

MEASURING BINARY GAS ADSORPTION EQUILIBRIA USING THE INTEGRAL MASS BALANCE (IMB) METHOD

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INTRODUCTION

Nanoporous materials, such as zeolites, are widely used to separate gases, in processes such as Pressure Swing Adsorption (PSA) and Temperature Swing Adsorption (TSA).¹ In order to assess the performance of a material for any particular separation, it is necessary to measure multicomponent gas adsorption isotherms. However, measuring multicomponent adsorption is experimentally challenging.^{2,3} Current techniques are often time-consuming and need relatively large samples. The most accurate approaches can require at least a day of experimental time and effort per data point.

We have recently introduced a new technique, called the Integral Mass Balance (IMB) method, which can rapidly and accurately measure a binary gas adsorption isotherm in a matter of only hours.⁴ It combines an open volumetric system with in-situ gravimetric analysis, eliminating the need to measure the total outlet flow rate – the main reason for the inaccuracy of open system measurements. We now describe and demonstrate the use of this new approach, using N₂/O₂ binary adsorption on a commercial zeolite 5A sample.

THE IMB METHOD

In the IMB method, a controlled gas mixture flows over a sample suspended from a microbalance and the gas composition at the outlet is determined using a mass spectrometer (MS). Measuring weight as a function of time, after applying a step change in inlet composition, allows the total amount of adsorption to be determined and provides a measure of the effective total outlet flow rate.

By using a mass balance expression, rather than the usual molar balance, the amount of each component adsorbed by the sample can then be calculated from integration of the difference in inlet and outlet flow compositions, as a function of time, and the differential weight change at each outlet composition. Steady state conditions before and after an inlet composition change can be confirmed by monitoring the microbalance and MS signals until they are constant. Full details were provided in a recent paper.⁴

