BET (Brunauer-Emmett-Teller) Method for Surface Area Determination

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BET isotherms empirically fits adsorption isotherm data

- BET method - procedure for determining surface area of finely divided solid by measurements of low-temperature gas adsorption
- BET isotherm

$$\frac{V}{V_m} = \frac{C(p/p_0)}{(1-p/p_0)[1+(C-1)(p/p_0)]}$$

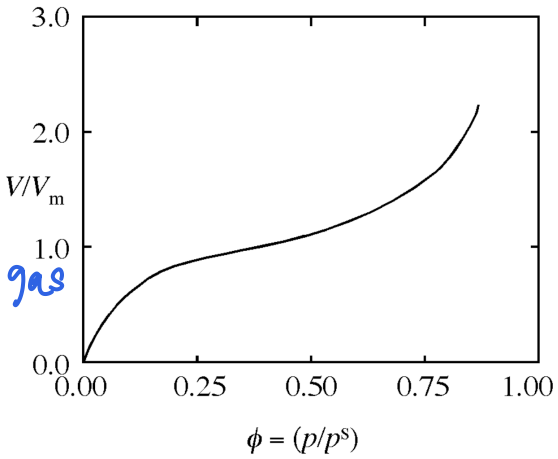
- Linear form of BET isotherm

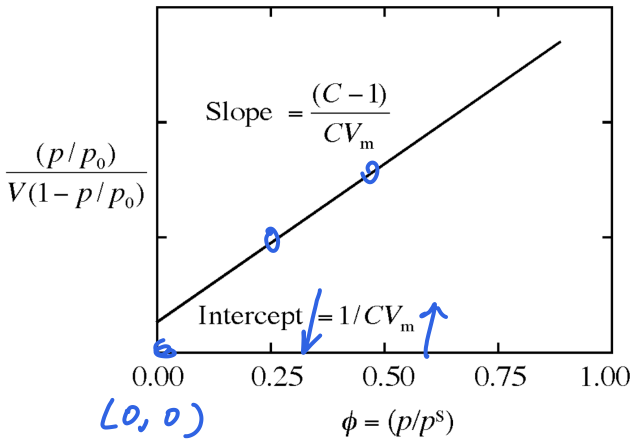
$$\frac{(p/p_0)}{V(1-p/p_0)} = \frac{1}{CV_m} + \frac{(C-1)(p/p_0)}{CV_m}$$

- Single point method: $C \gg 1$, so intercept ≈ 0

  Volume: $V_m = V(1-p/p_0)$

  Mass: $X_m = X(1-p/p_0)$





BET method allows surface area determination

- Volume of adsorbate desorbed by sample

$$V_{\text{des}} = V_c \frac{A}{A_c}$$

calibration ← A: sample integrator; A_c: calibration integrator } measurable

- Mass of adsorbate desorbed

$$X = \frac{p_a V_{\text{des}}}{RT} M_a = \frac{p_a V_c (A/A_c)}{RT} M_a$$

ideal gas law

- Mass of adsorbate desorbed when solid is covered by a single adsorbate monolayer

$$X_m = \frac{p_a V_c (A/A_c)}{RT} M_a (1 - p/p_0)$$

$X_m = X(1 - p/p_0)$

- Total surface area

measured

$$S_t = X_m \frac{N_0}{M_a} A_{cs}$$

N_0 ← Avg #; M_a: # molecules; A_{cs}: cross-sec area of molecule

- Specific surface area

$$\Sigma = \frac{S_t}{m}$$

m ← mass of sample

Initial configuration of FlowSorb Surface Area Analyzer

- Initial configuration
  - Power: OFF
  - Toggle valve: open
  - Sample holders are installed at DEGAS, TEST, and COLD TRAP locations
  - Insulating container is installed at sample holder for COLD TRAP

Startup procedure of FlowSorb Surface Area Analyzer

- Startup
  - Open inert gas flow (30% nitrogen, 70% helium), wait 5 min
  - Adjust flow meter to the calibration mark
  - Pour liquid nitrogen into an insulating container, and pour the liquid nitrogen to the insulating container at COLD TRAP, wait for 10 min
  - Power switch: ON, wait for 30 min

Preparing activated carbon sample for BET measurement

- Sample preparation
  - Weigh 0.02 g activated carbon in the sample holder
  - Place the sample holder on DEGAS and heat with a heating mantle, wait for 15 min (concurrent with start up)

Calibrating FlowSorb Surface Area Analyzer

- Calibration
  - Flush gas syringe with evaporated nitrogen gas above the liquid nitrogen at DEGAS
  - Fill the gas syringe with 1 mL nitrogen gas
  - Wipe needle tip free of frost, set aside needle to equilibrate to room temperature
  - Zero the instrument display with course and fine zeros. Switch to surface area (SA), and clear SA display
  - Insert needle at INJECT, inject nitrogen gas at moderate rate, and withdraw
  - Wait until reading is stabilized, then calibrate the instrument to **SA = 2.84**
    - Confirm calibration by repeated injection, if necessary

Measuring surface area of activated carbon with the BET method (adsorption)

- Adsorption measurement
  - Exchange sample holders between DEGAS and TEST, so the sample is at TEST
  - Wait until readings stabilize and clear SA display
  - Pour liquid nitrogen into an insulating container
  - Place the insulating container at TEST by immersing the sample holder with liquid nitrogen. Secure the container by flipping on the container holder
    - Nitrogen gas starts to adsorb to activated carbon
  - Wait until reading is stabilized, **record the value of SA** = adsorption surface area

Measuring surface area of activated carbon with the BET method (desorption)

- Desorption measurement
  - Remove the insulating container by putting down the container holder
    - Nitrogen gas starts to desorb form activated carbon
  - Wait until reading is stabilized, **record the value of SA** = desorption surface area

Shutdown procedure of FlowSorb Surface Area Analyzer

- Shutdown
  - Power: OFF
  - Remove the activated carbon from the sample holder, and place the sample holder back on TEST
  - Turn off the inert gas flow

Calculating the specific surface area of activated carbon

- Measured

  S_t: Adsorption SA, desorption SA

  m: Mass of activated carbon

- Calculated
  - $\boxed{\Sigma = \frac{S_t}{m}}$ - specific SA for adsorption and desorption

    Usually use desorption data to report Σ