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Compact, low energy CO_2 management using amine solution in a packed bubble column

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Abstract

A novel method for removing CO_2 from gas streams is described. The carbon dioxide continuous scrubber, CDOCS, takes advantage of the intimate liquid–gas contact afforded in a packed bubble column to both absorb the CO_2 from a gas stream, and to regenerate the solution. The design relations and performance of a prototype CDOCS system using amine solution are presented. Over a 30-day trial, 20 m^3/h of air was continuously scrubbed to 60–80 ppm. The CDOCS footprint is less than 0.18 m^3 with power consumption around 300 W. Applications for air scrubbing include alkaline fuel cells, small scale processing, and industrial safety. A concept is proposed to sweeten biogas from dairy farm effluent for on-farm electricity generation. While industrial processes involving packed or plate trickle columns are well known for CO_2 production and sweetening, these conventional methods are complex and expensive, and do not scale down to air scrubbing or to small scale biogas production.

Keywords: CO_2 scrubbing; CO_2 management; Amine; Packed bubble column

1. Introduction

The objective of this project was to develop a feasible technology solution for the problem of scrubbing CO_2 from the air supply of alkaline fuel cells. Although AFC's have been successfully deployed in the space program for many years, terrestrial application has always been problematic because the acid gas, CO_2, in atmospheric air reacts with the base liquid electrolyte to form precipitates which can degrade the electrode performance and reduce the activity of the electrolyte [1]. At the present time, researchers and system developers employ a solid absorbent, soda lime, to remove the CO_2 from the oxidizer stream. However, soda lime cannot be regenerated, must be replaced frequently, and adds considerable operating cost. Soda lime has never been considered as an option for commercial AFC systems. AFC stack materials and architecture have continued to develop but a method to continuously scrub CO_2 from air that meets the system requirements for AFC plants has not previously been found [2].

Several applications currently exist for managing CO_2 levels in air including submarines [3,4], space craft [5], diving re-breathers [6], and the operating theatre [7]. We suggest that if a low-cost air scrubber were technically feasible, then new applications may include high CO_2 work environments like breweries and coal mines. CO_2 removal could also possibly reduce HVAC energy requirements in crowded environments like meeting rooms. A market may even arise in the future for home or work environments to be conditioned to pre-industrial CO_2 levels.

CO_2 is separated from a wide range of gas flows in industrial processes. Combustion gas is stripped with aqueous amine solution in order to produce bottled gas and dry ice using the common industrial method of wet scrubbing. Packed bed or plate columns are used to contact a liquid adsorbent with the gas stream [8]. Typically, industrial wet scrubbers utilize a temperature or pressure swing process to separate CO_2 from petrochemicals, methane (natural gas sweetening), and other gas streams [9]. Industrial separation technology is complex and expensive, and thus economy of scale means that most wet scrubbing equipment is large scale. Using the height equivalent of theoretic plate (HETP) design process, a packed column to scrub CO_2 from air to below 10 ppm would approach an infinite number of theoretical stages.

Monoethanolamine (MEA) is the most widely employed solvent for CO_2 absorption, used for over 70 years in the CO_2

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production and gas sweetening industries. Though new amines and amine mixes have been developed, MEA is still the preferred absorbent for low pressure and low concentration CO_2 absorption. The properties and behavior of amine solutions, while not entirely understood, have been empirically documented over time to provide engineers with a useful database for design [10]. Absorption occurs at temperatures up to approximately 60 °C. The MEA–CO_2 reaction is exothermic and reversible by supplying heat to the system. The temperature swing absorption/evolution process reverses at approximately 70 °C [11]. The basic MEA temperature swing chemistry is given in Eq. (1):

$$CO_2 + 2C_2H_4OH\text{–}NH_2 + H_2O \leftrightarrow C_2H_4OH\text{–}NHCO_2{}^{-} + C_2H_4OH\text{–}NH_3{}^{+} \quad (1)$$

The packed bubble column has not been reported in literature or industry as an apparatus for CO_2 absorption from air or other gas streams. It is well known that a bubble column provides excellent liquid/gas contact. A high surface area, low volume packing in the bubble column increases the gas hold-up, reduces geysering, and maintains a small bubble size [12].