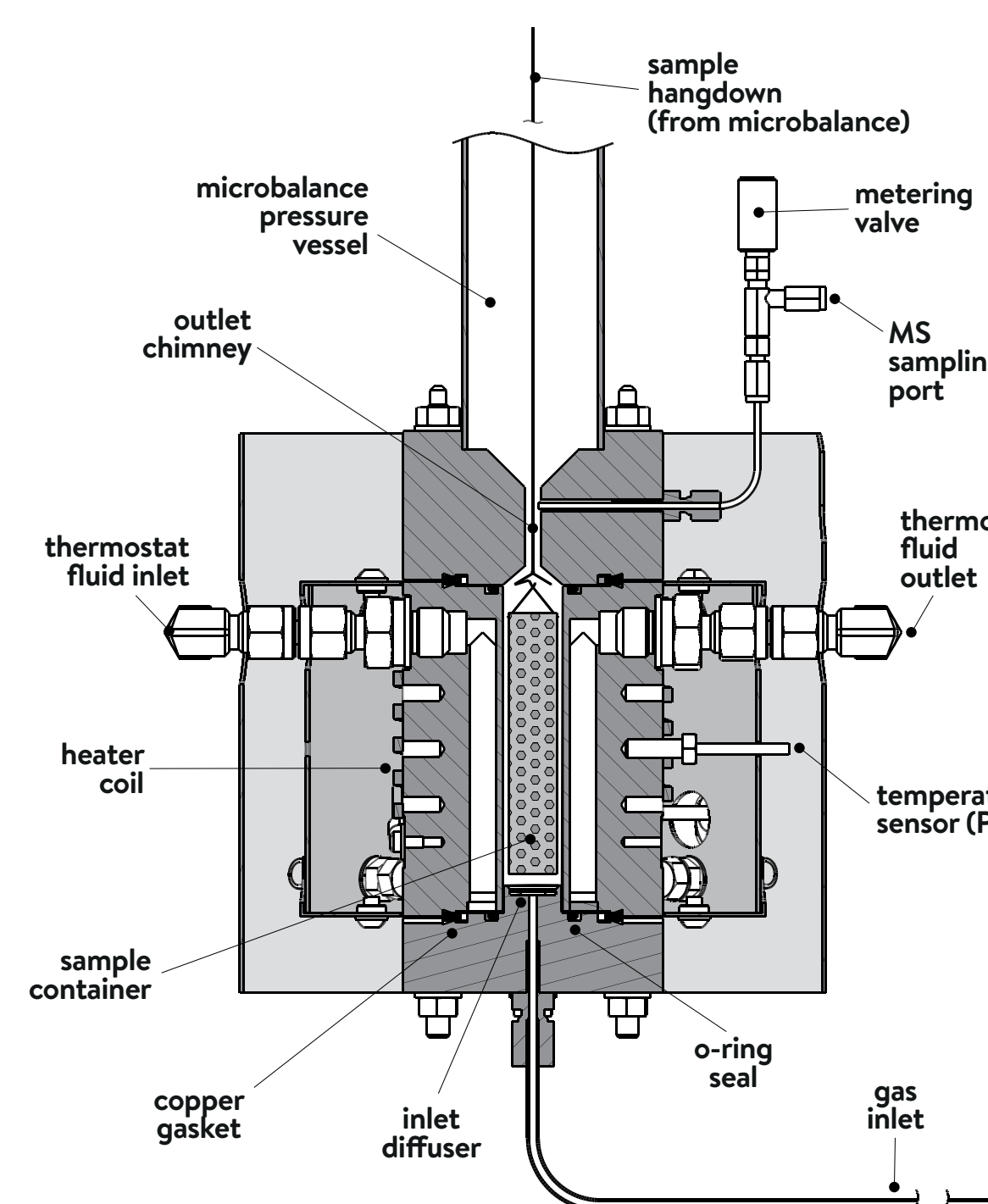
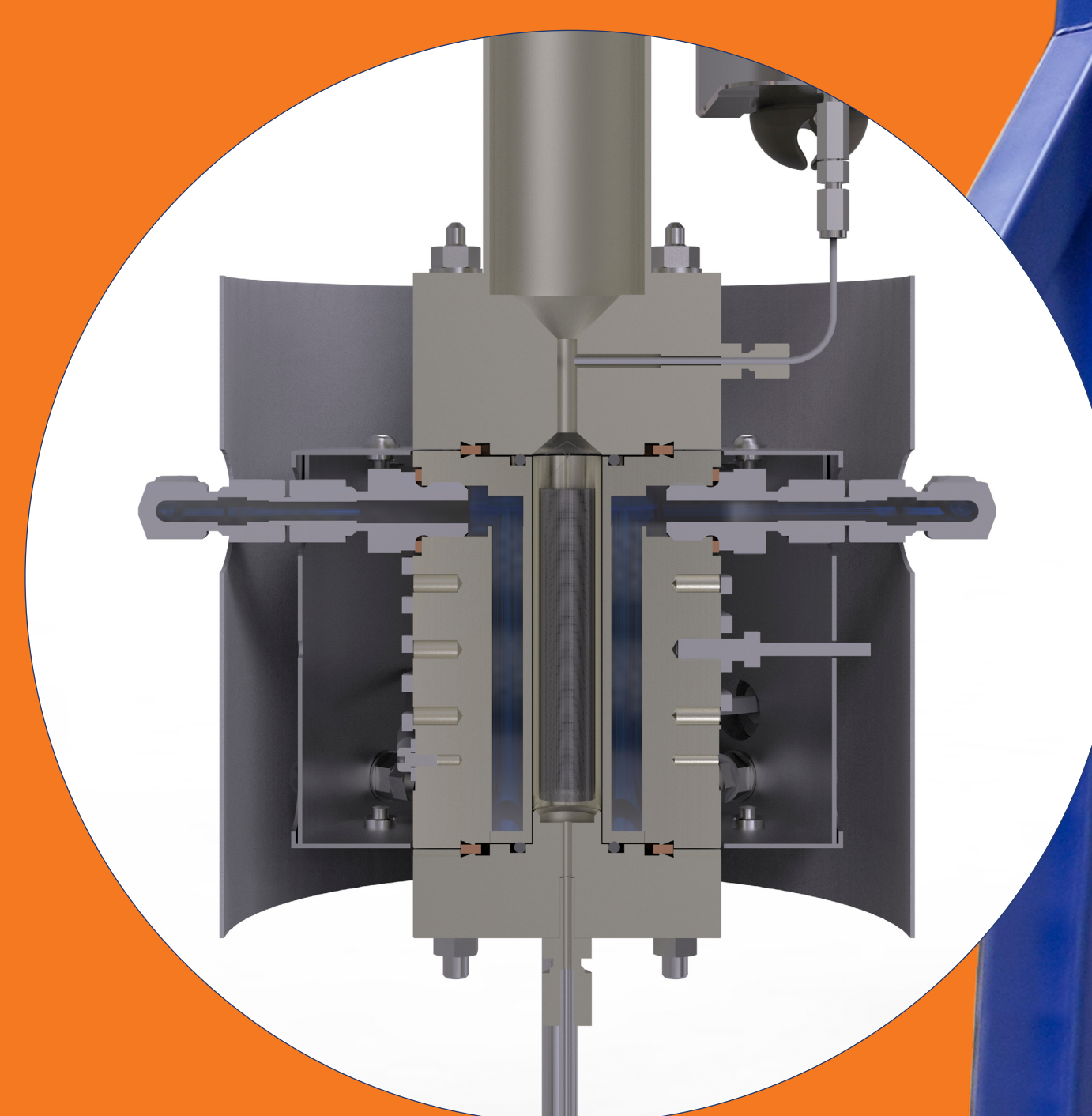


