

IMPROVING THE ROBUSTNESS AND REPRODUCIBILITY OF GAS ADSORPTION ISOTHERM MEASUREMENTS ON NANOPOROUS MATERIALS

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INTRODUCTION

Gas adsorption is commonly used to characterize the surface area, pore volume and pore size distribution of porous materials, as well as to assess adsorbents for use in gas storage and separation applications. Adsorption isotherms – plots of uptake versus pressure – can be measured using the volumetric and gravimetric techniques, and commercial instruments are widely available for this purpose. To determine the porous properties of materials, experimental isotherm data must then be analyzed using one of a number of different models.

Despite the widespread availability of commercial instrumentation and the associated data analysis software, published results have been subject to irreproducibility^{1 2 3}. This may be due to differences in the synthesis of different samples of the same material, material instability, measurement error, or inconsistencies in the analysis of experimental data⁴. This poster addresses the last two of these sources of irreproducibility, by describing five key steps that can be followed to make experimental gas adsorption isotherms on nanoporous materials more robust and reproducible⁵.



STEP ONE

MAKE CAREFUL MEASUREMENTS

- ▶ Ensure thorough sample degassing
- ▶ Use high purity gases
- ▶ Achieve equilibrium at every isotherm point
- ▶ Use degassed sample mass for calculations
- ▶ Use a sufficient amount of sample
- ▶ Control temperature carefully
- ▶ Determine He skeletal density with care
- ▶ Convert excess to absolute adsorption with reasonable assumption
- ▶ Use accurate equation of state at high pressures