The carbon dioxide continuous scrubber (CDOCS) system is a rather simple concept utilizing an MEA solution in a packed bubble column (PBC) as the CO_2 absorber, and a smaller PBC containing a heating element as the regenerator [13]. Air is scrubbed during bubbling through the absorber, and a flush gas is bubbled through the regenerator to carry away the regenerated CO_2 from the heated MEA solution. A low head flow, induced by the liquid lift from the air hold-up in the absorber PBC, is used to move solution from the top of the absorber, to the top of the regenerator, then return it to the bottom of the absorber.

Section 2 gives brief descriptions of the CDOCS system concept employed as an AFC air scrubber and for biogas sweetening. Section 3 outlines the operating theory and design relations for the CDOCS absorber and regenerator and the system integration dynamics. Section 4 describes the prototype system and the experiments performed. Section 5 gives the experimental results, with the conclusions following in Section 6.

2. CO_2 management applications

Current applications for CO_2 management by scrubbing with MEA exist in manned space flight, submarines and methane gas sweetening. CO_2 production also uses MEA solution in packed or plate columns to separate CO_2 from a combustion gas stream. The two future applications featured in this paper require small-scale, low-cost scrubbing. The relative simplicity of the dual packed bubble column DCOCS system is well suited to these emerging energy applications.

2.1. Alkaline fuel cells

The CDOCS was developed to specifically meet the requirements for a 5 kW AFC, but can be scaled for either smaller or larger systems. The air scrubber is positioned between the air blower and the humidification/pre-heating system and can be integrated with the air supply conditioning system to optimize space and power utilization [14]. A system schematic showing the operation of the CDOCS scrubber in the context of a typical AFC system is shown in Fig. 1. In this configuration, the previously scrubbed AFC exhaust gas is used as a source for the regenerator flush gas. Thermal integration includes use of AFC exhaust heat to pre-condition the incoming air. The incoming and outgoing regenerator flows pass through a counter flow heat exchanger to reduce heat energy input into the regenerator.

The CDOCS system prototype described in this paper was developed specifically to meet the system size, maintenance, and parasitic power requirements for a demonstration project. The demonstration system coupled a wind turbine, electrolyzer and AFC system to provide continuous power for a remote telecommunications facility. The system specifications limited parasitic power to 300 W and set a maintenance target of 30 days. The materials required for the CDOCS are also required to be compatible with the AFC.

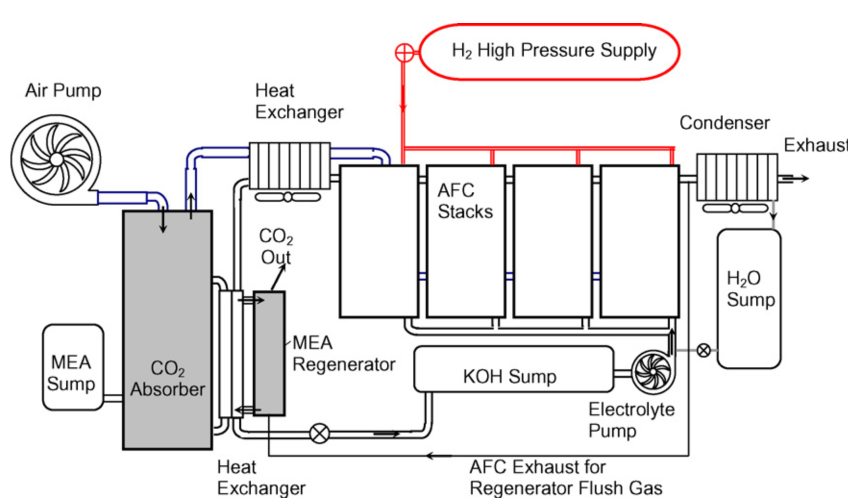

Fig. 1. Schematic diagram of an alkaline fuel cell power generation system with the CDOCS integrated into the air supply pre-conditioning, and utilizing AFC exhaust air as the regenerator flushing gas.