Figure 1: Specially designed IMB method reactor for the Hidden Isochema IGA-003-MC, with key components labeled (MS, mass spectrometer; PRT, platinum resistance thermometer).

EXPERIMENTAL DETAILS

The IMB method is demonstrated using a Hidden Isochema IGA-003-MC gas sorption analyzer, equipped with a specially designed reactor, shown in Figure 1. Outlet composition was determined using a Hidden Analytical Dynamic Sampling Mass Spectrometer (DSMS).

N₂/O₂ adsorption by a Tosoh 5A zeolite sample was measured by changing the inlet N₂/O₂ composition, using MFCs, from 100% N₂ to 100% O₂ in 5% steps, at a total pressure of 915 kPa (9.15 bar). These conditions matched those of previously published data,⁵ and were chosen for validation purposes. Approximately 3.5 g of sample was used, and the entire binary isotherm measurement took approximately four hours. Nine further repeat measurements were made, to demonstrate repeatability.



RESULTS AND DISCUSSION

Figure 2 shows the ten binary N₂/O₂ adsorption isotherms measured on the Tosoh 5A zeolite sample in this study. Excellent repeatability was observed. Also shown, as open symbols, are the original data measured by Talu et al,⁵ which were measured using two different volumetric systems, at Cleveland State University and Air Products and Chemicals, Inc., in Allentown, PA, USA. It can be seen that good agreement between the results of the current study and the original data was achieved.

Figure 3 shows the N₂ selectivities, as a function of O₂ concentration, calculated from the data presented in Figure 2. The results from the present study are compared to the original experimental data of Talu et al,⁵ calculated uncertainty bounds from that study, and Ideal Adsorbed Solution Theory (IAST) calculations performed by Talu et al.⁵ It can be seen that the calculated selectivities from the present study generally lie between the original results of Talu et al and the IAST calculations.

