

CALCIMETER CALIBRATION TO DETERMINE CALCITE CONTENT IN CALCAREOUS SANDS

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INTRODUCTION

Calcareous sand deposits are abundant along the eastern coastline of Sri Lanka. These form in a dynamic marine environment with carbonate rich compounds. Calcareous sands have fragile grain structures due to physical and chemical activities continuing after deposition. Such changes cause their engineering properties to change with time; hence carbonate content is an important parameter that governs engineering designs.

Chemical precipitates present in calcareous sands are quantified in terms of Calcite Equivalent which is the equivalent CaCO₃ mass. ASTM D4373 (2002) discusses the use of a Calcimeter to determine Calcite Equivalent of soils.

The chemical equation $\text{CaCO}_3 + 2\text{HCl} \rightarrow \text{CaCl}_2 + \text{H}_2\text{O} + \text{CO}_2\uparrow$ shows that 0.01 mole of CaCO₃ (i.e. 1.001g) reacts with 20 mL of 1M HCl solution (i.e. 0.02 moles) to give 0.01 moles of CO₂ gas. ASTM D4373-02 recommends a calcimeter with a constant volume of 374 ml. It also provides a calibration curve to correlate measured partial pressure of CO₂ to Calcite Equivalent. It also recommends to use an HCl solution of 20 ± 2 ml. This is an important consideration since solubility of CO₂ gas in HCl solution increases with solution volume. The calibration curve proposed in ASTM D4373-02 is valid only for the standard calcimeter described.

The calcimeter described in this study uses a mercury manometer to measure partial pressures and accounts for volume change due to generated CO₂. The study proposes a correction to account for dissolved CO₂ gas when a standard HCl solution volume is not used.

METHODOLOGY

Fig. 1 shows the experimental set up used to measure pressure of CO₂ generated, ΔP , the changes in volume and temperature during the chemical reaction. The combined gas law computes the partial pressure, ΔP_0 at constant volume V_0 and a temperature of 20 °C. The number of moles of carbon dioxide released, n is computed using the Ideal Gas Law.

Fig. 2 shows manometer readings that computes the change in partial pressure head, ΔP . This is expressed as $\Delta h = 0.5(h_{1R} - h_{0R} + h_{0L} - h_{1L})$. Fig. 3 shows typical measurements of ΔP and Temperature, T made with time. The stable values are then used to compute n . Reagent grade Calcium Carbonate specimens of mass 0.2 to 1g were prepared as per ASTM D4373-02 and were made to react with 1M HCl solution with volumes 5, 10, 20, 30 and 40 ml. Mass of CaCO₃ computed based on number of CO₂ moles produced was compared with the mass of CaCO₃ used.

RESULTS AND DISCUSSION

Table I lists the findings of this study. Column 8 computes the correction applicable to the mass of CaCO₃ measured based the number of moles of CO₂ generated. This is expressed as

$$\varepsilon = \frac{\text{Column 1} - \text{Column 7}}{\text{Column 1}}$$

It is evident that the error in measured partial pressure of CO₂ increases with the volume of HCl solution used (Fig. 4). The said variation could be

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attributed to CO₂ gas being dissolved in HCl solution.

Fig. 4 shows that the correction increases as a power function, for the range of HCl volumes used in this study. Fig. 5 compares mass of CaCO₃ used in this study (Column 1) versus mass of CaCO₃ computed based on the number of CO₂ moles produced. The two curves show the observed variations without correction (Column 7) and with correction (Column 9).