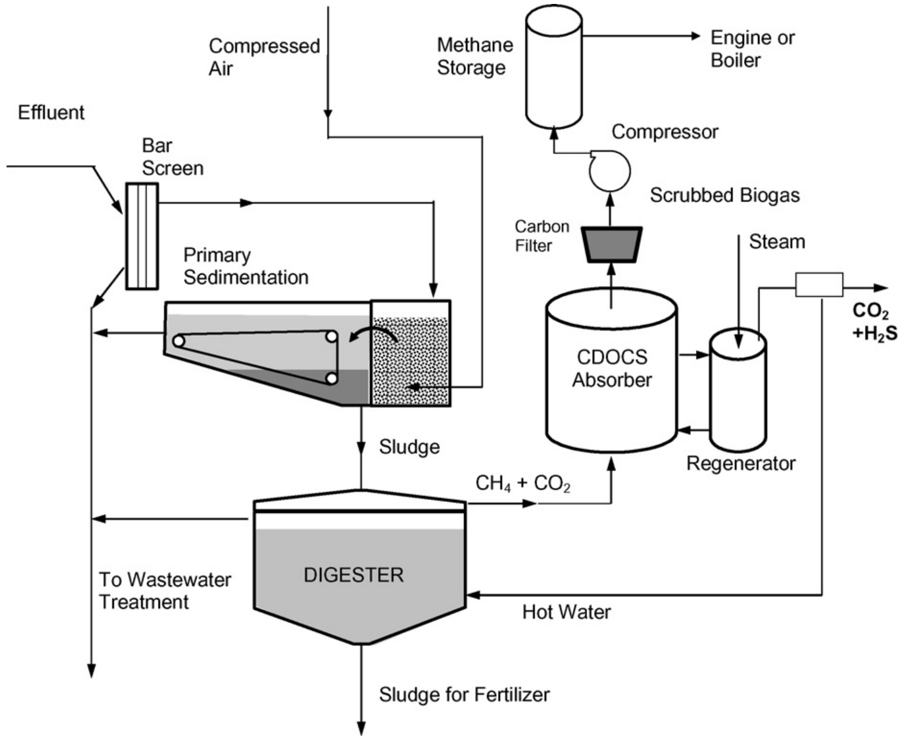

Fig. 2. Conceptual schematic diagram of an on-farm biogas processing system with the CDOCS system providing fuel gas sweetening and de-watering prior to storage or injection into an engine.

2.2. Small scale biogas sweetening

Intensive animal husbandry and industrial agriculture improve economic performance in the agricultural industry, but generate large animal and plant processing waste streams. In particular, swine and dairy farming have high localized effluent accumulation near housing sheds and milking barns. This kind of waste poses issues of odors, water pollution, and greenhouse gas emissions, particularly methane, CH_4, during decomposition. These agricultural waste streams must be treated to mitigate environmental impact, and the biogas energy potential has been investigated [15]. One of the negative energy aspects is transporting the waste to a large processing plant. On-farm biogas generation has been developing in recent years [16], but a major issue with biogas is the variable and possibly large portions of CO_2 and moisture, as well as the presence of H_2S. Biogas can be used directly from the digester for heating. However, for storage or use in an internal combustion engine, corrosion and variability of the gas heating value and combustion characteristics due to CO_2 are costly issues. A low-cost method for separating the acid gases and moisture from the biogas, termed “sweetening”, could facilitate higher value uses and storage.

The schematic diagram in Fig. 2 is a concept of an on-farm biogas system with integrated sweetening. An MEA and glycol solution readily absorbs water vapor from the gas stream when the water in the solution is less than 30 wt%. In an experiment with air and a solution with 10 wt% water, the solution absorbed 3 l of water from 1000 m^3 of atmospheric air. The absorbed water can be removed through the regenerator and either condensed and collected or exhausted as vapor. The absorption of CO_2 on a per mass basis increases with MEA concentration, but water is required during regeneration or degradation of the MEA can occur. The concept biogas CDOCS system is integrated with the biogas energy system by using waste heat from boiler combustion or an engine to supply heat for the regenerator.

An important property of amine solutions is the absorption of acid gases, including the H_2S which will form SO_2 upon combustion. The H_2S is regenerable from the solution with heat in the same process as the CO_2 regeneration [17]. The anaerobic digester can be operated at higher pressure (2.5–3 bar) which suppresses ammonia production, but also increases the CO_2 fraction [18]. This pressure is used in the concept system to drive the gas flow through the CDOCS system. Once the biogas is scrubbed, a compressor can be used to raise the pressure to a level suitable for storage and injection into an internal combustion engine or gas turbine generator.

