African Journal of Environmental Science and Technology

Full Length Research Paper

Removing carbon dioxide from a stationary source through co-generation of carbonate/bicarbonate: The case of Mugher cement factory

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The cement industries alone emit 5% of the world's total green house gases. In Ethiopia, the current CO_2 emission from this sector is about five fold of the countries overall CO_2 emission in 2002. Mugher cement factory (MCF), a case study plant in this paper, alone emits 463,844 tons of CO_2/year on average based on emission test and mass balance performed. Yet it accounts for less than 3% of the current CO_2 emission from this particular industry in the country. Of those technical approaches, separation of CO_2 before it joins the immediate atmosphere is getting wide spread interest. In relation to this, experiment on absorption of CO_2 is conducted to determine the effect of flow rate of the gas sample, concentration of the sodium hydroxide, flow rate of solvent and temperature of absorbent on absorption of CO_2 using the "Armfield" gas absorption column followed by the titrimetric analysis. Except temperature of solvent, all study variables showed strong relation with the amount of carbon dioxide absorbed (with a P-value < 0.05). Uniquely, this study has evaluated the potential for sodium bicarbonate production from the CO_2 absorbed using gravimetric analysis. It is also possible to recover over 28% crystal sodium bicarbonate.

Key words: Green house gas, CO_2, absorption, caustic soda, sodium bicarbonate.

INTRODUCTION

It is estimated that the global average temperature will rise between 1.4-5.8°C by the year 2100. The contributors to greenhouse effects are carbon dioxide (CO_2), chlorofluorocarbons (CFCs), methane (CH_4) and nitrous oxide (N_2O). The contribution of each gas to the greenhouse effects is CO_2- 55%, CFCs- 24%, CH_4- 15% and N_2O- 6% (Moazzem et al., 2012). The cement industry alone emits about 5% of the world's CO_2 emis-

sion (IPCC, 2005; Huntzinger and Eatmon, 2009).

Hydraulic (chiefly portland) cement, the binding agent in concrete and most mortars, is an important construction material. Portland cement is made primarily from finely ground clinker, a manufactured intermediate product that is composed predominantly of hydraulically active calcium silicate minerals formed through high-temperature burning of limestone and other materials in a
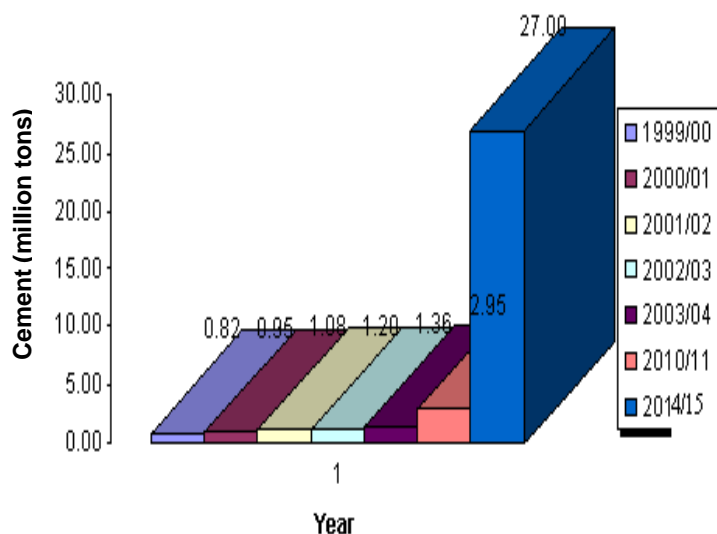
Figure 1. Cement production trend of Ethiopia in unit of tons per year with plan.

kiln. This process typically requires approximately 3.2 to 6.3 GJ of energy and 1.7 tons of raw materials (chiefly limestone) per ton of clinker produced and is accompanied by significant emissions of, in particular CO_2, but also nitrogen oxides, sulfur oxides and particulates. The overall level of CO_2 output, about 1t on/ton of clinker, is almost equally contributed by the calcinations of limestone and the combustion of fuels and this makes the cement industry one of the top two manufacturing industry sources of this greenhouse gas (Van Oss and Pandovani, 2003).

The concentration of CO_2 in the flue gases of cement plants is between 15-30% volume by volume, which is higher than in flue gases from power and heat production (3-15% by volume). Therefore, in principle, the post-combustion technologies for CO_2 capture could be applied to cement production plants (Hendrik et al., 2000).

Cement production sector is booming these days in Ethiopia (Figure 1) to meet the current demand for massive construction of infrastructures, which is mushrooming from time to time and related as well to economic growth of the country. The current average cement production of the country is about 2.95 million tons per year. It is expected to reach 27 million tons by 2017/18 and all needs attention on CO_2 emissions according to Ethiopian Investment Agency, (2011). In other words, by the year 2019/20 the cement production of the country could double India's current production.