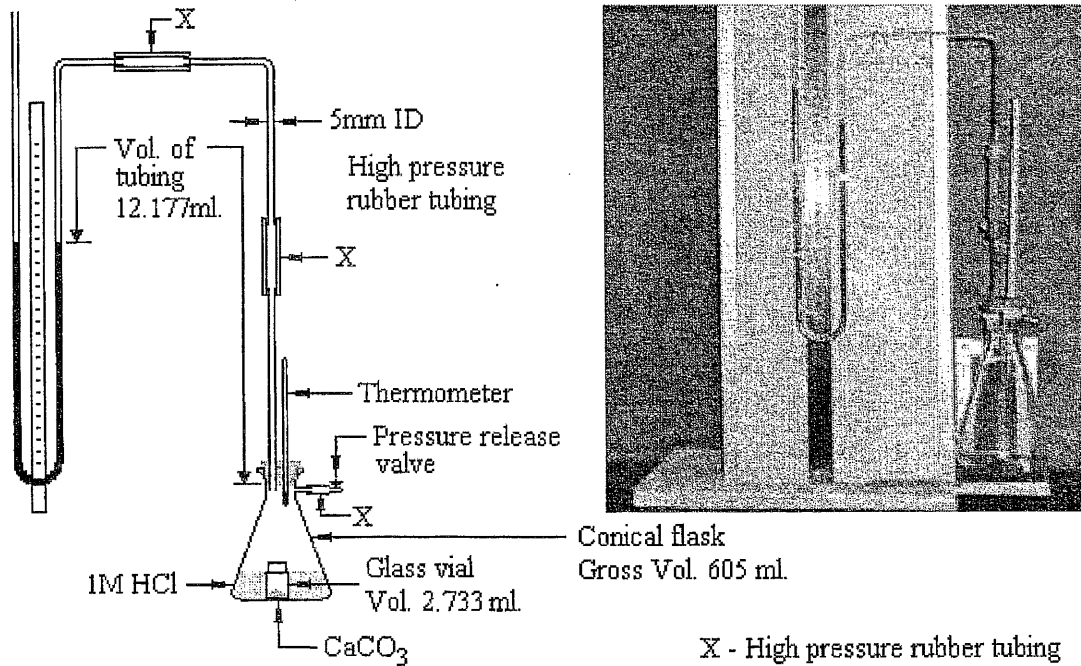


Fig. 1: Calcimeter apparatus

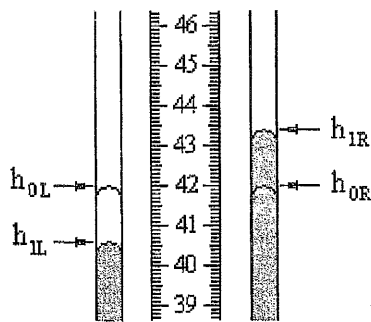


Fig. 2: Mercury manometer.

CONCLUSIONS/RECOMMENDATIONS

The proposed method is an inexpensive and rapid method to determine calcite equivalent for calcareous sands and calcareous matter present in calcareous sands. The correction for dissolved CO₂ gas, as a function of HCl volume is important when specimens with higher calcite content are measured.

REFERENCES

ASTM D4373 (2002), Standard Test Method for Rapid Determination of Carbonate Content of Soils, November 2002.

ACKNOWLEDGEMENTS

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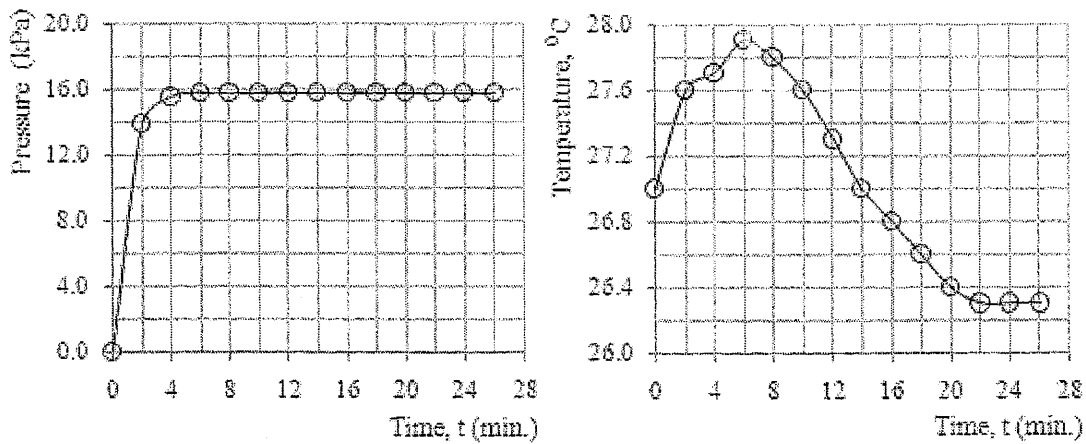