3. Continuous scrubber design

This paper presents the results of research to determine design relationships and develop the system architecture for this novel application of a packed bubble column. The CDOCS system requires an injection gas pressure equal to the solution depth (in the range of 3 kPa_g). The gas stream is injected into the base of the packed bubble column with a depth of absorbent solution set by the level of CO_2 removal required. The packing breaks up the air stream into small bubbles and creates a well-mixed frothy liquid. Liquid lift caused by the gas hold-up is used to drive flow of the solution through a heated regenerator. The regenerator is also a packed bubble column with a small stream of flushing gas provided by a diaphragm pump. Corrosion and oxidation were found to be manageable due to the low temperature regeneration and stainless steel construction. The following sections describe the two key system components, the absorber and regenerator, and the system integration with an overview of the governing design and operating relations.

3.1. Absorber

The CDOCS system makes novel use of a packed bubble column (PBC) for both CO_2 absorption and regeneration. While the PBC has been well known in Chemical and Process Engineering, it is not as widely used in industry as packed trickle columns. A PBC is a liquid reservoir filled to depth, h, with the absorbing liquid with chemical solubility for CO_2 component, ε_0. Air with CO_2 concentration, C_{amb}, is injected at the bottom of the PBC, broken up into small bubbles by the packing material, and rises due to buoyancy. The residence time of the air flow, Q_a, in the liquid absorbent in a PBC of cross-section area, A, is given as $t_r = hA/Q_a$. The PBC has proven an effective means to remove CO_2 from air because of the high gas–liquid contact area and vigorous mixing [19].

The PBC absorber for a CDOCS system can be designed using the following relation for scrubbed CO_2 concentration:

$$C(t_r) = BC_{amb}\exp(-h_m A_p t_r) + \varepsilon_0 C_{amb} \tag{2}$$

where B is an entrance condition constant for a given solution which is determined experimentally for whetted packing but no liquid depth, h_m is the bubble flow mass transfer coefficient, and A_p is a packing factor expressing a representative bubble cross-section area. In previously reported investigations, the liquid depth for high efficiency scrubbing (98+% CO_2 removal) was determined to be 250–300 mm for 50 wt% MEA solution [19].

3.2. Regenerator

In our previous research we have shown that a high concentration aqueous solution (50 wt% MEA) buffered with glycol (35 wt%) can be regenerated at atmospheric pressure and temperatures as low as 70 °C without boiling when a packed bubble column is used. The PBC regenerator performance was investigated with the major finding being that regeneration at 100 °C in a PBC is faster than in a stirred beaker at 140 °C or higher [20]. Nitrogen was used as the flush gas in the previous regeneration studies of fully CO_2 saturated solution. CO_2 was measured in the exhaust gas stream, and the regeneration rate was shown to increase with flush gas flow and with temperature. The solution was re-saturated with CO_2 by dropping in dry ice, and regenerated as many as 10 times without loss of regeneration capacity or change in regeneration rate. Experimentally determined optimal regeneration conditions were used for the prototype system design; a regenerator heater temperature of 120 °C, residence time of at least 10 min, and flush gas flow capable of producing a fully frothy condition.

3.3. System dynamics

The operating dynamics of the CDOCS system used to model the system are shown in Fig. 3. For continuous scrubbing, the rate of CO_2 removed from the regenerator must equal the rate of CO_2 scrubbed from the air stream. The rate of solution flow through the regenerator, Q_s, is the primary system control parameter for achieving continuous scrubbing performance. Performing a mole balance on CO_2, the necessary flow can be related to the absorber efficiency, η_A, the regenerator efficiency, η_R, the mole rate of CO_2 influx to the scrubber with the air, $\dot{c}$, the mole fraction of MEA in the solution, f_{MEA}, and the CO_2 loading of

