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Enhancement of CO_2 desorption using ultrasound and vacuum in water scrubbing biogas upgrading system

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Abstract−Ultrasound and vacuum were respectively employed to enhance CO_2 desorption in a water scrubbing biogas upgrading system. Results showed that incomplete CO_2 desorption could cause a high CO_2 content in the water and seriously affect the purity of the product gas. Vacuum had a strong enhancement effect on CO_2 desorption. When a vacuum of 0.04 MPa was used to enhance CO_2 desorption, the amount of the stripping air could be reduced to 1/16-th of that without enhancement, indicating that vacuum could greatly enhance CO_2 desorption and significantly decrease the amount of the stripping air, which was expected to reduce a large amount of energy consumption. In contrast, the enhancement effect of ultrasound was not so obvious for CO_2 desorption in the desorption column with air stripping, since the solution could be well desorbed by gas stripping, though ultrasound could strongly affect the static CO_2 desorption.

Keywords: Water Scrubbing, Biogas Upgrading, Vacuum, Ultrasound, Desorption

INTRODUCTION

Biogas upgrading is a vital step to producing a high quality fuel called biomethane with above 90% CH_4 by removal of CO_2. The upgraded biogas has similar property as natural gas and can be injected into the natural gas grid or used as vehicle fuel [1-3]. Several techniques, including high pressure water scrubbing (HPWS), pressure swing adsorption, organic physical scrubbing, chemical scrubbing, cryogenic and membrane technology, have been developed for the removal of CO_2 [4-6]. Among them, HPWS is the simplest, cost-effective and environment friendly method that has been developed to be the most extensively implemented technology with nearly 40% market share around the world [7-9].

HPWS works based on the solubility difference of gases in water. CO_2 is more soluble in water than CH_4. The solubility of CO_2 in water at 25 °C is approximately 26 times higher than that of CH_4 [9-11]. In the HPWS process, biogas is introduced to the bottom of the absorption column and flows up. Water enters the column at the top and flows downward, so that mass transfer occurs in a counter-flow way. Upgraded biogas leaves the column at the top, and water saturated with CO_2 is let out at the bottom. The scrubbed water can be used once in a single pass system or re-circulated and reused for the upgrading process after removal of dissolved gases. CO_2 stays in water in case of a single pass system or is released into atmosphere as an off-gas in case of water recirculation mode [12-15]. The concentration of CH_4 in the product gas can reach more than 96%. And after optimization of the essential parameters of the absorption process, 99% impurities can be removed from the raw biogas [16-18]. In our previous study [19], various factors such as water flow, gas inflow, absorption pressure and absorption temperature were studied with tap water (measured pH 7) as the inlet water of the absorption column. CO_2 concentration in the gas product (Y_{prod}) could be controlled below 3%, which could meet the technical standard of compressed natural gas for vehicle fuel.

It is a distinct advantage that chemicals are not required during the entire HPWS process. But the disadvantage of the system is that a large amount of water is required even with regeneration [18,20]. The HPWS system without recirculation can be suitable for the plants with low cost water (e.g. wastewater treatment facility) [12]. But freshwater use can be a major expenditure if potable water is the only source of water. For this reason water recycling is economically attractive. The most commonplace method for CO_2 desorption is air stripping in a desorption column by decompression at atmospheric pressure. With good desorption and cooling systems, almost completely closed water circulation can be achieved. However, the water recycling process is equipped with gas desorption and water cooling, which means greater investment and maintenance costs [7,13,21]. Generally, the larger the amount of the stripping air, the better the water regeneration effect, but more electric energy will be consumed. And the incoming air would transfer a large amount of heat to the water, which would need to be cooled before entering the absorption column. So the energy conservation

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of the regeneration step is crucial for this upgrading technology [22]. Several techniques such as ultrasound, microwave, high gravity and vacuum, have been proven to have an enhancing effect on gas desorption [22-25]. In this paper, ultrasound and vacuum were, respectively, investigated to enhance CO_2 desorption in order to reduce the amount of the stripping air.

MATERIALS AND METHODS

1. Materials

Considering the solubility similarity of CH_4 and N_2 and safety in operation, experiments were carried out with N_2 as the makeup gas instead of CH_4. The simulated biogas (contained 48 vol% CO_2 and 52 vol% N_2) was prepared by mixing a stream of CO_2 (>99.99%) and a stream of N_2 (>99.99%), which were provided by Jinan Deyang Special Gas Co., Ltd. Tap water was used as the inlet water of the HPWS system. Ethanol, NaOH, potassium hydrogen phthalate and phenolphthalein were analytical grade reagents and used as received.