Among several techniques for CO_2 separation, chemi=-cal solvent scrubbing, physical solvent scrubbing, adsorption, membranes, cryogenics and solid sorbents are the major ones. Gas absorption, as applied to the control of air pollution, is found feasible with regards to the removal of one or more pollutants from a contaminated gas

stream by treatment with a liquid, though the cost implied (Mohammad et al., 2007), is also an issue. The necessary condition is the solubility of these pollutants in the absorbing liquid.

Of those gas absorption equipments, the packed column as applied in this study is by far the most commonly used for the absorption of gaseous pollutants (Reynolde et al., 2002). Currently the favoured method for post-combustion removal of CO_2 from flue gases uses chemical solvents. Generally, CO_2 capture from cement plants is more efficient than that from pulverized coal fired power plant or related others (Shuangzhen and Xiaochun, 2012). A sodium hydroxide solution provides a liquid sorbent that is more easily cycled through a piping system than a calcium hydroxide suspension in absorbing CO_2. Sodium hydroxide solution is selected as absorbent in this study due to availability as matter of sustaining the solution and manageability. Its binding energy is strong enough and its reaction kinetics is fast enough to prevent the need for heating, cooling or pressurizing the air. Because CO_2 is so dilute, any such action would result in an excessive energy penalty. The hydroxide solution avoids all such complications (Davison and Thambimuthu, 2005).

In another scenario, CO_2 is used as input in various applications (Chapel et al., 1999) including industrial ones. Thus, this study has also considered the production of bicarbonate from the recovered CO_2.

This study has first characterized the flue gas composition of the case plant, MCF, and assembled material balance around the kiln. Thus, it determined the country's over all CO_2 emission from this particular sector. Following that, absorption experiment is conducted on the gas sample, which is 20% CO_2 on average, with dilute caustic soda solution. At the same

time, it determined the carbonate and bicarbonate as desirable products using titrimetric and gravimetric analysis.

The objective of this study was to quantitatively determine the CO_2 emission from cement plants and direct the options to turn this green house gas to useful product by applying chemical absorption method. From doing so, the study can be considered as options to produce useful industrial products not from use of fossil fuel, thereby avoiding purchase of foreign currency. While at the same time, it evaluates the factors in chemical absorption of CO_2 to suggest the optimum.

MATERIALS AND METHODS

Chemicals

The method of wet scrubbing chosen to treat a given gaseous pollutant is always specific to the given pollutant present in the gas stream treated. A "standard" wet scrubber does not exist (Wang et al., 2004). However, dilute sodium hydroxide solution as solvent is used in this study. Hydrochloric acid and acetic acid were used for the titrimetric determination of carbon dioxide absorbed and gravimetric determination of the sodium bicarbonate produced. Barium chloride solution was used for titration process. Carbon dioxide is the analyte of interest in this study, and distilled water was used for the titration process. Reagent-methyl orange and phenolphthalein were used for end-point detection of the acid-base titration.

Materials

In this study, a range of equipment and materials were used including gasbags, crucibles, oven, compressors, pumps, gas cylinders, heat exchangers, titration apparatus, analytical balance and reagent chemicals as well as gas analyzer.

Sampling has been made using pumping system built in the cement plant following particulate removal by the bag house and cooling by shell and tube method. The analysis of gas sample was made using a gas analyzer (LI-820 CO_2 Analyzer, LI-COR Europe).

Thermometer was used to monitor temperature of room and solvent. pH meter was used to monitor the basic level of the sodium hydroxide solution. Electronic balance is used in the weighing of pellets and crystals mainly. In addition, spoon, stopwatch, conical flask, volumetric flask, beakers, burette and burette stand, measuring cylinder, pipette, gasbags and other equipments were also used.

"Armfield" gas absorption column was used to run the experiment. Thermostat was used as regulated heat source to heat the solvent. The titrimetric determination of carbon dioxide absorbed and gravimetric determination of the sodium bicarbonate produced was performed in this study respectively using bunch of analytic chemistry wares.

Experimental design

The response variable in this study was CO_2 separated followed by the bicarbonate produced. The study variables in this paper include temperature, concentration and flow rate of the liquid and flow rate of the gas. Each factor was checked at two levels: T_1, T_2, (°C) for temperature, C_1, C_2, (mg/l) for concentration, Q_{l_1}, $Q_{l/2}$ for liquid flow as well as Qg_1 and Qg_2 all in lit/min for the gas flow rates. Thus, the type of experimental design involved was full factorial design.

Experimental setup

Gas absorption experiment was carried out in vertical counter current packed column 75 mm in diameter in which there are two lengths of contacting column each 70 cm long. The liquid solvent was fed at the top of the column and was distributed over the surface of the packing by nozzle (Figures 2 and 3). Pressure tapping was provided at the base, centre and top of the column to determine pressure drops across the column jointly with other measurements in need.

Sampling points were provided for the gas at the same three points. The liquid outlet stream and feed solution were also equipped with sampling point. Suitable manometer measurement was included. The solvent is taken from a sump-tank and it is pumped into the column using a calibrated flow meter attached on the board.