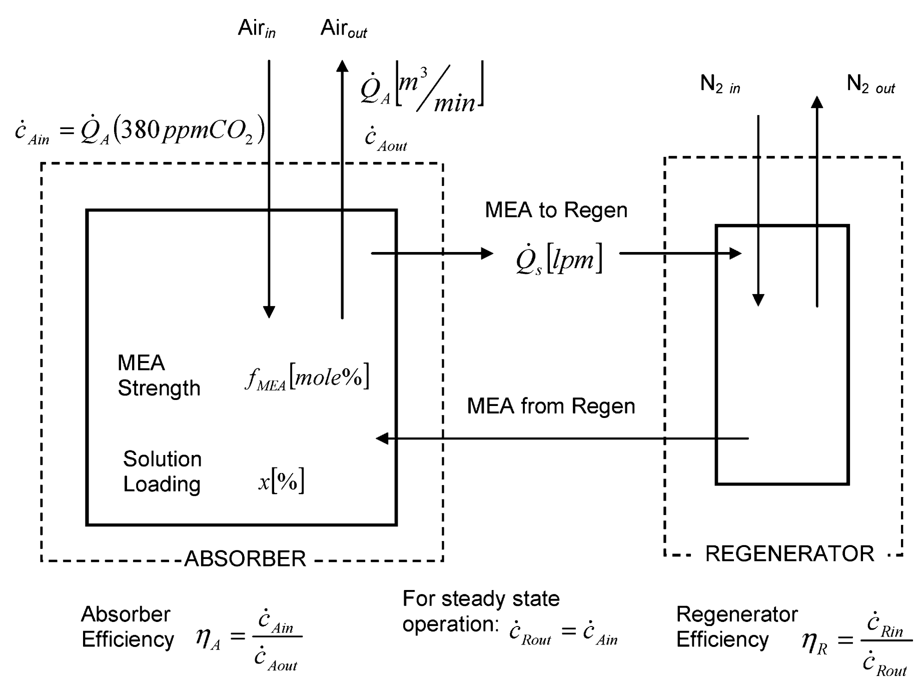

Fig. 3. Flow balance on the absorber and regenerator showing the definitions of the efficiencies for CO_2 stripping and for regeneration, respectively.

the absorber solution, α:

$$Q_s = 2\frac{\eta_A}{\eta_B}\frac{\dot{c}}{\alpha f_{MEA}}\frac{M_s}{\rho_s} \tag{3}$$

where M_s and ρ_s are the solution molecular weight and density, respectively.

4. Experimental set-up

A prototype CDOCS system was constructed to evaluate long-term performance [21]. Tests have not yet been conducted on methane or biogas, but the chemistry is well known for this process, and the design relations and system performance for air should be applicable to other small-scale scrubbing applications. The regeneration temperature and the regenerator flow rate are the primary variables which affect the steady state performance of the system, as shown in Eq. (3). The regenerator liquid flow was controlled with a manual valve and the regenerator element temperature was thermostatically controlled.

The experimental set-up is shown schematically in Fig. 4. The overall dimensions of the prototype system is 0.49 m × 0.35 m × 0.6 m. The fill level of MEA solution in the absorber was 300 mm and determined via a sight glass. Seventeen liters of fresh solution (50 wt% MEA, 35 wt% glycol, 15 wt% water) were placed in the absorber at the beginning of each experiment in the long-term testing. The dynamic liquid level of approximately 500 mm is caused by the gas hold-up in the PBC during operation. This dynamic head is used to collect fluid at the top of the PBC and generate the gravitationally fed flow through the regenerator. The air-induced solution flow from the absorber through the regenerator was determined to be in the range of 4 l/h for steady operation.

During operation, some solution is absorbed into the coalescer material and escapes in the exhaust as vapor. The liquid level was maintained over the course of the investigation via a peristaltic pump from a solution sump. While the testing program was carried out using 20 m^3/h air flow, the prototype absorber, with dimensions 500 mm height and 260 mm diameter was capable of scrubbing well over 40 m^3/h air flow [21]. The long-term tests used a lower flow rate because the low-cost blower would overheat if run at high power for long continuous periods.

The regenerator was constructed from stainless steel tube and fittings with dimensions 300 mm height and 22.5 mm diameter. The flush gas was atmospheric air supplied by a diaphragm pump at a rate of 1–4 l/h and pre-heated by passing through a coil in an oil bath. The flush gas was cooled in a stainless steel coil condenser before passing through the sampling chamber of a Drager infrared CO_2 detector. The continuous flow through the regenerator was cooled by a fan and stainless steel radiator prior to being returned to the absorber. In this experiment, the regenerator was not insulated and 310 W was continuously supplied to the heating element.

Atmospheric air was supplied to the absorber by a blower at a rate determined from a float flow meter. The air injection at the base of the rolled stainless steel absorber was accomplished by a simple 8-port sparger from a swimming pool filter. The absorber and the regenerator were packed with commercially available 15 mm diameter stainless Pall rings. Wire mesh stainless mist eliminators were layered on top of the packing to