2. Experimental Set-up of the HPWS System

The experimental setup of the HPWS system is shown in Fig. 1. It mainly includes an absorption column, a flash column, a desorption column, a plunger pump (TBP1002, Shanghai Tongtian Biology Co., Ltd), a thermostatic water bath (DC-1006, Shanghai Bilon Instruments Co., Ltd), two back pressure valves, two peristaltic pumps, three mass flowmeters (SY-9312, Beijing Shengye S&T Development Co., Ltd) and a biogas analyzer (Gasboard-3200, Wuhan Cubic Optoelectronics Co. Ltd). Both the absorption column and the desorption column were made of plexiglass, had a height of about 2.0 m and an inner diameter of 25 mm, filled with an 1.1 m-high φ5 mm×5 mm Dixon ring stainless steel packing, and had water bath mezzanines around them. In addition, a water circulating vacuum pump (SHB-IIIA, Zhengzhou Greatwall Scientific Industrial and Trade Co., Ltd) and an ultrasonic cleaner (KQ-250DE, Kunshan Ultrasonic Instruments Co., Ltd) were used this study.

3. Experimental Process of the HPWS System

First, the water was pressurized by a plunger pump and sent to the top of the absorption column after passing a thermostatic water bath. The simulated biogas entered the absorption column from the bottom after passing a mass flowmeter and the thermostatic water bath. Gas-liquid mass transfer was carried out in the absorption column. The product gas was led out from the top of the absorption column, passed through a back pressure valve. CO_2 concentration in the gas product (Y_{prod}) was analyzed by a Gasboard-3200 biogas analyzer. The water was discharged from the bottom of the absorption column and then entered into the flash column by control of the valve. The flash gas was discharged from the top of the flash column after passing through another back pressure valve which was set at 0.4 Mpa (absolute pressure, the same below). The water discharged from the bottom of the flash column was collected in an Erlenmeyer flask and sent to the top of the desorption column by a peristaltic pump. A stream of air entered from the bottom of the desorption column, contacted with the water and was discharged from the top as waste gas. The water was discharged from the bottom of the desorption column by another peristaltic pump and its pH value was measured. CO_2 concentration in the water was determined by titration method [26]. Conditions of absorption: water flow rate 100 mL/min, biogas inflow rate 800 mL/min, temperature 25 °C, pressure 0.9 MPa. Conditions of desorption: water flow rate 100 mL/min, temperature 25 °C.

4. Enhancement of CO_2 Desorption of a Static Aqueous Solution by Ultrasound

An amount of water was added into a high-pressure reactor, which was put in an ultrasonic cleaner at 25 °C. The reactor was sealed and filled with CO_2 three times to replace the air. CO_2 was introduced into the reactor to a pressure of 1.1 MPa for 6 hours and released slowly to or near atmospheric pressure. Then the ultra-