Gas was taken from a pressure regulated cylinder through a calibrated flow meter, and mixed with air, supplied and monitored from a small compressor in a pre-determined (but variable) mixed ratio. The mixture was fed to the base of the tower, in which a liquid seal was provided. The exit gas leaves the top of the column. The apparatus is designed to absorb CO_2/air mixture into an aqueous NaOH solution flowing down the column.

The emission is first pre-treated with cyclone and bag house. The analyte and air is compressed to a cylinder from which regulated flow of gas is fed to absorber from bottom and the diluted caustic soda from top. Samples of liquid are analysed from the exit valves of the column (S_1, S_2 and S_3) while the bottom exist is further treated for carbonate/bicarbonate determination (Figure 2).

Procedure and analysis

Initially, the characterized flue gas is simulated using the commercially available carbon dioxide in a given purity. This is achieved by mixing such commercial gases and the atmospheric air. Before the start of the absorption, the pressure regulated CO_2 cylinder located in a suitable storage rack was adjusted in rear side of the column. The gas was connected to the column by a low-pressure regulator that was located at the rear of the vertical backboard.

The predetermined molar solvent was prepared after weighing the commercial grade NaOH pellet mixed with distilled water. Thus, several runs with aqueous NaOH solution were made as specified by the design of experiment. After equilibrium stage was maintained, from using the gas and solvent flow rate regulation through their valves, samples of exit solvent as well as gas were collected. The exit sample from each run was tested five times using the arm field gas absorption manual on titration, coded as UOP7.

The amount of bicarbonate, which precipitates out of solution at sufficiently high concentration, was determined using gravimetric analysis (Olutoye and Mohammed, 2006). The data generated from conducted experiments was analyzed using software-STATISTICA Version 8 (Stat Soft.Inc.2008), Design-expert version 7.0.0 (Stat-Ease, Inc. 2005) and CHEMCAD 5.2.0.

RESULTS AND DISCUSSION

Characterization of CO_2 in the flue gas

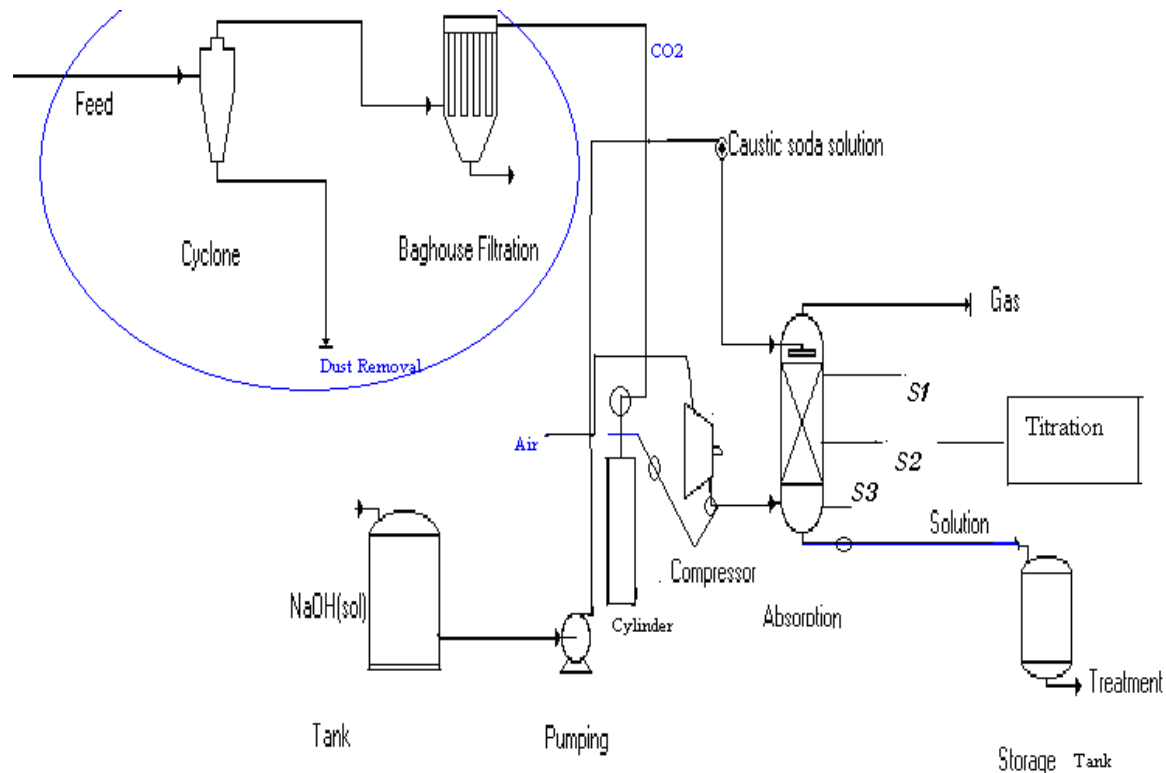The flu gas in cement industries is generated when

Figure 2. Process flow diagram showing the setup from sampling to absorption experiment.