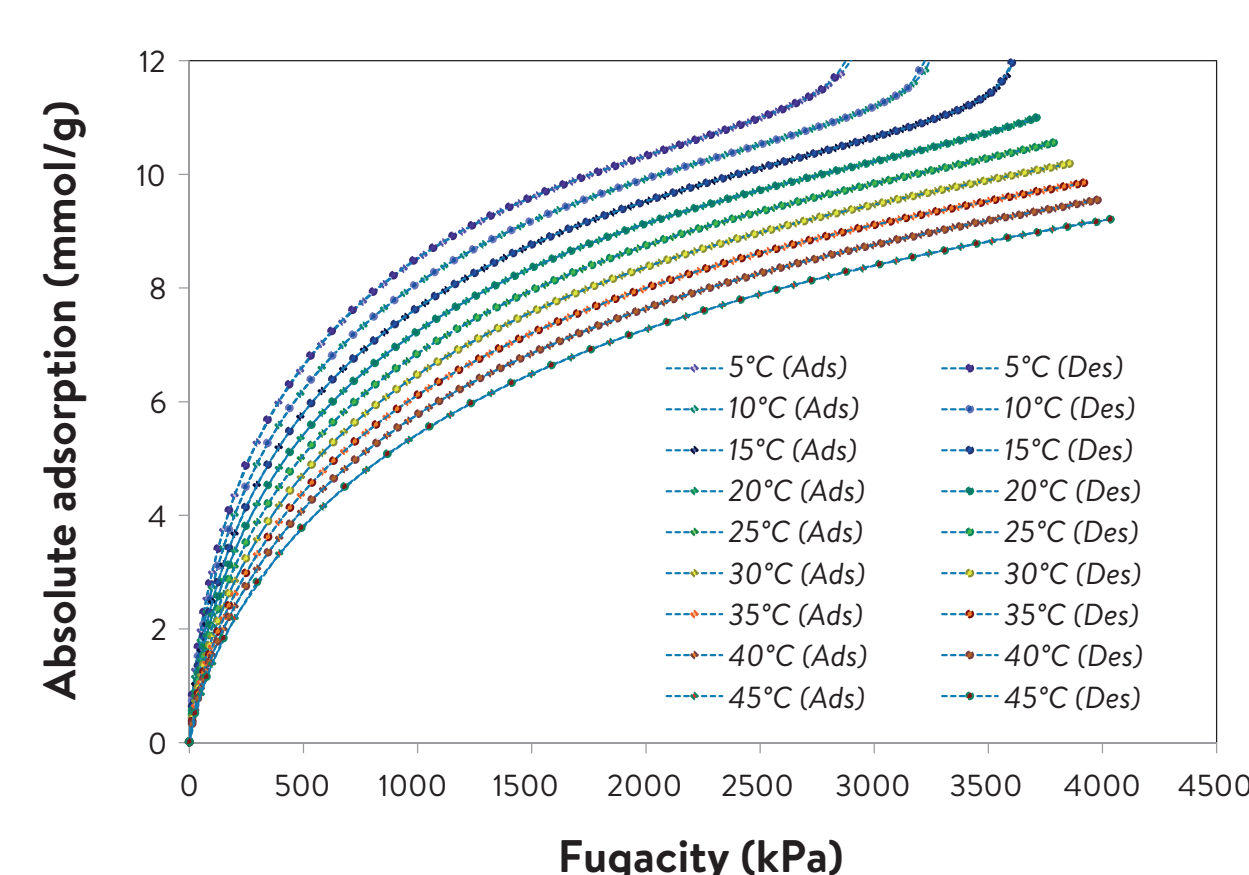


Figure 1: CO₂ adsorption isotherms for F400 porous carbon, measured on a Hidden Isochema XEMIS-001 in the temperature range 5°C to 45°C.

STEP TWO

VALIDATE MEASUREMENT PROTOCOLS AND DATA

Perform validation measurements on certified reference material (CRM)

Perform cross-checks:

- ▶ Prolong degassing and repeat measurement
- ▶ Increase degassing temperature
- ▶ Prolong equilibration time
- ▶ Change sample size and repeat measurement
- ▶ Measure isotherms at different temperatures and calculate ΔH_{iso}
- ▶ Change number of isotherm points

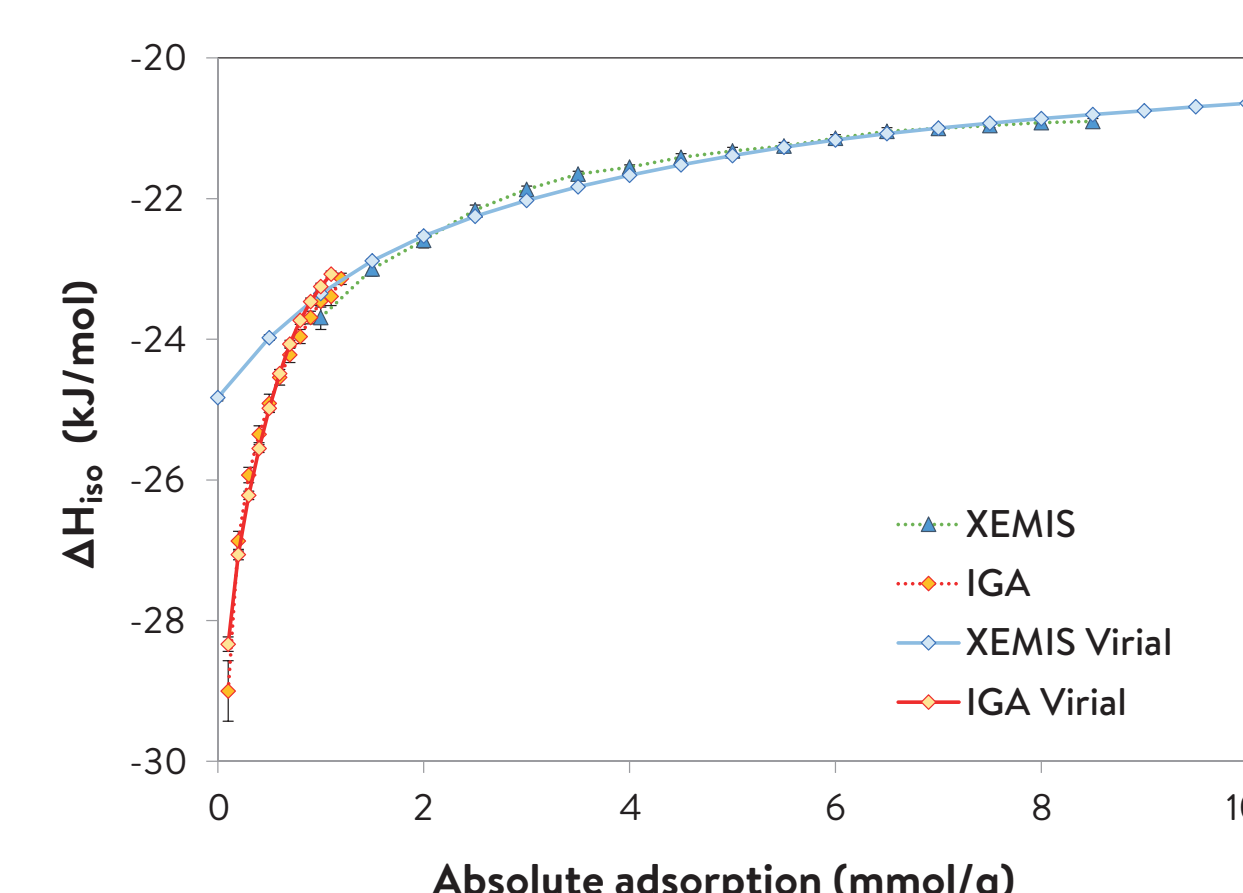
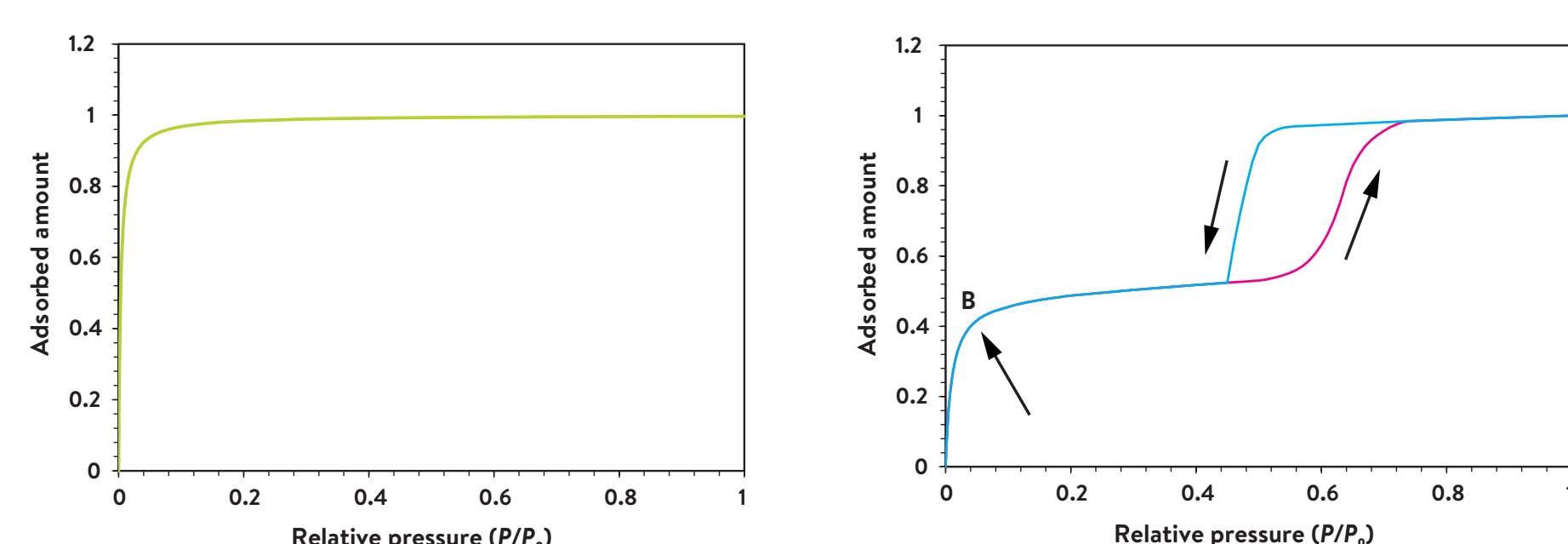