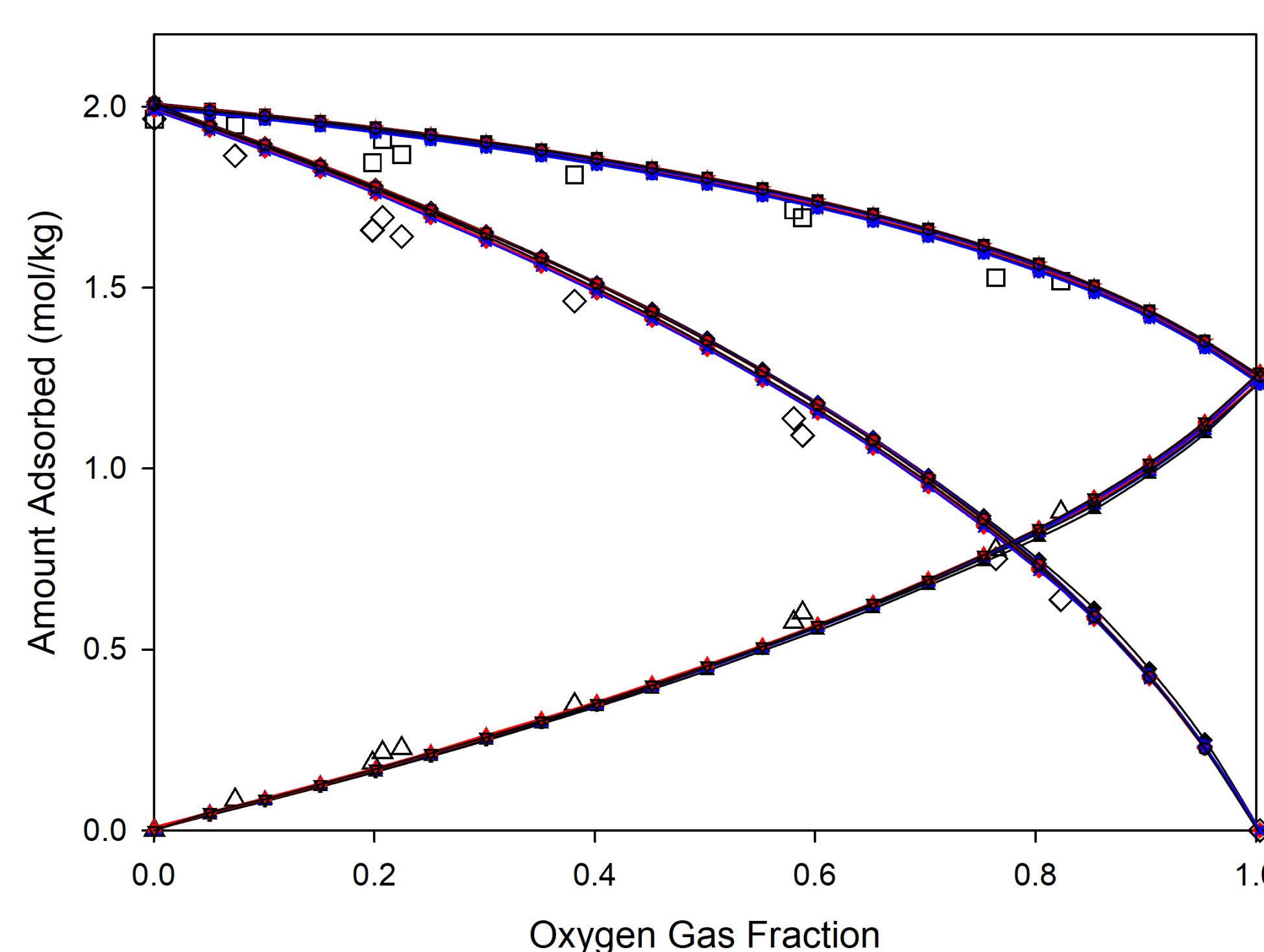


Figure 2: Binary N₂/O₂ adsorption isotherms for zeolite 5A measured on an IGA-003-MC

