also on the loading of the scrubbing liquid. An increasing gas load requires an elevated liquid flow of scrubbing fluid and additional electrical heating [5]. Fig. 2 represents an operation point with its conditions for an F factor of 1 Pa^{0.5}.

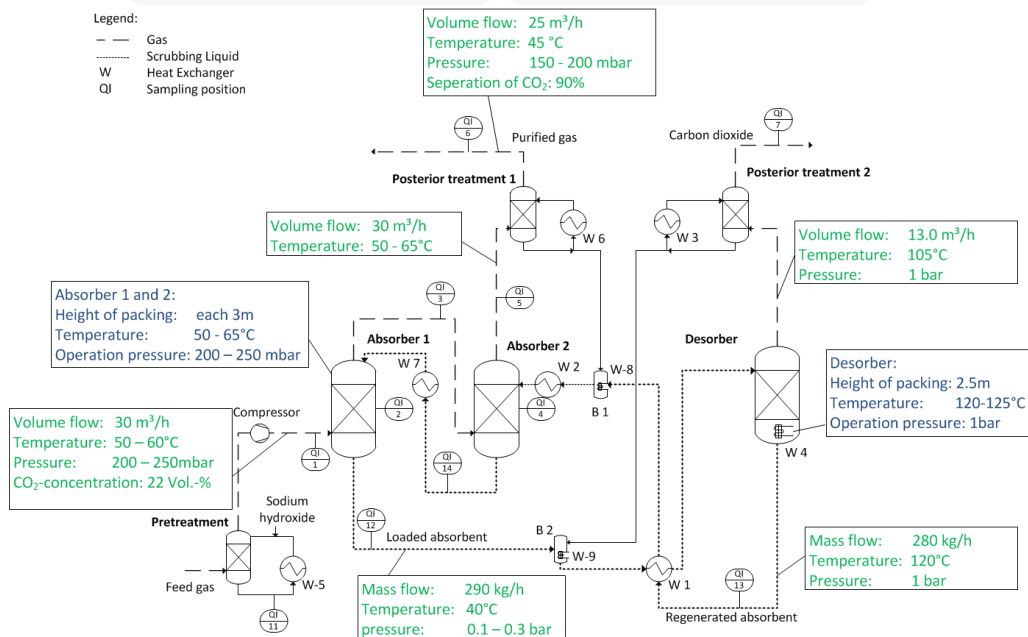


Fig. 2. Operation conditions for a gas load factor F of 1 Pa^{0.5}

3.2. Technical specifications and equipment planning

Based on the process simulation the equipment is sized and the technical specifications are determined [3].

Structured packings are employed for the columns with a total height of 6m packing for the absorption and 2.5m for the desorption. The additional columns for the pre- and posterior treatment contain 1m of structured packing each. With the height of the packing in the columns, the technical drawings of the apparatuses are drafted. For safe operation of the plant, strict safety regulations are required. Therefore, the whole pilot plant is planned,

constructed, and operated under European explosion protection ATEX/II2G. For safety reasons, a *HAZOP* analysis is conducted and the results are transferred to the process concept. The next step is the design of a three-dimensional (3D) model, which is necessary for the piping and the positioning of the apparatus in the condensed space of the mobile modular pilot plant.

Fig. 3 shows a 3D model of the whole pilot plant within the steel framework. This highlights the main challenge for the design of the plant, namely, the limited space. Each module has a height of 2.8 m, is 5.3 m wide and 2.3 m deep. The total height including the handrail on the upper level is 6.7m. Within the framework, apparatus need to be affixed. In addition to the pre-treatment columns, the two post-treatment columns, the two absorption columns, and the desorption column, the plant contains a compressor, ten pumps, and two liquid tanks. To handle this challenge, a comprehensive 3D model is developed in AVEVA PDMS[®] to ensure access for maintenance and operation. With the sole exception of piping for the pressurised air, the 3D model contains the piping for all utilities, gas, and liquid streams. The positioning of the gas pipes is of especially great importance due to their comparatively large diameter.