Figure 2: The isosteric enthalpy of adsorption of CO₂ on F400 porous carbon, calculated using polynomial interpolation and fits to the virial equation using both Hidden Isochema IGA and XEMIS-001 data.

STEP THREE

ANALYSE DATA APPROPRIATELY

- ▶ Apply Gurvich rule with care for pore volume estimation
- ▶ Calculate BET area using Rouquerol criteria to select P/P_0 range
- ▶ Select pore size distribution model with care (some models only apply to mesoporous materials)^{1 4}



STEP FOUR

REPORT EXPERIMENTS IN SUFFICIENT DETAIL

Key information:

- ▶ Instrument specifications
- ▶ Gas purity
- ▶ Sample size
- ▶ Sample degassing protocol
- ▶ Temperature control method and measurement accuracy
- ▶ Equilibration times or criteria

Full experimental details must be included in any report, in order to allow other researchers to replicate the results and confirm reproducibility.

STEP FIVE

REPORT RESULTS ACCURATELY AND ACCESSIBLY

- ▶ Use new Adsorption Information File (AIF) to report published data!



SUMMARY

- ▶ Two sources of irreproducibility for gas adsorption data are measurement error and inconsistencies in the analysis of experimental data
- ▶ Making careful gas adsorption measurements requires attention to be paid to several experimental issues
- ▶ Data must be validated by making measurements on CRMs and performing cross-checks
- ▶ Experimental data must be analysed using models appropriate to the nanoporous material
- ▶ Experimental details must be fully reported and results published in an accessible format

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