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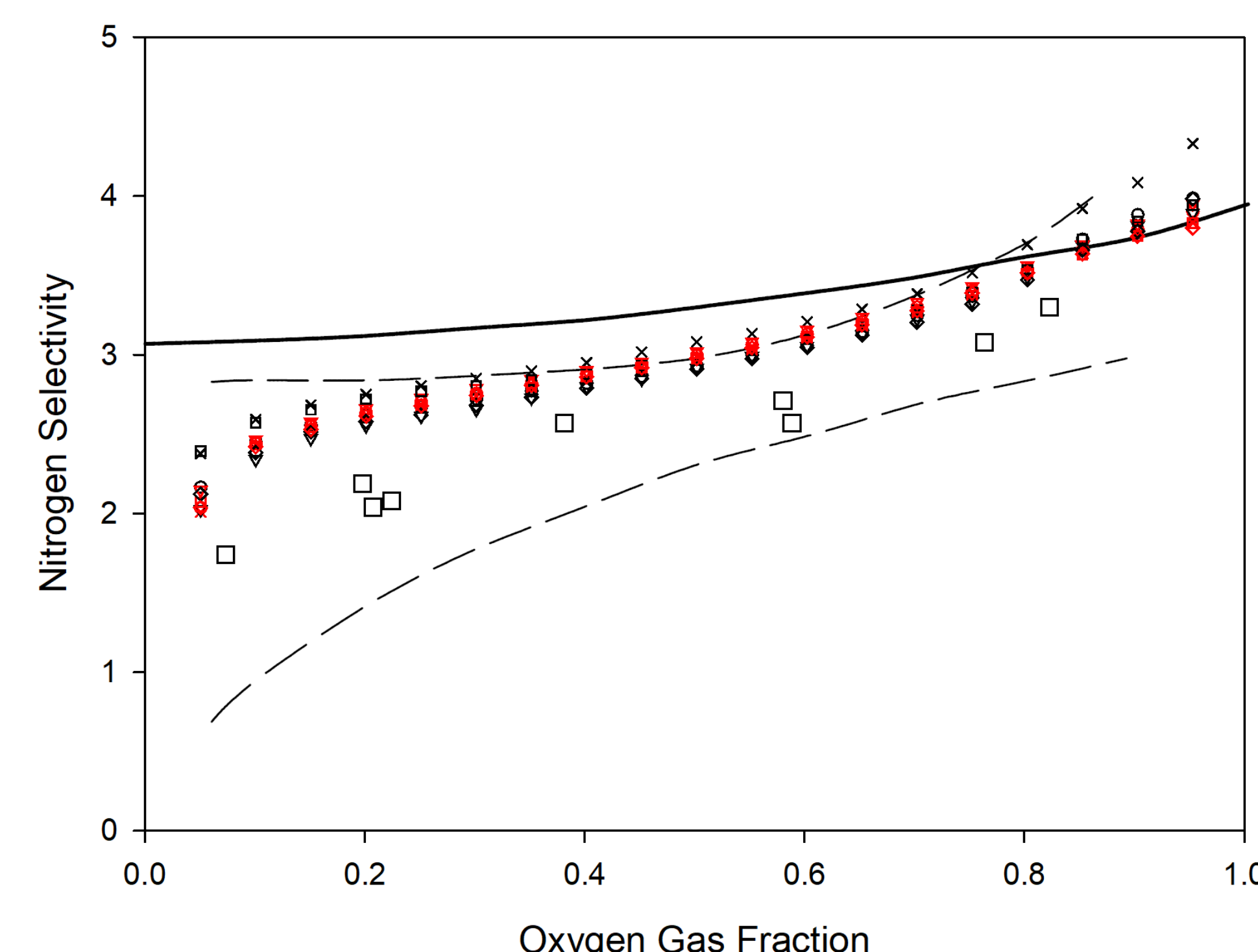


Figure 3: N₂/O₂ selectivities for zeolite 5A calculated from data measured on an IGA-003-MC

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SUMMARY

► Characterizing nanoporous materials for gas separations requires the measurement of multicomponent gas adsorption.

► The IMB method is fast, accurate, and can be performed using only a few grams of sample.

► N₂/O₂ adsorption by a Tosoh 5A zeolite sample has been used to demonstrate and validate the method.

► This approach can readily be extended to measure adsorption from ternary and higher mixtures.

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