EXPERIMENT No. 7
Study of a non-Catalytic Gas-Solid Reaction by Thermogravimetric Technique

Objective:
To determine the reaction rate constant for the decomposition of cylindrical sodium bi carbonate pellet and find out the activation energy in a temperature range of 100 to 250^{O}C. The stoichiometric equation:

$$2\mathrm{NaHCO_3} = \mathrm{Na_2CO_3} + \mathrm{CO_2} + \mathrm{H_2O} \qquad \text{---- (1)}$$

Theoretical background:
For the above reaction the un reacted core model which has found to satisfy the most of the non catalytic gas-solid reaction is applied. In case of bicarbonate decomposition, chemical resistance at the interface, diffusional resistance of the boundary layer and the product layer may individually or simultaneously control the reaction rate.
The previous studies have shown that the effect of sweeping gas flow rate (air) is negligible on the conversion at different temperatures. So at low conversion (thin product layer) it may be concluded that the two diffusional resistances, e.g. gas film diffusion and ash layer diffusion are absent and the reaction rate is controlled by the chemical resistance. Lievenspiel's equation may be used to convert the weight-loss at any instance into the weight-loss per unit area of interface by introducing a dimensionless factor f which represents the fractional thickness of the reacted solid at any instance. When f attains the value of the ration of length to diameter (a) of the cylindrical reactant pellet, the reaction is complete.
The rate equation may be written as:

$$R_0 d_0 f = kt \qquad \text{--- (2)}$$

Where,

R_0	= Initial radius of the pellet, cm
d_0	= Density of the reactant pellet, gm/cc
t	= Reaction time
k	= Modified rate constant

The spherical pellet and the cylindrical pellet with same length and diameter, the relation between conversion *x* and *f* is

$$f = 1 - (1 - x)^{1/3} \qquad \text{--- (3)}$$

For cylindrical pellet with any ratio of length/diameter, reaction conversion is given by:

$$x = 1 - \frac{(a-f)(1-f)^2}{a} \qquad \text{--- (4)}$$

So for any value of *x* we can get *f* from the above relation and calculate $R_0 d_0 f$ for each time *t*. Under the isothermal condition $R_0 d_0 f$ vs *t* plot will give straight line for chemical resistance control. At different temperatures, we are expected to get different $R_0 d_0 f$ vs *t* straight lines from the slipe w of which we can find out the activation energy by plotting *ln*k vs 1/T.

Apparatus:

A thermogravimetric set up used in the present study consisting of the following:

A) An analytical chainometric (keroy) balance from the left pan of which is suspended a Pt wire carrying sample holder, made of 200 mesh SS Screen.

B) A tube furnace consisting of mullite tube of length 45.72 cm and diameter 5.08 cm. The furnace bottom is packed with quartz particle made up to some height for preheating the sweeping gas.

C) A voltage stabilizer and control device are introduced to avoid the fluctuations in voltage and temperature.

Preparation of the solid sample:

The pellets of any length and diameter are made by putting AR Grade sodium bicarbonate powder in a cylindrical metallic die and pressing the sample in a laboratory hydraulic press. The pressure may be varied as we desire.

Experimental Procedure:

(i) The calibration of the thermocouple should be done with pure solid substances having definite melting point.

(ii) Two hours before the actual experiment. The heating of furnace is started. At the same time the sweeping gas flow rate of desired quantity is usually maintained.

(iii) The cylindrical pellet is then placed into the basket and lowered into the reactor (just above the thermocouple sheath).

(iv) From loss of the pellet, the conversion x is calculated

$$x = \Delta W / \sum \Delta W$$

Where ΔW = total weight loss = W(0) – W(t)

(v) The temperature recorded by the thermocouple near the basket is assumed to be the pellet temperature. Alternatively the reaction can be carried out in non-isothermal condition within the range when the temperature is linear with time.