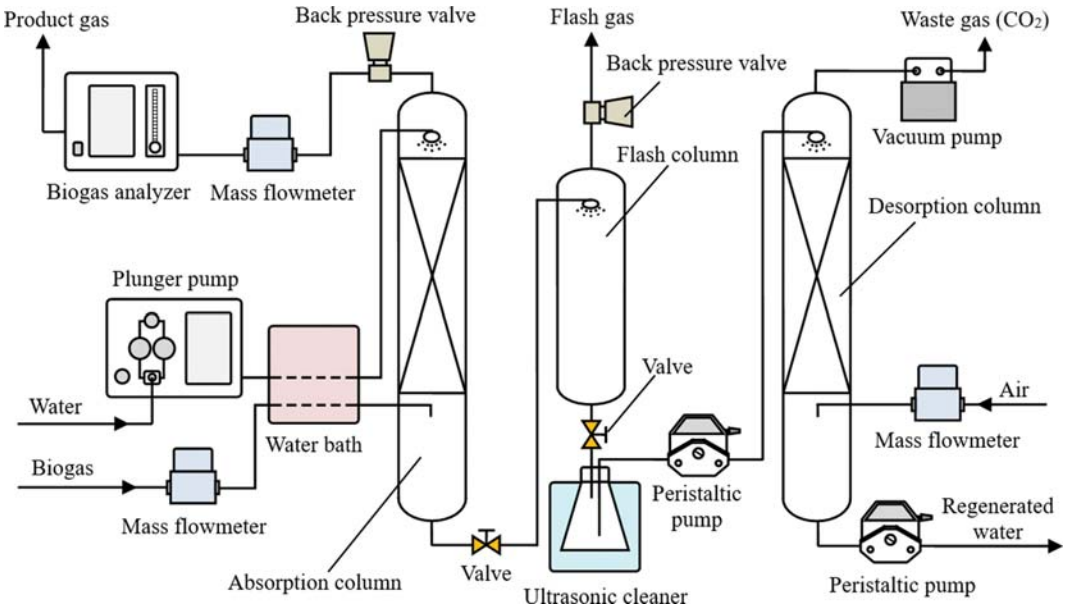

Fig. 1. The experimental setup of HPWS system.

sonic cleaner was opened. The speed and the volume of CO_2 desorbed from the solution were measured. And pH value of the solution was also measured.

5. Enhancement of CO_2 Desorption using Ultrasound in the HPWS System

On the basis of the experimental process of the HPWS system mentioned above, the water discharged from the bottom of the flash column was collected in an Erlenmeyer flask. After ultrasonification (250 W, 40 kHz) at 25 °C for 2 minutes, the water was sent to the top of the desorption column by a peristaltic pump.

6. Enhancement of CO_2 Desorption using Vacuum in the HPWS System

On the basis of the experimental process of the HPWS system, a water circulating vacuum pump and a vacuum controller were connected to the exhaust pipe at the top of the desorption column for controlling the vacuum degree.

RESULTS AND DISCUSSION

1. Effect of CO_2 Concentration of the Feed Water (C_{feed}) on CO_2 Removal

The HPWS process for biogas upgrading is based on the difference of solubility of CH_4 and CO_2 in water. When fresh water was used as the feed water of the absorption column, Y_{prod} could be very low with a high water flow rate [18,19]. When the regenerated water from the desorption column was used as the feed water, C_{feed} had an important influence on CO_2 removal, since it was related to the mass transfer driving force of CO_2 dissolution. Theoretically, the limit minimum Y_{prod} ($Y_{prod\text{-}min}$) should be the equilibrium Y_{prod} ($Y_{prod\text{-}eq}$) corresponding to C_{feed} and could be calculated according to Henry's law (Eq (1)) [27,28].

$$P_{CO2\text{-}eq}=X_{feed}\times H \quad (1)$$

where $P_{CO2\text{-}eq}$ is the equilibrium partial pressure of CO_2, X_{feed} is the mole fraction of CO_2 in the feed water and H is the Henry's constant. In fact, the actual partial pressure of CO_2 (P_{CO2}) must be greater than $P_{CO2\text{-}eq}$ in the absorption column.

$$Y_{prod\text{-}min}=Y_{prod\text{-}eq}=P_{CO2\text{-}eq}/P_{abs}=X_{feed}\times H/P_{abs} \quad (2)$$

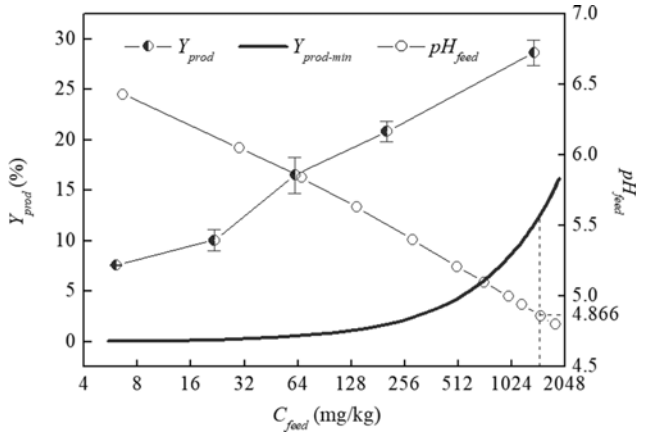

Fig. 2. The effect of C_{feed} on CO_2 removal in the absorption column.

where P_{abs} is the operating pressure of the absorption column.

Fig. 2 shows the effect of C_{feed} on $Y_{prod\text{-}min}$, Y_{prod} and pH value of the feed water (pH_{feed}), respectively. As can be seen, $Y_{prod\text{-}min}$ and Y_{prod} increased rapidly and the measured pH_{feed} correspondingly decreased with the increasing of C_{feed}.