Fig. 3: Variation of (a) pressure and (b) temperature with time for 0.4058g of CaCO₃.

[1] CaCO ₃ (g)	[2] HCl (ml.)	[3] T (°C)	[4] ΔP (kPa)	[5] ΔP ₀ (kPa)	[6] n-CO ₂ (moles)	[7] CaCO ₃ (g)	[8] ε (%)	[9] CaCO ₃ (g)
0.2073	5.0	26.3	8.0	8.0	0.00197	0.1970	4.9	0.2065
0.2025	10.0	25.8	7.7	7.7	0.00188	0.1880	7.1	0.2018
0.4016	10.0	26.5	15.6	15.6	0.00378	0.3781	5.8	0.4060
0.2135	20.0	27.5	8.3	8.1	0.00192	0.1922	9.9	0.2142
0.4009	20.0	27.1	15.5	15.1	0.00361	0.3612	9.8	0.4026
0.6014	20.0	28.0	23.2	22.7	0.00540	0.5395	10.2	0.6014
0.8013	20.0	28.2	30.4	29.7	0.00707	0.7073	11.7	0.7885
0.2033	30.0	25.9	7.7	7.5	0.00176	0.1756	13.5	0.2020
0.4058	30.0	26.3	15.7	15.2	0.00357	0.3571	11.9	0.4107
0.6010	30.0	25.6	22.9	22.2	0.00524	0.5239	12.8	0.6025
0.8027	30.0	25.0	30.4	29.5	0.00699	0.6986	12.9	0.8034
1.0017	30.0	26.9	38.4	37.1	0.00873	0.8730	12.8	1.0040
0.2016	40.0	26.0	7.6	7.2	0.00167	0.1666	17.3	0.1971
0.4013	40.0	26.5	15.2	14.4	0.00333	0.3328	17.0	0.3936
0.6033	40.0	25.3	22.7	21.6	0.00501	0.5012	16.8	0.5928
0.8033	40.0	25.4	31.6	30.1	0.007	0.6999	12.8	0.8278
1.0049	40.0	25.0	38.4	36.7	0.00854	0.8543	14.9	1.0104

Table I: Observations and Results.