Figure 3. The "Armfield" gas absorption experiment column at the mass transfer laboratory of the Addis Ababa Institute of Technology, Addis Ababa University, Addis Ababa, Ethiopia.

making clinker mainly in the kiln at which high temperature roasting of calcium carbonate is achieved (Figure 4). The suspended particles with very high temperature rose up to bag houses where particles other than air are filtered. This exit gas then moves to cooling section where it looses its high temperature down to 45°C using shell and tube heat exchanger. The gas that already lost its warmth is tapped in to empty gasbags before it is tested for composition using the gas analyzer.

The analysis for composition of the 'clean' gas is made using gas analyses at the premises of The Federal Environmental Protection Authority of Ethiopia. The composition expressed in percentage by volume of those gases tested many times showed close results and the CO_2 is found to be 20% on average (Table 1).

Mass balance

The cement process involves gas, liquid and solid flows with heat and mass transfer, combustion of fuel, reactions of clinker compounds and undesired chemical reactions that include sulfur, chlorine, and alkalis. It is important to understand these processes to optimize the operation of the cement kiln, diagnose operational problems, increase production, improve energy consumption and lower emissions (Figure 4).
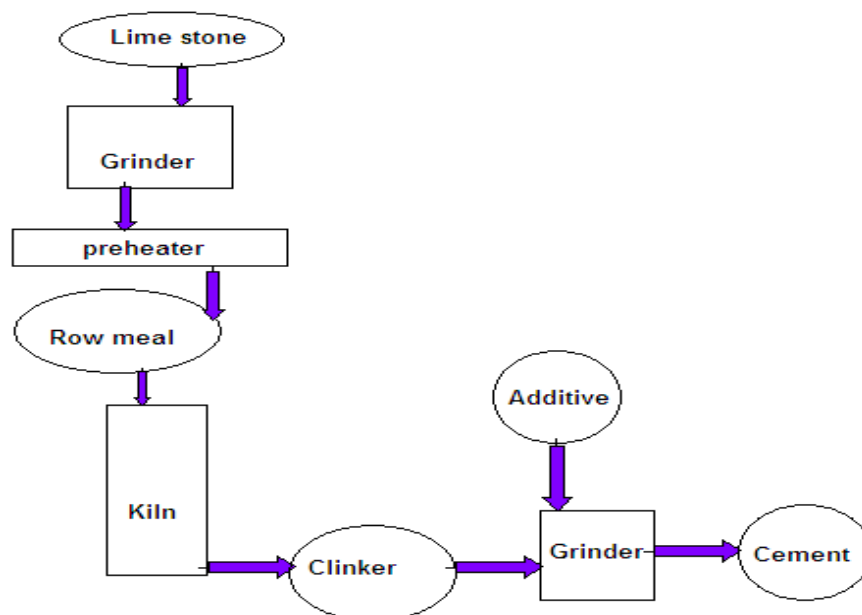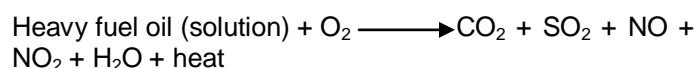
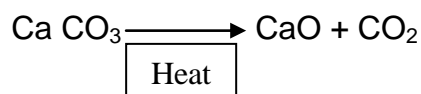The calcination and the fuel combustion chemistry

Figure 4. Simple schematic layout of central of cement making process at MCF (using CHEMCAD 5.2.0).

Table 1. Result of emitted gas analysis of MCF for CO_2 in percent by volume.

Parameter analyzed	Sample gas bags (percentage by volume)			Average (percentage by volume)
	A	B	C	
Carbon dioxide (CO_2)	20.0	19.8	20.2	20.0
SOx	0.4	0.45	0.4	0.4
Total nitrogen	78	78	80	78.7

involve the following reaction:

$$Ca\,CO_3 \xrightarrow{\text{Heat}} CaO + CO_2$$

Heavy fuel oil (solution) $+ O_2 \longrightarrow CO_2 + SO_2 + NO + NO_2 + H_2O +$ heat

The heavy fuel oil consumption rate, based on four years average of most recently collected data, is 102.025 litters/ton of clinker, with working days of 261.24 days/year, production rate of 515382 tons of clinker/year. The row meal factor of the plant is about 1.73 and the row material is $CaCO_3$.

The 20.0% (percentage v/v) of the emission is contributed by CO_2 which conforms to the literature reports of 15-30% and is above average (Davison and Thambimuthu, 2005). From the mass balance performed on 1 kg clinker basis (Figure 5), it is clear that for every 1 kg of clinker formed almost 0.6 kg of CO_2 is released to the atmosphere from this study plant. This gives to current sub total annual average of 309,229.2 tons from calcining process alone. Additionally, 0.3 kg of CO_2 is emitted from combustion of fuel/1 kg of clinker produced. This figure shows that the emission of CO_2 from this particular sector alone in the country double and exceeds the overall country emission recorded by Ministry of Transports and Communications of Ethiopia for 2002 even though there are still new factories in buildup process. However, according to data by The World Bank Ethiopia, it is still least emitter of CO_2 per capita even within the sub Saharan region.