It was found that pH value of the out water (pH_{out}) from the desorption column often stabilized between 5.8 and 6 after the regeneration with air stripping, $Y_{prod\text{-}min}$ would be about 0.50%. When the water was regenerated without air stripping, pH_{out} was 4.866 and CO_2 concentration of the out water (C_{out}) reached 147 mg/kg. If this water was used as the feed water of the absorption column, $Y_{prod\text{-}min}$ would reach 12.50% and Y_{prod} would be much higher than this concentration. It indicated that C_{feed} or pH_{feed} had an important effect on the HPWS system.

C_{out} was affected by the amount of the stripping air, and the latter was directly related to the maintenance costs of the HPWS system [13,21,22]. To reduce the stripping air, ultrasound and vacuum were, respectively, investigated to enhance CO_2 desorption.

2. Effect of Ultrasound on CO_2 Desorption of a Static Aqueous Solution

Sonochemical effects are due to the phenomenon of cavitation, which is the nucleation, and the behavior of bubbles in a liquid [29,30]. The microbubbles undergo oscillation, growth, contraction

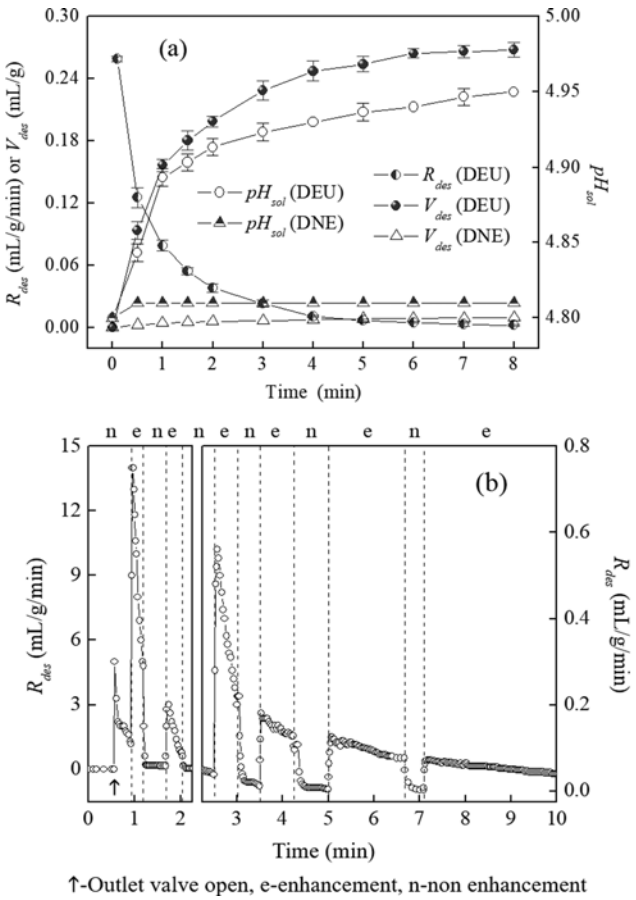

Fig. 3. The effect of ultrasound on CO_2 desorption of a static aqueous solution under continuous ultrasonification (a) and under intermittent ultrasonification (b).

and collapse. The generated cavitation bubbles grow continuously and gather to the gas-liquid interface under the action of directional diffusion to promote gas desorption.

The effect of ultrasound on CO_2 desorption of a static aqueous solution was studied, as shown in Fig. 3. As can be seen from Fig. 3(a), desorption without enhancement (DNE) could only obtain a very small desorption amount of CO_2 (V_{des}) and pH value of the aqueous solution (pH_{sol}) almost unchanged. For the desorption enhancement by ultrasound (DEU), the desorption rate of CO_2 (R_{des}) was large at the early stage of continuous ultrasonification and decreased with time and, correspondingly, V_{des} and pH_{sol} increased rapidly at first and then gradually trended gently. Fig. 3(b) shows the variation of R_{des} with time under the intermittent ultrasonification. As can be seen, every time the ultrasound was turned on, R_{des} increased abruptly and then decreased. Every time the ultrasound was turned off, R_{des} dropped precipitously. R_{des} measured under ultrasonification was about ten-times of that measured under no ultrasonification. The results indicated that the ultrasonification could obviously enhance R_{des} of a static aqueous solution.

