B-T-014

1-point BET measurement for carbon black by BELCAT

Objective.

The BET specific surface area of carbon black (M11-02) is measured using BELCAT. (Flow method)

experiment

Equipment	: BELCAT
Detectors	: TCD (semi-diffusion type 4 elements)
Sample used	: CB (M11-02)
Sample weight	: 0.1930 g
Gas used	: 30% N2/He (He dilution)
Calibration tube volume (S.T.P.)	: 1.565 ml
Gas used for measurement	: 30% N2/He (He dilution)
Pretreatment conditions	: 305°C, 2h carrier gas distribution

Pretreatment

1. Place M11-02 into the sample tube and connect the sample tube to the connector port on the heater side.
2. The heater is raised to 305°C while 30% N2/He is circulated and held for 2 hours, then lowered to room temperature.

Adsorption and desorption measurement

3. Connect the sample tube to the BET measurement port.
4. Circulate 30% N2/He in the sample section to stabilize the TCD baseline.
5. Immerse the sample section in liquid nitrogen while 30% N2/He is circulating.
6. When the peaks have finished appearing and the baseline has stabilized, remove the liquid nitrogen from the sample section and wait for the baseline to stabilize again.

Caliblation

7. Connect 100% nitrogen gas to the Pulse gas port.
8. Stabilize the baseline of TCD by circulating 30% N2/He in an empty sample tube.
9. Open AV1 and purge the calibration tube with nitrogen gas.
10. Close AV1 and refrain from setting the temperature and pressure in the calibration tube.
11. The nitrogen gas in the calibration tube is pumped into the sample tube, and after the peak appears, the baseline of TCD is set at a safe level.
    Wait for it to settle.

Adsorption (desorption) amount calculation formula

$$\text{caliblation Factor} = \frac{ml(STP)}{mV \times \sec}$$

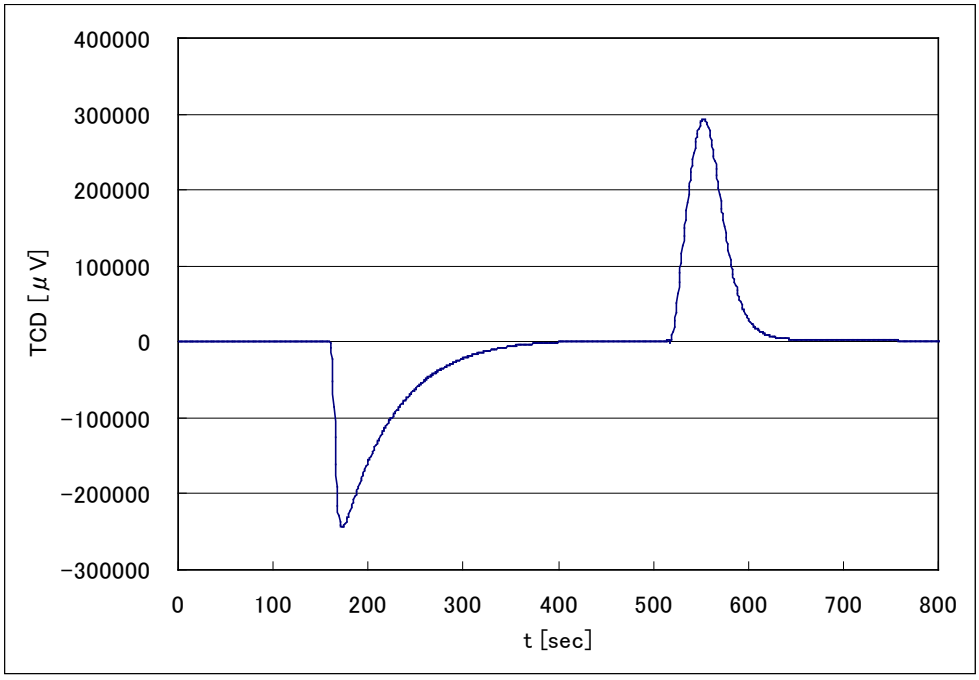
$$\text{Adsorption amount(ml/g)} = \frac{\text{Peak area x CF}}{\text{Sample weight}}$$

Monolayer $: Vm(ml((S.T.P.)/g) = V(1 - P/P_0)$

Specific surface area(S_{BET}) $= \frac{Vm\ x\ 6.022E{+}23\ x\ Adsorption\ cross\ section\ x\ 10E{-}18}{22414}$

Result

Figure 1 shows the TCD output behavior by N2 adsorption/desorption, and Table 1 shows the calibration values and the values obtained from each adsorption/desorption peak. in the BET single-point method, the desorption peak is generally sharper than the adsorption peak, so the specific surface area is generally obtained using the desorption peak. Therefore, the BET specific surface area of this CB is 72.7 m2 • g-1,



which is consistent with the value obtained by BELSORP.

Figure 1. N2 adsorption/desorption peak

Table 1 Numerical data

Calibration measurement	Sample weight /g	area	Introduction volume / ml	CF	Adsorption volume / ml (S.T.P.)	monolayer adsorption volume / ml (S.T.P.)	Specific surface area / m2 • g-1
Blank		1538206	0.523	3.40E-07			
Adsorption peak	0.1903	15568000			5.29	3.70	84.7
Desorption peak	0.1903	13367750			4.54	3.18	72.7

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