The CO_2 absorption experiment

The rate of CO_2 absorption and hence bicarbonate formation is affected by such variables as the state of completion of the chemical reaction, the liquid flow rate, the absorbing surface area, the liquid temperature and the concentration of CO_2 gas (Treybal, 1981).

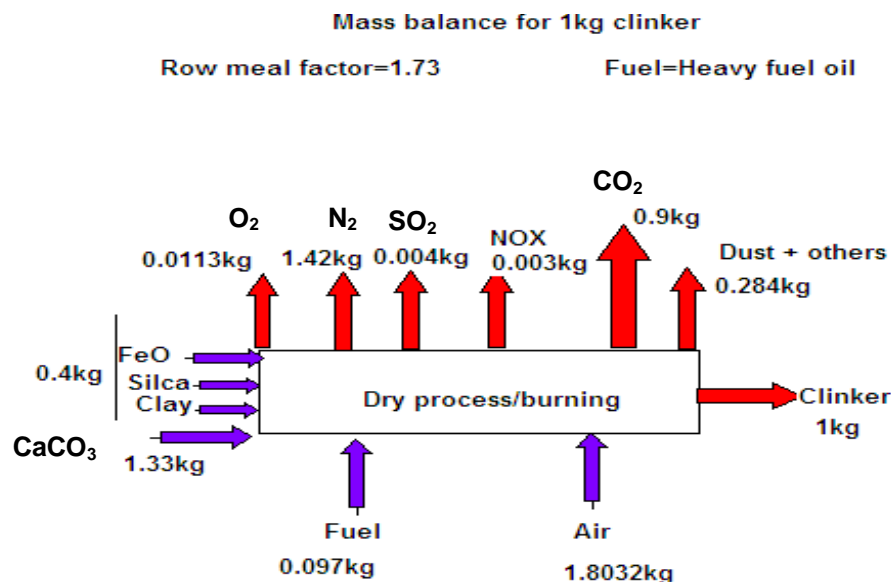As per the design of experiments, 16 run for the factors

Mass balance for 1kg clinker

Row meal factor=1.73   Fuel=Heavy fuel oil

Figure 5. Summary of the mass balance performed for the case plant on 1 kg clinker basis (CHEMCAD 5.2.0).

Table 2. Summary of the results of the absorption experiment.

Run	C_{N_aver} (g-mol/Lit)	CO_2_Xaver (g-mol/sec)	Conc (M), NaOH	Q_{liq} Lit/min	Q_{gas} Lit /min	% CO_2	% air	Temperature (°C)	Run time (min)
1	0.06395	0.001599	0.2	1.5	4	16.7	83.3	22.8	13
2	0.0528	0.00129	0.2	1.5	5	20	80	22.8	13
3	0.0544	0.001118	0.2	1	5	20	80	23.2	14
4	0.0504	0.000946	0.2	1	4	16.7	83.3	23	14
5	0.0166	0.000818	0.2	1	5	20	80	47.2	14
6	0.00172	3.17E-05	0.1	1	5	20	80	25	14
7	0.0137	0.000343	0.1	1.5	5	20	80	44.6	12
8	0.00994	0.00028	0.1	1.5	5	20	80	23.6	11
9	0.0108	0.000181	0.1	1	5	20	80	43.6	12
10	0.023	0.000575	0.2	1.5	5	20	80	46.6	12
11	0.0166	0.000818	0.2	1	4	16.7	83.3	47.2	14
12	0.023	0.000575	0.2	1.5	4	16.7	83.3	46.6	12
13	0.0108	0.000181	0.1	1	4	16.7	83.3	43.6	12
14	0.0137	0.000343	0.1	1.5	4	16.7	83.3	44.6	12
15	0.00172	3.17E-05	0.1	1	4	16.7	83.3	25	14
16	0.01518	0.000566	0.1	1.5	4	16.7	83.3	23.6	11

of study: concentration of the caustic soda, gas flow rate, solvent flow rate and temperature of solution were conducted all at two levels. Data of interest including temperature of room, pH of solvent and others were recorded. The results of each run were tested/observed five times using the arm field gas absorption manual (titration) coded as UOP7. Each test was made after thermodynamic equilibrium conditions were attained, within 10-15 min based on velocity control on gas and solvent flow rates.