The pipes reaching out of the steel frame are the gas inlet and outlet. In addition, cable trays are positioned at the top of each module for supplying electricity and controlling all devices.

A basin below the lower module is installed to catch liquids in case of leaks or emergencies. Components, such as pumps and electrical heaters are placed at the bottom to facilitate maintenance work.

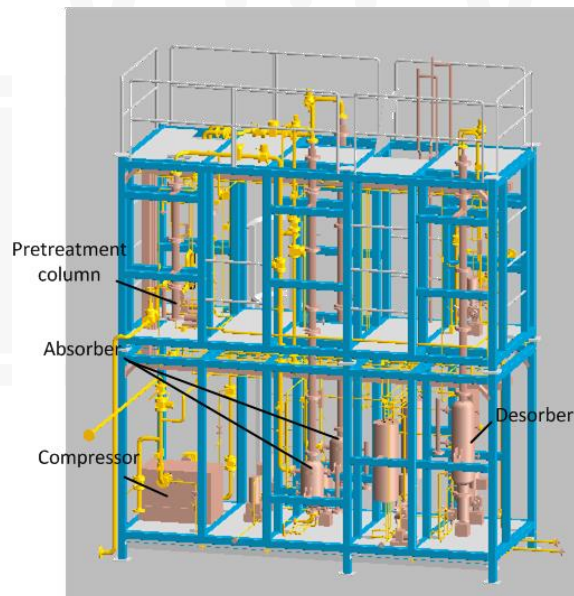


Fig. 3 3D model of the plant with apparatuses and the complete piping

Fig. 4 shows two photographs of the current state of construction work at TU Berlin. Due to the considerable height of the pilot plant, both the lower and the upper modules are separated and worked on side by side.



Fig. 4 Photos of the lower module (left) and of the upper module (right)

3.2 Process automation and analytics

The plant is automated using ABB's Freelance 700F process control systems. More than 30 temperature, 13 pressure, 8 level, and 13 flow indicators provide the required information for the process control. In addition, control sequences are implemented for the start-up, shut-down, emergency shut-down, and inertisation procedures.

As mentioned before, the main goal is the separation of CO₂ from the feed gas. Hence, samplings are intended to measure the CO₂ concentration within the gas stream by an infrared photometer of ABB (*Multiwave Model 3502*). This device enables online measurements at different positions. The positions are shown in Fig. 2. In total, seven positions are intended for accurate measuring. To keep the coefficient of absorption under surveillance, positions QI1, QI3, and QI5 are of great importance and are equipped with automated valves. In addition, manual sampling locations are positioned in the liquid pipes for the analysis of the scrubbing fluid in order to measure the load of carbon dioxide within the fluid.

4. Conclusion and Outlook

This contribution tackles the challenge of overcoming the step between lab experiments and the first industrial application. Therefore, a modular and mobile pilot plant is constructed to gain experience in the separation of CO₂ from industrial gases. Besides evaluating the novel scrubbing liquid without detailed thermodynamic knowledge or operation experience, the possibilities of a faster process development are investigated.

For basic engineering, it is necessary to clarify the operating conditions and to provide a conceptual design. Following the process simulations and the sizing of equipment, a detailed 3D model is required which puts a larger emphasis on safe operation and maintenance of the plant.

The goal of this work is to evaluate aspects such as the long-term robustness and the applicability, and to prove the industrial viability of the scrubbing liquid under real conditions. For this reason, the continuous operation of the process for more than 500 hours is planned. Furthermore, a more complex and detailed analysis of the liquid and gas phase is intended in order to increase the availability of the process.

Abbreviations

ATEX	– European directives on equipment and work in explosive atmospheres,
CO ₂	– carbon dioxide,
MEA	– monoethanolamine,
n/n	– mole per mole.

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