As is known, ultrasound has been introduced to enhance the stripping of CO_2 and SO_2 by some other researchers [31-33]. It was thought that ultrasound could lead to cavitation and nucleation in the liquid and thus the formation of bubbles. Once formed, it would be relatively easy for bubbles to grow as more gas diffused to the bubbles and became part of the bubble. In this way ultrasound could make it easy for gas to escape in the form of bubbles.

3. Effect of Vacuum on CO_2 Desorption in the Desorption Column

Vacuum could change the system pressure and then affect the solubility of CO_2 according to Henry's law [27,28]. It could improve CO_2 desorption kinetics by increasing the driving force of the mass transfer [34,35]. Fig. 4 shows the influence of the desorption pressure on CO_2 desorption when the desorption is carried out under vacuum condition. As can be seen, C_{out} and the equilibrium C_{out} ($C_{out\text{-}eq}$, calculated according to Henry's law) decreased rapidly, and pH_{out} decreased from 6.17 to 4.98 with increasing the desorption pressure from 0.01 MPa to 0.10 MPa. It was obviously attributed to the principle of gas-liquid equilibrium. The vacuum caused the decreasing solubility of CO_2, and the latter increased

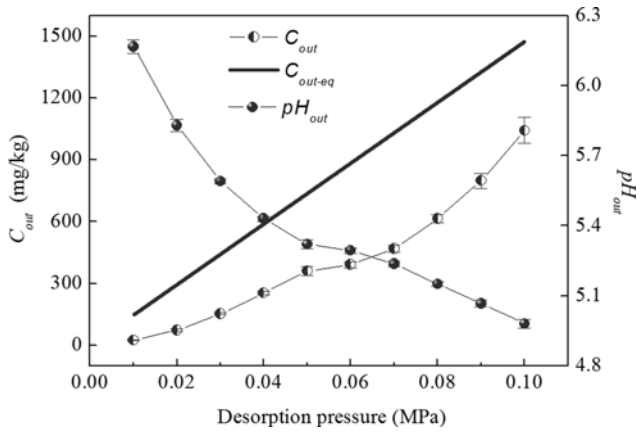

Fig. 4. The effect of the desorption pressure on CO_2 desorption in the desorption column without air stripping.

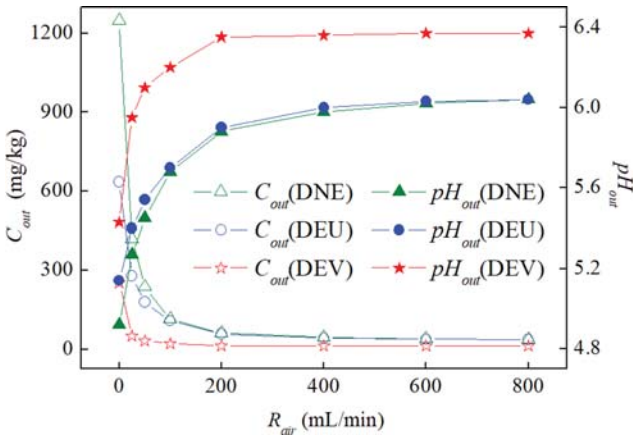

Fig. 5. The effect of R_{air} on CO_2 desorption under different desorption conditions.

the mass transfer driving force of CO_2 desorption. In addition, the experimental C_{out} was lower than $C_{out\text{-}eq}$ at under each pressure, which might have been caused by a small amount of other gases in the desorption column, including a small amount of water vapor, a small amount of N_2, and a small amount of air because the desorption system cannot achieve absolute sealing.

4. Effect of Air Stripping Rate (R_{air}) under Different Desorption Conditions

Fig. 5 shows the influence of R_{air} on CO_2 desorption under different desorption conditions including DNE, DEU and desorption enhancement by vacuum (DEV, at absolute pressure 0.04 MPa). As can be seen, under all the three desorption conditions, C_{out} decreased rapidly and pH_{out} increased rapidly at first, and then they trended gently with the increase of R_{air}. At each amount of R_{air}, C_{out} of DEV was much smaller than that of DNE and pH_{out} of DEV was significantly larger than that of DNE. The desorption efficiency of DEV with a R_{air} of 50 mL/min could reach that of DNE with a R_{air} of 800 mL/min, that is, R_{air} of DEV could be reduced to 1/16-th of the traditional R_{air}. The results showed that vacuum could obviously enhance CO_2 desorption and significantly reduce the amount of the stripping air, which was expected to reduce a large amount of energy consumption in the HPWS system.

