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Absorption of Carbon Dioxide by Water under Pressure in a Packed Tower

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In the industrial manufacturing of hydrogen, which is to be used under considerable pressure, for example, for ammonia synthesis or coal hydrogenation, from water gas it is necessary, as the first step of purification, to remove CO_2 from the mixture of hydrogen and CO_2 obtained by the catalytic reaction between steam and water gas. Although the CO_2 removal in the above cases has been successfully carried out by means of the pressure absorption in water through the packed towers, data sufficient to evaluate the performance of commercial absorption towers or to design them are not available.

The author carried out preliminary experiments on pressure absorption of CO_2 by a semi-technical-scale absorption tower to obtain the capacity coefficient $K_L a$ as well as additional data concerning the loss of hydrogen accompanied by the CO_2 absorption.

Apparatus and Procedure

The apparatus employed is indicated diagrammatically in Figure 1. The absorption tower used was 450 mm. in diameter, dumped packed to a height of 2,300 mm. with 29 mm. (diameter) \times 50 mm. (length) aluminium rings, the free volume of the packed bed being 87.4%. The tower, constructed of steel cylinder, had the total height of 4,000 mm. and withstood normal working pressure of 20 kgs/cm^2 . Some details of the absorption tower are shown in Figures 2, 3 and 4.

Water as solvent was underground water which, once pumped up into a storage tank, was fed at the top of the tower at about 20 kgs/cm^2 pressure by a triple-acting plunger pump. The pump, driven by a direct current shunt motor, had an adjustable feed capacity, its maximum capacity being about 6.5 m^3/hr . One litre of feed water contained about 3—6 c.c.* CO_2 .

* Calculated at 0°C and 760 mm Hg.