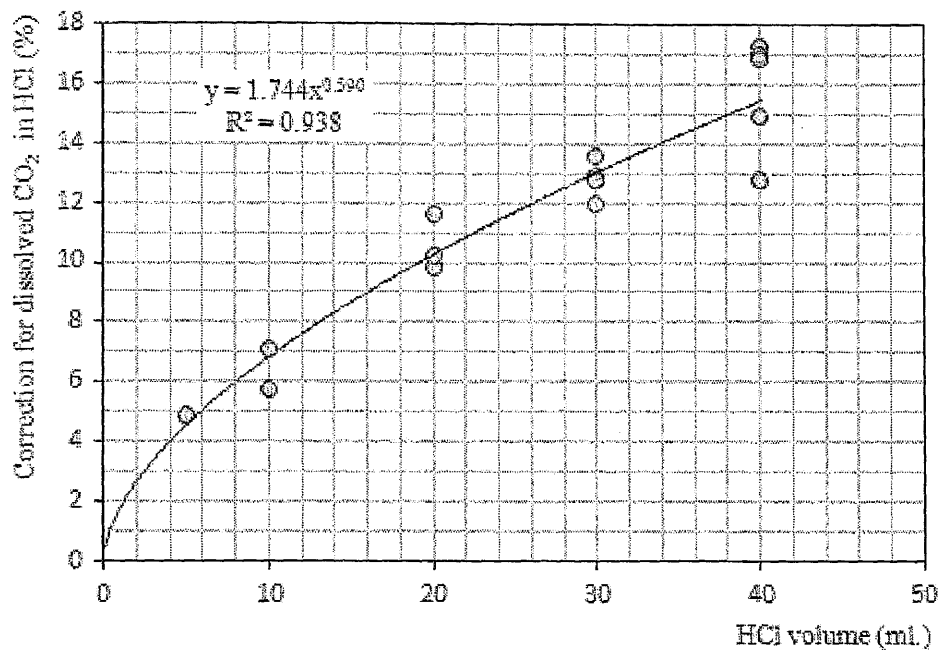


Fig. 4: Variation of Correction Factor versus HCl volume.

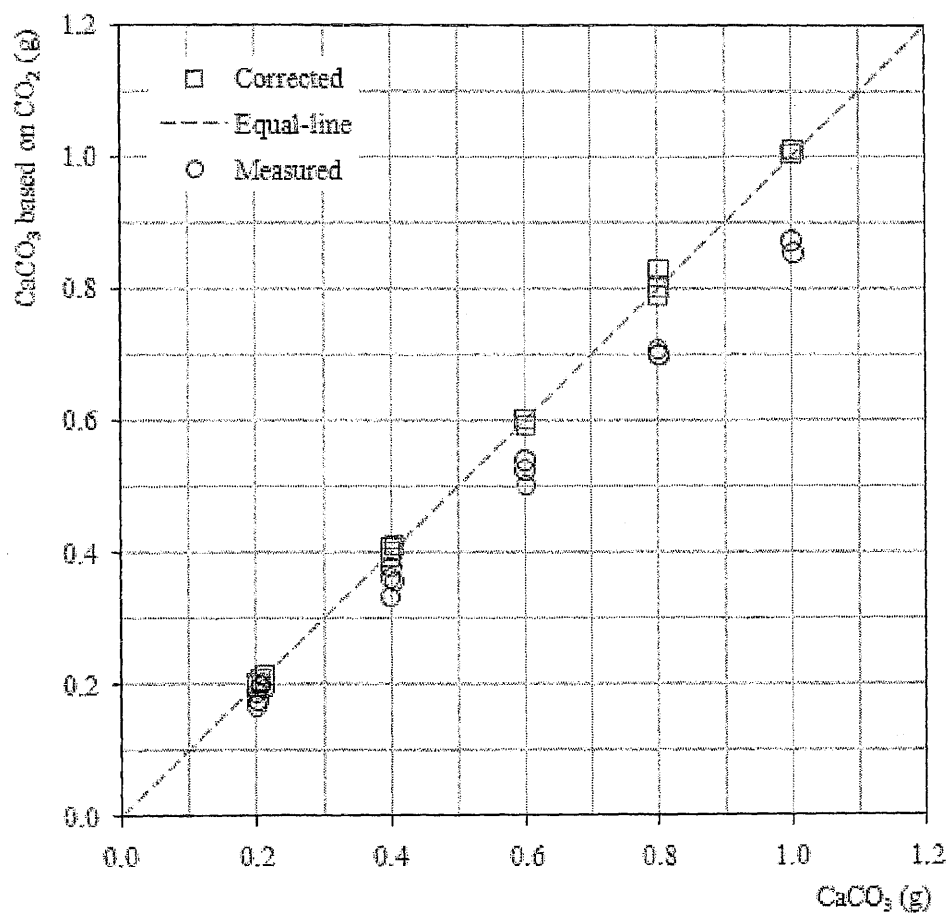


Fig. 5: Variation of estimated CaCO₃ versus CaCO₃ used.