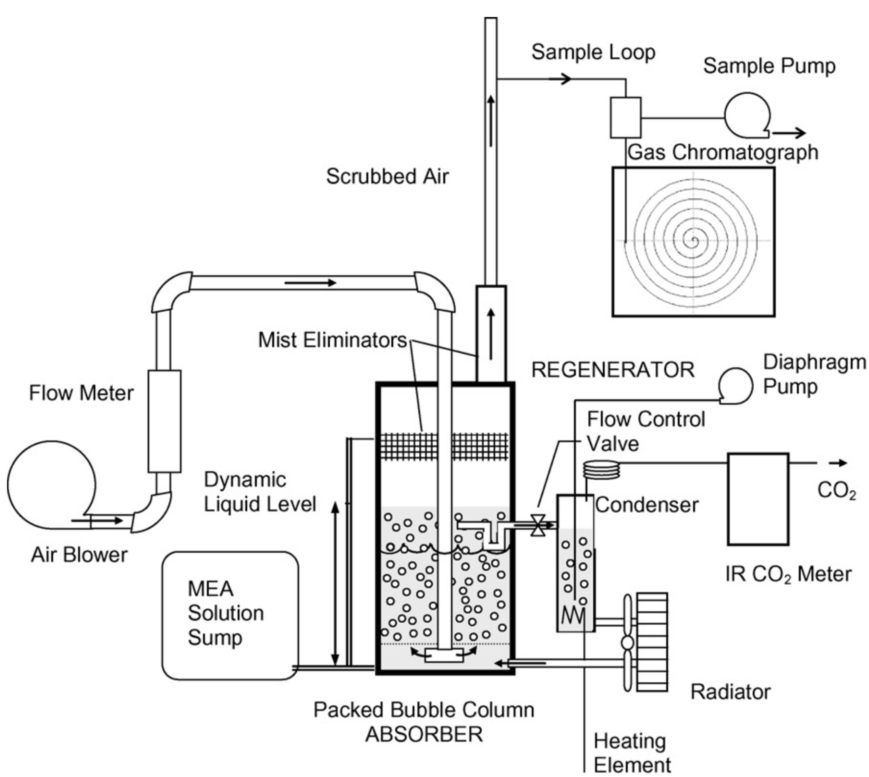

Fig. 4. Schematic diagram of the CDOCS prototype system used in the experimental investigations.

collect droplets. A commercial Pall coalescer containing pleated paper-like filter eliminated mist from the scrubbed air outlet, and collected fluid drained back into the absorber.

A Shimadzu GC-9A Gas Chromatograph with Thermal Conductivity Detector was used to measure the CO_2 concentration in the processed air stream. The inlet air CO_2 concentration was measured with a Draeger Polytron Infra Red CO_2 detector, and atmospheric levels in our laboratory were found to be 380 ± 5 ppm. The MEA concentration in the solution was monitored at regular intervals by titration of the solution with acid (0.1 M HCl) and methyl red indicator. The acid reacts only with free MEA in the solution, not with MEA which is bound to CO_2 but still regenerable (carbamate). The solution pH was also monitored using an Orion Research 701A digital Ionalyzer.

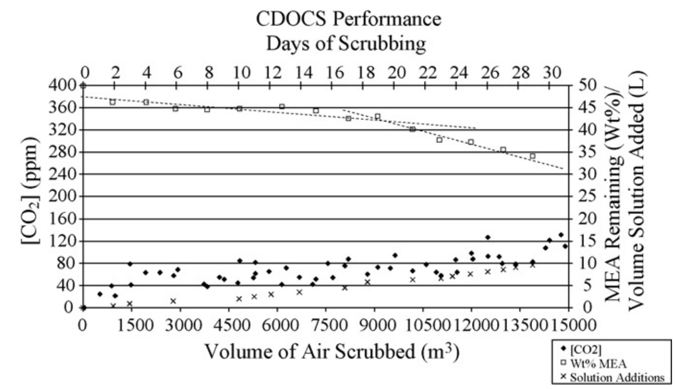

Fig. 5. Long-term scrubbing performance of DCOCS system with measurements of the MEA solution strength and tracking of consumption of solution from the scrubber sump.

5. Results and discussion

The experimental prototype system was used to investigate the scrubbing performance relationships with several operational variations, including solution strength, solution depth, scrubbing solution addition and regenerator temperature over several extended periods of 20–30 days. Table 1 gives a summary of the results of the experiments with the CDOCS system. As a comparison, air was scrubbed with the regenerator heater switched off, and a linear increase in CO_2 in the scrubbed air was measured from 0 to 380 ppm over 12 days.

The regeneration temperature of 120 °C provided good long-term performance without risking solution degradation. Higher regenerator flush gas flow enhanced performance, but with the current prototype, 4 l/min is the upper limit without causing operating issues with the fluid level at the top of the regenerator.