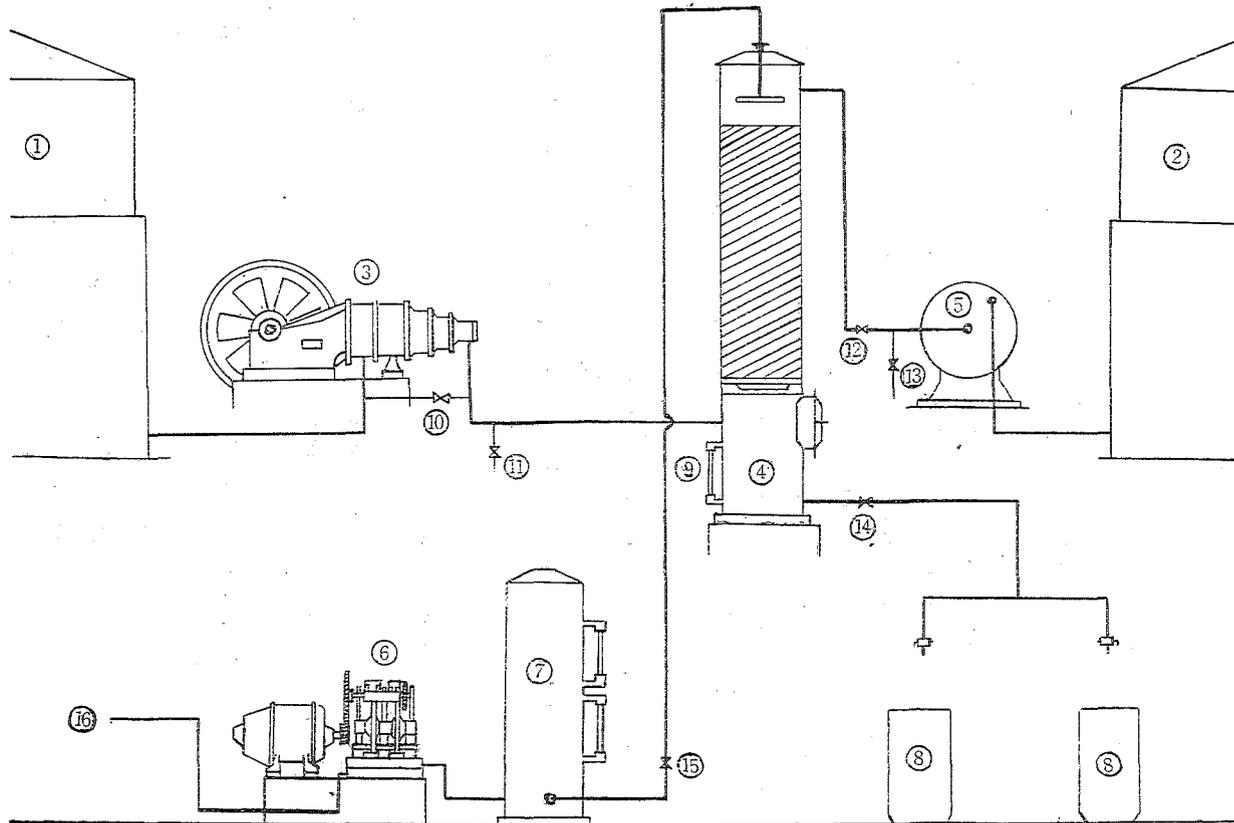


Fig. 1. Flow Diagram of Absorption Tower System

- | | | |
|------------------------------|--------------------------------------|-----------------------------|
| 1. Gas holder for raw gas | 7. Storage vessel for pressure water | 13. Cock for gas sampling |
| 2. Gas holder for washed gas | 8. Vessel for measuring waste water | 14. Pressure reducing valve |
| 3. Gas compressor | 9. Glass level gauge | 15. Valve |
| 4. Absorption tower | 10. Bypass valve | 16. Feed water |
| 5. Gas meter for washed gas | 11. Cock for gas sampling | |
| 6. Plunger pump | 12. Pressure reducing valve | |

Raw gas, 100 — 200 m³ in each experiment, was prepared either by the catalytic reaction of water gas with steam or by mixing waste hydrogen from coal hydrogenation process with commercial liquified CO₂. Thus the raw gas was composed of 14.2—26.4 % CO₂ and 85.8—73.6 % solute-free gas, and its main component was hydrogen (table 1). A three-stage gas compressor was used, with its normal suction capacity of 250m³/hr. and normal working pressure of 20 kgs/cm².

The volume of raw gas consumed, was measured by a 280 m³ gas holder every twenty minutes in a run, and that of a washed gas, after its pressure was relieved, was measured by a 250 m³/hr. gas meter and another 280 m³ gas holder at the same time intervals.

Water rate was determined by measuring the volume of waste water by using two vessels, the capacity of each being about 150 liters.

When starting a run, the gas compressor was driven and air in the tower was replaced by raw gas; the plunger pump was also driven and water was stored in a high pressure storage vessel. The pressure of gas in the tower was gradually raised to a working pressure, 20 kgs/cm². Then, water was fed at the pressure of 22 — 23* kgs/cm² through a spray nozzles distributor located at the top of the tower, and water flowed down through the packing making a countercurrent to gas stream.

The level of the water absorbing CO₂ at the bottom part of the tower was held exactly at a fixed point in a glass level gauge throughout a run, by adjusting the feed capacity of the pump as well as the opening of a pressure reducing valve of waste water. Residual CO₂ content in the washed gas leaving the tower was measured by alkaline solution (Hempel's gas analysis method) at intervals of five minutes.

After the concentration of CO₂ in the washed gas became constant under a prescribed operation condition, the apparatus was allowed to run for an hour in a steady state.

During the time of a steady state operation, measurements of flow rates of gas and water, temperatures, pressures and analyses of the gas stream at both points of inlet and outlet of the tower, were taken for calculation of the coefficient of material transfer.

Calculation of Capacity Coefficient, K_La

For the purpose of engineering computation of the capacity coefficient, K_La , in the present experiments, the author assumed that Henry's law was applicable

* Delivery pressure of the pump.