To study the influence of ultrasound on CO_2 desorption in the HPWS system, the water discharged from the flash column was sent to the desorption column after ultrasound treatment. It was found that ultrasound could impact CO_2 desorption when R_{air} was small, and almost had no effect when R_{air} was large, as shown in Fig. 5. In addition, we added an ultrasonic vibrator (60 W, 40 kHz) on the middle part of the outer wall of the desorption column and did not find a distinct enhancement effect, although Fig. 4 had shown that ultrasound could enhance CO_2 desorption in a static aqueous solution.

The above results could be explained in terms of the driving force of the desorption process, which was related to the supersaturation degree of gas dissolved in liquid phase [36,37]. When the partial pressure of the gas in liquid phase was greater than that in gas phase (namely, supersaturation), desorption would occur. Depressurization, heating and gas stripping are three frequently used

strengthening ways to cause supersaturation. Only when the supersaturation exceeded a certain value were bubbles produced [36]. Obviously, ultrasound could greatly reduce the value, so it could greatly enhance CO_2 desorption in a static aqueous solution.

For the desorption in the column, when R_{air} was small the desorption was not sufficient, and ultrasound could impact the desorption; when R_{air} was large, bubble growth and gas-liquid mass transfer were sufficient and the water discharged from the desorption column was already unsaturated. On this basis, the enhancement effect of ultrasound was not so obvious, though ultrasound could strongly affect the static CO_2 desorption.

In addition, according to the thermodynamics of gas-liquid mass transfer enhanced by external physical fields, the external fields can change the gas-liquid equilibrium to a certain extent, and the degree of change is related to the change of the chemical potential caused by the external fields [38]. As a result, enhancement by ultrasound is not only for gas supersaturated liquid, but also for not saturated liquid [39,40]. Because the cavitation bubbles by ultrasound are vacuum bubbles at the beginning, this means the driving force of gas from liquid into the bubbles is high and the liquid could evaporate into the cavitation bubbles as well; then the bubble will expand rapidly and not be easy to collapse by the surface tension. Definitely, the intensity of the enhancement effect is related to the unsaturation degree of the solution.

Not only can a vacuum reduce the partial pressure of the gas in gas phase but also decrease that in liquid phase, thus decreasing the solubility of the gas. Accordingly, vacuum could greatly enhance the desorption when R_{air} changes from small to large. According to the experimental results, it could be derived that the combination of vacuum and gas stripping was more efficient and could achieve satisfactory desorption effect.

CONCLUSION

CO_2 desorption was directly related to the efficiency of the HPWS system for biogas upgrading. Experimental results showed that ultrasound could strongly affect the static CO_2 desorption and vacuum had a strong enhancement effect on CO_2 desorption in the desorption column. Vacuum could greatly enhance CO_2 desorption and significantly reduce the amount of the stripping air. Results indicated that the combination of vacuum and gas stripping was more efficient and could achieve satisfactory desorption effect, which was expected to reduce a considerable amount of energy consumption in the HPWS system. In addition, when the feed gas contained H_2S, inert gas stripping was needed in the regeneration step to avoid the formation of sulfur. It was more meaningful to reduce the gas volume of stripping. More studies are certainly needed to verify the energy-saving effect in industry.

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NOMENCLATURE

C_{feed} : CO_2 concentration of the feed water [mg/kg]
C_{out} : CO_2 concentration of the out water [mg/kg]
$C_{out\text{-}eq}$: equilibrium CO_2 concentration of the out water [mg/kg]
H : Henry's constant [kPa]
pH_{feed} : pH value of the feed water
pH_{out} : pH value of the out water
pH_{sol} : pH value of the aqueous solution
P_{abs} : operating pressure of the absorption column [kPa]
P_{CO2} : actual partial pressure of CO_2 [kPa]
$P_{CO2\text{-}eq}$: equilibrium partial pressure of CO_2 [kPa]
R_{air} : air stripping rate [mL/min]
R_{des} : desorption rate of CO_2 [mL/g/min]
V_{des} : desorption amount of CO_2 [mL/g]
Y_{prod} : CO_2 concentration in the gas product [vol%]
$Y_{prod\text{-}eq}$: equilibrium CO_2 concentration in the gas product [vol%]
$Y_{prod\text{-}min}$: limit minimum CO_2 concentration in the gas product [vol%]
X_{feed} : mole fraction of CO_2 in the feed water [mol%]

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