The titrimetric analysis for determining absorption rate for the exit liquid was made with standard method for neutralization of not reacted sodium hydroxide followed by the stochometric analysis, carbonate and bicarbonate. With this method it is assumed that there is no free CO_2 in the liquid and the absorption rate is determined after the concentration of NaOH and $NaCO_3$ are calculated (Table 2). Fast reaction kinetics and high affinity of CO_2 towards caustic soda kept the side reactions to a minimum that can be neglected for this paper.
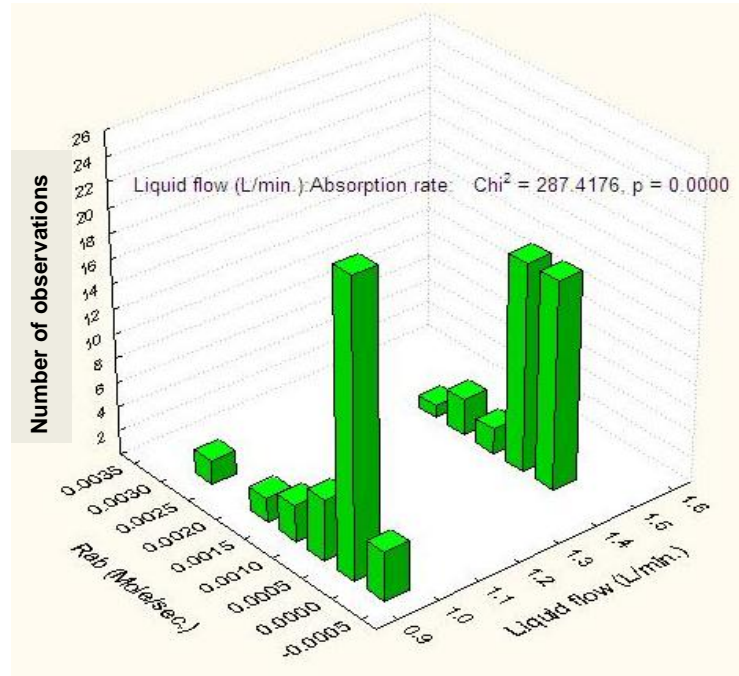
Figure 6. Bivariate histogram showing rate of absorption against liquid flow rate.

The amount of CO_2 absorbed across the column as measured from samples taken simultaneously from the sump tank feeding the column top and at the bottom outlet, is given by:

$$CO_{2\,absorbed} = liquid\ flow\ rate \times [(C_N)o - (C_N)i]$$

or

$$CO_{2\,absorbed} = liquid\ flow\ rate \times \frac{1}{2}[(C_N)i - (C_N)o]$$

Similarly, over a time period, θ seconds after a first sample is taken from the sampling point:

$$CO_{2\,absorbed} = liquid\ flow\ rate \times [(C_N)o - (C_N)i]$$

Where, the subscript 0 here is t=θ, and 'i' is time at t = 0
Caustic soda solution absorbs carbon dioxide by chemical reaction in two steps:

I. $NaOH + CO_2 \longrightarrow Na_2CO_3 + H_2O$
II. $CO_2 + Na_2CO_3 + H_2O \longrightarrow NaHCO_3$
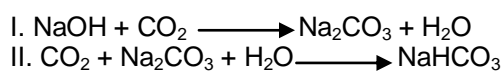
As shown by the above equations, 1 kg of pure sodium hydroxide absorbs 1.1 kg of carbon dioxide. One-half is absorbed in each step; however step I takes place much faster than step II. Obviously, each reaction has special characteristics that may affect the operation of an absorber; for example, reaction II is reversible in that sodium hydroxide will react with sodium bicarbonate to form sodium carbonate (Pflug et al., 1957).

Liquid flow rate

The solvent flow rate selected as a study independent variable, set by the investigator, is shown to accompany statistically significant response (Figure 6). This result agrees with other study that showed the rate of CO_2 absorption to be directly proportional to the liquid flow rate on average. It is also evident that carbon dioxide absorption is a surface phenomenon; consequently, the contact area between the absorbing fluid and the gas has an important effect on the rate of absorption. A similar expression otherwise from literatures is also evidencing as stated like that at a fixed gas velocity, the gas-pressure drop increases with increased liquid rate, principally because of the reduced free cross section available for flow of gas resulting from the presence of the liquid (Pflug et al., 1957; Treybal, 1981; Lin and Chen, 2007; Thomsen, 2002). However, there are also deviant results ascribed to error.

Liquid temperature

Absorption of a pollutant is enhanced by: lower temperatures, greater liquid-gas contact surfaces, higher liquid-gas ratios and higher concentration of the pollutant
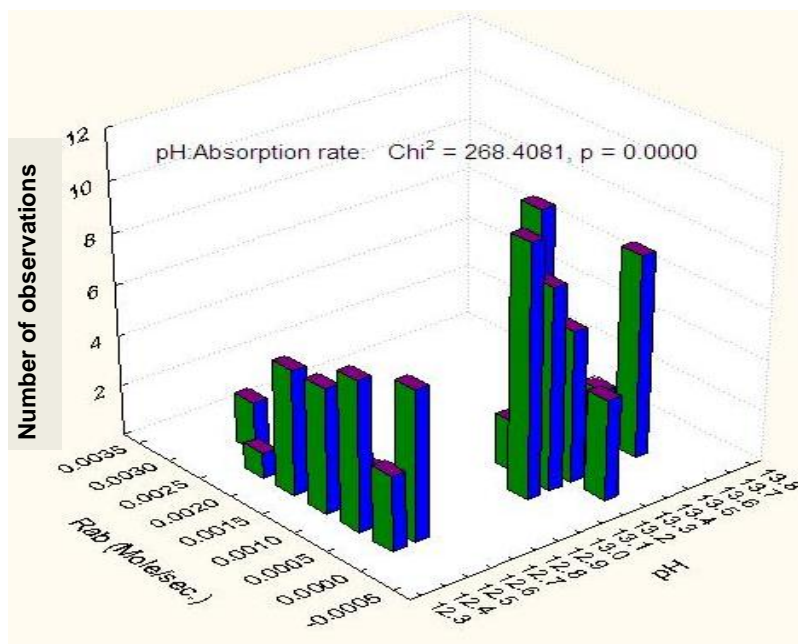
Figure 7. Bivariate histogram showing rate of absorption against pH of solvent.