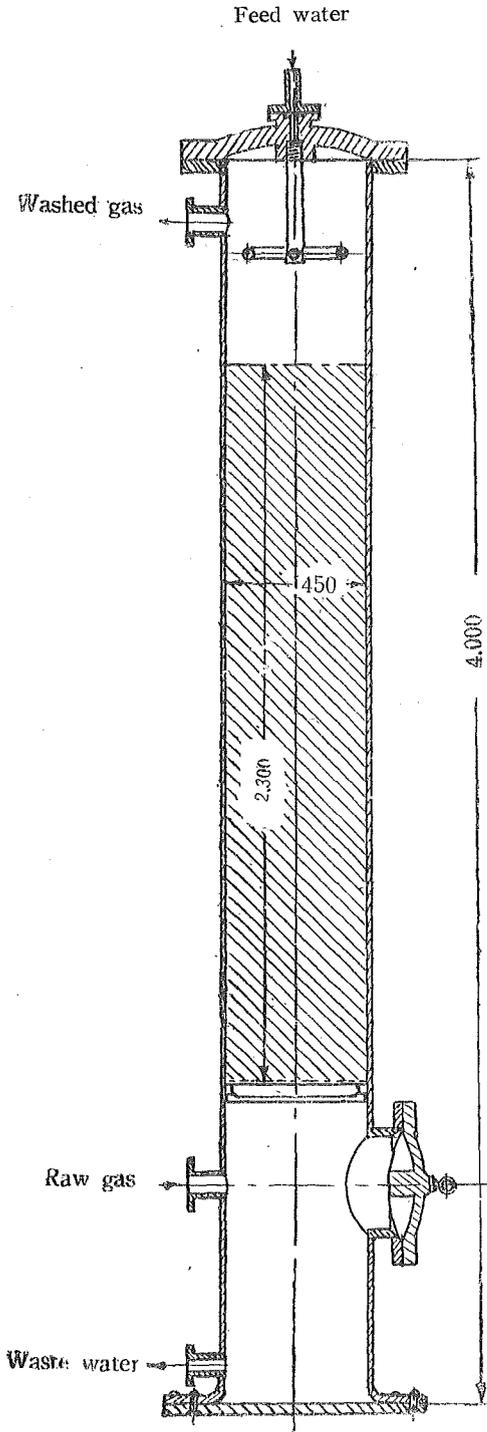


Fig. 2. Absorption Tower

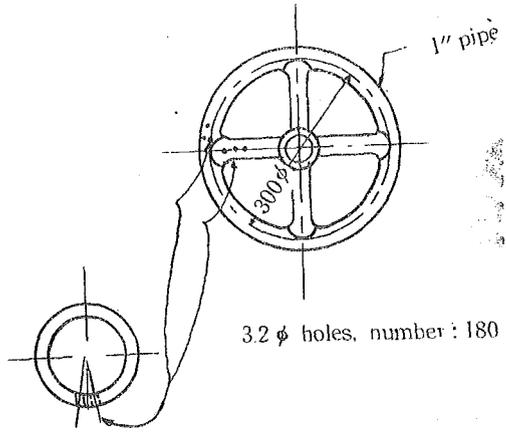


Fig. 3. Water Distributor

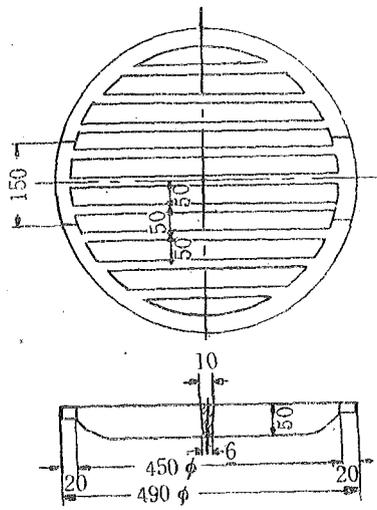


Fig. 4. Packing Support

over the range of the experimental conditions, and employed a simplified procedure, a numerical calculation instead of the usual graphical integration method, in which the following symbols and industrial units were adopted.

L : water rate, (m³/hr.).

G : gas flow, (m³/hr.).

β : ratio of water rate to gas flow, (L/G).

x : CO₂ concentration in liquid, (m³-CO₂) / (m³-water).

y : CO₂ concentration in gas, (m³-CO₂) / (m³-solute-free gas).

x_1 : CO₂ concentration of the liquid leaving the bottom (gas inlet), (m³-CO₂) / (m³-water).

y_1 : CO₂ concentration of the gas entering the column,* (m³-CO₂) / (m³-solute-free gas).

x_0 : CO₂ concentration of the liquid fed at the top (gas outlet), (m³-CO₂) / (m³-water).

y_0 : CO₂ concentration of gas leaving the top of the column, (m³-CO₂) / (m³-solute-free gas).

x_g : CO₂ concentration corresponding to equilibrium with the CO₂ concentration in the main body of the gas, (m³-CO₂) / (m³-water).

π : working pressure, (absolute atm.).

α : Bunsen's solubility coefficient of CO₂ in water,¹⁾ (m³-CO₂) / (m³-water) (atm.).

H : height of packed bed, (m).

S : sectional area of column, (m²).

Then

$$G (y - y_0) = L (x - x_0) \quad (1)$$

$$x_g = \alpha \pi \frac{y}{1 + y} \quad (2)$$

$$\int_{x_0}^{x_1} \frac{dx}{x_g - x} = \frac{K_L a \cdot S \cdot H}{L} \quad (3)$$

Formulas (1) and (2) are obtained from material balance in the column in a steady state and Henry's law, respectively. Then $K_L a$ was obtained by calculating the left term of formula (3) as follows:

from (1), (2) and (3)

* In the present experiments, CO₂ concentration of the entering gas was measured at the inlet pipe, 11 in Figure. 1.