Using water to maintain the operating liquid level resulted in diluted solution over time and poor performance. Several MEA solutions were investigated for long-term behavior. A slight improvement in performance may be gained by using the 60/25/15 solution. The hygroscopic nature of the 80/20/0 solution became apparent when 3 l of water were absorbed from the first 1000 m^3 of scrubbed air. The MEG-free solution had markedly reduced performance and a much higher rate of solution loss due to the higher solution vapor pressure.

Using the optimal operating conditions determined by the experimental test program, the CDOCS system was operated over a period of 32 days. The test results for CO_2 concentration and solution additions over the month-long test are shown in Fig. 5. The CO_2 concentration was reduced to less than 60 ppm for the first 11,000 m^3 of scrubbed air, and to around 80 ppm for a further 3000 m^3 scrubbed air. A sudden darkening of the solution at 11,000 m^3 coincided with an increase in the rate of solution addition and a change in the rate of MEA wt% remaining in the solution as determined by the titration analysis. Over the whole test, the system required 10 l of solution over the 1-month period, or 0.7 l/1000 m^3 scrubbed air.

The CDOCS concept appears to be well suited to several CO_2 management applications, including air scrubbing for alkaline fuel cells, and biogas sweetening. The experimental results presented in this paper provide the basis for a scrubber system design for integration into a small AFC power plant. This was the original motivation for the work and further design for manufacturing will be done as required by AFC system developers. Further experimental development of system designs for biogas production on farms or at waste treatment facilities may continue in the future. The basic principle of utilizing a PBC rather than a standard packed column means that the CDOCS device can handle much higher gas flow rates within a small device footprint. The innovative use of the dynamic head to drive a regeneration cycle means lower system complexity, low parasitic power, and longer maintenance schedule than a solid absorbent scrubber.

Table 1
Results of long-term air scrubbing investigations, time to reach steady state operation, and the steady scrubbing performance, with effects of processing variables; regeneration temperature, flush gas flow, water and MEA solution additions

Regeneration temperature (°C)	MEA/MEG/H_2O (wt%)	Flush gas (l/min)	Water add rate[a]	Solution add rate[a]	Time to reach SS	Steady State CO_2 (ppm)
100 ± 10	50/35/15	1.8	2	0	9 days, 4500 m^3	160 ± 20
120	50/35/15	4	2.2	0	6 days, 3000 m^3	120 ± 20
120	50/35/15	4	0	0.38	4 days, 2000 m^3	80 ± 20
120	60/25/15	4	0	0.6	4 days, 2000 m^3	60 ± 20
120	80/20/0	4	0	0	12 days, 5000 m^3	120 ± 20
120	50/0/50	4	0	1.5	4 days, 2000 m^3	80 ± 20
120[b]	50/35/15	4	0	0.7	4 days, 2000 m^3	60 ± 20 80 ± 20[b]

[a] Liquid additions measured in l/1000^3 scrubbed air.
[b] Optimal system 30-day test: 60 ppm scrubbing for 23 days, 80 ppm thereafter.

This combination of features is critical for AFC application and for remote locations. Further research in CDOCS development will focus on product development for specific applications.

6. Conclusion

A prototype carbon dioxide continuous scrubber (CDOCS) system has been developed which utilizes the packed bubble column (PBC) gas–liquid contactor for absorbing CO_2 from a high volume flow of air in a compact space, and for regenerating the absorbent solution at low temperature. The novel concept involves wet scrubbing with an aqueous solution of monoethanolamine (MEA) and glycol. The gas hold-up in the absorber is used to drive the flow through the regenerator, a smaller, heated PBC which is flushed with air. The PBC allows for a small footprint (0.1 m^3) for scrubbing capability of at least 40 m^3/h.

The performance of the overall system is determined by the balance between absorption and regeneration. The prototype system was tested over 1 month of continuous operation for scrubbing 20 m^3/h air. The air was scrubbed to less than 40 ppm for the first 4 days of operation, to 60 ± 20 ppm for the following 11 days, then to 80 ± 10 ppm for the remainder of the 30-day test period. Over 30 days of operation, 15,000 m^3 of air was scrubbed to acceptable CO_2 concentration for alkaline fuel cells while consuming 12 kg of MEA and 310 W electric power for the regenerator heater, which is within the specifications for a 5 kW (output) AFC demonstration system.

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