in the gas phase (Davison and Thambimuthu, 2005). This study has also showed rate of absorption to be indirectly related to solvent temperature but it was not statistically significant. This could be explained thus: as the temperature of liquid increase the velocity at which the moving liquid goes through the absorption column also increases. That implies the retention time would be low and hence absorption of the gas decreases as well affecting the overall rate of absorption. However, some investigators showed that increasing the temperature of the absorbing fluid generally increase the rate of absorption; however, temperature has a greater effect on reaction II than on reaction I (Treybal, 1981).

Sodium hydroxide concentration (pH)

The pH of the scrubbing liquor is often an important process parameter. Low-pH liquor is required for ammonia scrubbing, neutral or high pH is needed for acid gas scrubbing (Wang et al., 2004). Dry solid, commercial grade caustic soda that contains 98.8% NaOH, available as regular flake, is used in this study. On analysis, concentration of the caustic soda showed a statistically significant relation with rate of absorption (Figures 7 and 8).

Pollutant gas (CO_2) velocity

Increasing the gas velocity beyond needed does not support the absorption process rather it tends to cool the

solvent temperature (Pflug et al., 1957). Elevating the gas and hence air velocity could create resistance to liquid fall- the phenomena called loading, if increased still could lead to flooding thus it has to be variable of interest in the design of the mass transfer equipment (Thomsen, 2002). This study has also demonstrated a statistically significant difference between gas velocity and rate of absorption (Figure 9).

Cosidering factor interactions, the four factors of absorbtion were analysed by using Design-Expert version 7.0.0 (Stat-Ease,Inc.2005) for developing a model equation that can be used in order to develop the laboratory scale finding to an application scale. The ANOVA for the data enterd proved the model to be significant (Table 3). In relation to this factor interactions were points of interst and the factors coded AD and BD showed significant effect where A refers to concentration, B refers to liquid flow rate, C refers to gas flow rate and D refers to temperature.

Further more, the *F- value* for the model is 4.27 which, in another check, implied the model to be significant. The A, D, AD, BD were the model terms that showed significant effect. A related parameter called "*Adeq Precision*" measures the signal to noise ratio. A ratio greater than 4 is desirable. Here ratio of 7.324 indicates an adequate signal. Therefore, this model can be used to navigate the design space.

Sodium bicarbonate production

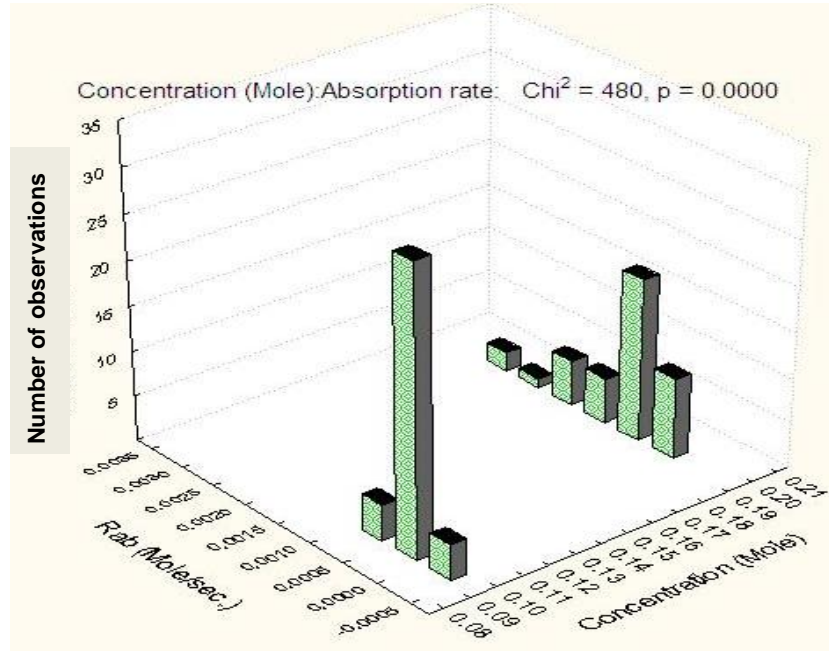Despite the fact that CO_2 has climate change impact, it is