1) Landolt : Tabellen, 5 Aufl., S. 768 ; Seidell : Solubilities, Vol. 1, p. 221 (1940).

$$\begin{aligned}
 \int_{x_0}^{x_1} \frac{dx}{x_0 - x} &= \int_{x_0}^{x_1} \frac{(y_0 - \beta x_0 + 1 + \beta x) dx}{\alpha\pi (y_0 - \beta x_0) + (\alpha\pi\beta - y_0 + \beta x_0 - 1) x - \beta x^2} \\
 &= \int_{x_0}^{x_1} \frac{(A + \beta x) dx}{C + 2Bx - \beta x^2} \\
 &= -\frac{1}{2} \left[\ln (C + 2Bx - \beta x^2) \right]_{x_0}^{x_1} + \frac{A + B}{\sqrt{4B^2 + 4\beta C}} \left[\ln \frac{2B - 2\beta x - \sqrt{4B^2 + 4\beta C}}{2B - 2\beta x + \sqrt{4B^2 + 4\beta C}} \right]_{x_0}^{x_1}
 \end{aligned}$$

where

$$\begin{aligned}
 A &= y_0 - \beta x_0 + 1 \\
 2B &= \alpha\pi\beta - y_0 + \beta x_0 - 1 = \alpha\pi\beta - A \\
 C &= \alpha\pi (y_0 - \beta x_0)
 \end{aligned}$$

A, 2B, C, S, H and L are quantities either measured experimentally or obtained indirectly from experimental measurements.

Results

The data obtained are shown in Table 1. So far as the present preliminary experiment was concerned, the author recognized the following points that almost agreed with other investigators' results²⁾ of CO₂ absorption under atmospheric pressure.

- 1) $K_L a$ increased with water rate in Exps. 5 a and 5 b. Although the water rate in Exp. 12 was increased as compared with that in Exps. 7 a and 7 b, $K_L a$ showed practically no change. The fact seemed to be attributable, partly, to a remarkable non-uniformity* of liquid distribution in the tower which occurred when water rate exceeded 5 m³/hr.
- 2) $K_L a$ was slightly effected by gas velocity in Exps. 7 a and 7 b.
- 3) The effect of temperature on $K_L a$ was noticed in Exps. 5 b and 7 a.

Loss of Solute-free Gas Dissolved in Water

In experiments 7 a to 12, a portion of the water dissolving CO₂ was withdrawn into a small steel vessel (675 c.c.) under working pressure of 20–21 kgs/cm², and, after its pressure was relieved, the dissolved gas (CO₂ as well as the other gases) was allowed to evolve out into a glass bottle. From the volume and the composition of the evolved gas analysed by Hempel's gas analysis method, the dissolved

2) Thomas K. Sherwood : Absorption and Extraction, 1st. Ed., p. 181, (1937).

* The fact of non-uniformity was revealed in the successive experiments on liquid distribution in this packed column (unpublished).

Table 1 — Experimental Data

Exp. No.	5 a	5 b	7 a	7 b	8	11	12
Composition of raw gas		CO ₂ ,	H ₂ ,	N ₂ ,	CO, and CH ₄		
CO ₂ in raw gas (Vol %)	22.4	22.4	19.8	19.8	15.1	26.4	14.2
Solute-free gas in raw gas (Vol %)	77.6	77.6	80.2	80.2	84.9	73.6	85.8
H ₂ in solute-free gas (Vol %)	79.1	79.1	92.7	92.7	94.2	88.5	88.9
Raw gas (m ³ /hr)	30.0	47.4	48.6	85.0	67.5	46.0	62.9
Feed water (m ³ /hr)	2.71	4.07	3.93	3.93	4.02	3.75	6.83
Temperature of water (°C)	23	23	17	18	17	21	18
Working pressure (absolute kgs/cm ²)	20.5	20.5	21.1	21.1	21.0	21.0	21.5
Residual CO ₂ in washed gas (Vol %)	1.3	1.1	0.8	6.0	1.8	1.4	0.4
$K_{1a} \frac{m^3 - CO_2}{(hr)(m^3) \left(\frac{m^3 - CO_2}{m^3} \right)}$	25.8	45.6	34.9	38.4	47.7	36.6	36.8
Loss of solute-free gas (m ³ - gas) / (m ³ - water) (Vol %)	—	—	0.60	0.44	0.49	0.46	0.62
	—	—	6.0	2.5	3.5	5.0	7.8

Remarks : gas volume is calculated at 0°C and 760 mm Hg.

quantity of the so-called solute-free gas was determined. The last two lines in Table 1 show these figures. In operation of these packed absorption towers where the residual CO₂ content in washed gas is to be minimized under 1%, the loss of the CO₂-free gas (hydrogen) would exceed 5% at least. That loss of hydrogen should be taken into account in the CO₂ absorption process of industrial manufacture of hydrogen.

The work described above* was carried out in the Fuel Research Institute. The author wishes to express his thanks to the Director, Dr. M. Kurokawa for his guidance to the investigation project and permission to publish.

(to be continued)

* Read in part at the 44th Annual Meeting of the Society of Chemical Industry, Japan in 1941.