

FRAUNHOFER-INSTITUT FÜR WERKSTOFF- UND STRAHLTECHNIK IWS



RAPID SURFACE AREA ANALYSIS

Task

The assessment of specific surface area in advanced functional materials is a key task for the characterization of porous materials such as activated carbons, porous polymers, metal-organic frameworks, zeolites, nanoparticles and catalysts. However, established techniques (nitrogen physisorption at 77 K) are time consuming and often limit rapid discovery, screening, and process quality assessment. For hightroughput characterization and process control a rapid surface area analysis is desireable.

Solution

The INFRAsorp device measures the total heat of adsorption released upon exposure to *n*-butane towards a porous material. In a dynamic flow,

an optical sensor detects the temperature increase on the surface of the material (Fig. 3a). The temperature decay at the tail of the peak is caused by the constant flow of *n*-butane (101.3 kPa, 298 K, $p/p_0 = 0.42$). The peak area (Fig. 3b) reflects the total heat of adsorption released and hence the amount of gas adsorbed. Calibration using reference materials provides a direct measure of the single point BET surface area equivalent.

Our offer

- surface area determination within 5 minutes
- small sample amounts below 30 mg
- no cooling required
- no liquid nitrogen required
- measuring at 298 K
- small footprint, compact design
- automatization
- portable version upon request

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Application

Porosity and gas storage ability are key targets in the development of porous materials. INFRAsorp is used to identify high surface area materials in a very short time with high accuracy.

As indicated in Fig. 3c, the specific surface areas from INFRAsorp measurements of two activated carbons (AC) and one metal-organic framework (MOF) sample are in very good agreement to the reference specific surface areas (N_2 , 77 K).

It has to be pointed out that the specific surface area is determined ten times faster by using INFRAsorp than commonly used physisorption experiments.

Our offer

INFRAsorp system:

- basis version (dynamic, single point, *n*-butane)
- option for sample activation
- static mode (full isotherm)
- high pressure version
- additional CO₂ option for ultramicropore assessment



Advanced materials evaluation:

- screening of advanced porous materials
- surface area determination
- adsorption kinetics
- filter evaluation
- chemisorption experiments
- adsorption/desorption cycling stability testing
- process optimization: catalyst extrudates, binder pore blocking, residual filter capacity
- different adsorptives: CO₂, NH₃, H₂S, volatile organic compounds (VOCs), e.g. methane, ethanol



c) Specific surface areas which are determined by INFRAsorp and reference N₂ physisorption



Key parameters of the standard measuring setup

Detection: Measuring temperature: Measuring pressure: Gas flow: Adsorptives: Volume of sample holder: Sample pre-treatment optical IR sensor room temperature 0.2 - 6 bar max. 500 ml/min N₂ equivalent *n*-butane (optional: VOCs, CO₂, NH₃, H₂S) ca. 0.1 ml vacuum activation option at high temperatures (up to 473 K) available www.iws.fraunhofer.de/infrasorp

- 1 Different porous materials
- 2 INFRAsorp device