Figure 8. Bivariate histogram showing rate of absorption against NaOH concentration.
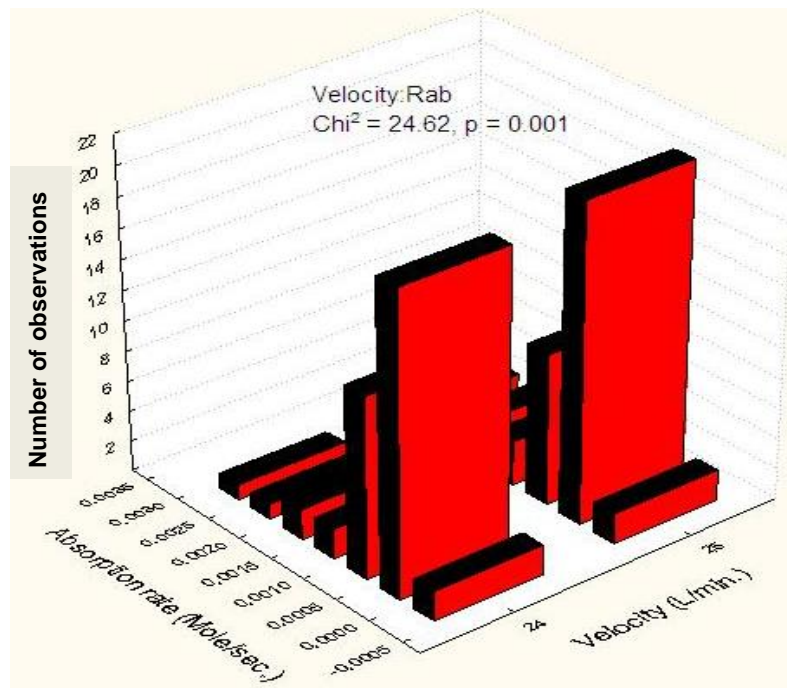

Figure 9. Bivariate histogram showing rate of absorption against gas velocity.

being produced even from use of fossil fuels for various application (Chapel et al., 1999); including beverages, hospital use and others (Table 4).

In the CO_2 capture and storage attempts, a number of investigations showed stripping of the separated CO_2 to store it at pressure somewhere underground. In this study, however, production of sodium bicarbonate is considered than stripping process, which is aimed at

Table 3. Analysis of variance for the selected factorial rate of absorption model.

Source	Sum of squares	Degree of freedom	Mean square	F-value	P-value Prob>F	Remark
Model	1.761E-005	15	1.174E-006	4.27	<0.0001	
A-concentration	1.107E-005	1	1.107E-005	40.26	<0.0001	
B-liquid flow rate	7.898E-007	1	7.898E-007	2.87	0.0950	
C-gas flow rate	1.006E-007	1	1.006E-007	0.37	0.5474	Significant
D-temperature	1.478E-006	1	1.478E-006	5.38	0.0236	
AD	1.854E-006	1	1.854E-006	6.74	0.0117	
BD	1.158E-006	1	1.158E-006	4.21	0.0442	

Table 4. Some examples of the use of CO_2 in the industry.

Food industry	Process industry
Breweries	Enhanced Oil recovery (EOR)
Carbonated beverages	Precipitated Calcium Carbonate for paper
Quick freezing of meats and vegetables	Inert gas for welding
Flash drying of food	Casting
Grain fumigation	Methanol production
Photosynthesis enhancement	Neutralizing of alkalis in process water
Slaughterhouses	Inert gas blanketing

large on managing the problem and at the same time to produce CO_2 based sodium bicarbonate (Ellison, 1984). Sodium bicarbonate is the chemical compound with the formula $NaHCO_3$. Sodium bicarbonate has much application and it is an industrial chemical. Among the various uses of this chemical cooking/leavening agent, neutralization of acids and bases, as a deodorizer, medical uses, cosmetic uses, as a cleaning agent and as a fabric softener in laundry can be mentioned.

The yield from absorption column is at left stand still for minimum of 2 h to separate the solid by precipitation. The moisture of the precipitate is evaporated using evaporating dish, oven and outdoor light at temperature below 70°C for a long time in order to avoid heat breaking of the $NaHCO_3$. Thus, this study, based on gravimetric analysis (Marı´a et al., 2004) produced up to 28% pure sodium bicarbonate where the rest constitute sodium carbonate and un-reacted caustic soda.

Conclusion and recommendation

Cement production in the country increased from < 0.1 million tons per year in 1999/00 to 8 million currently and even in progress towards reaching 27 million by 2015 owing to massive construction. MCF emits more than 463,844 ton of CO_2/year on average currently and yet it accounts for only <3% of overall country emission from this particular sector. An end-of-pipe technology to reduce carbon emissions may be CO_2 removal. Probably,

use of chemical solvent scrubbing could be the favoured method. In absorbing the pollutant gas, temperature did not have statistically significant effect unlike the concentration and pH. This study results partly help in planning an effective CO_2 removal method applying solvent scrubbing. Again, production of bicarbonate can be considered as an advantage of recovering this gas from such waste streams. However, further significant research should be done to complete the work by providing a concrete and useful framework plan with quantitative technical recommendations. Governments need to support such action in order for it to be approached at pilot scale. Further research including consideration of other option to manage such problem has to be done.

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