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The BET Method for Microporous Materials

Applying the Rouquerol criteria for validating the BET surface area of a type I isotherm.

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Introduction

The Brunauer-Emmett-Teller (BET) method is the most common method for the evaluation of specific surface areas. It is applicable to sorption isotherms Type II and Type IV and the evaluation is carried out in the relative pressure range P/P_0 from 0.05 to 0.30 as recommended by IUPAC.[1] This range is used because, to oversimplify, it is around these pressures that the completion of the monolayer occurs. However, the presence of micropores can interfere with the isotherm in the same region and cause reported values to not represent the true surface area of a material.

Therefore, Rouquerol *et al.* expanded the BET method for Type I isotherms by developing the so-called Rouquerol-criteria. In-part, these criteria state that the BET constant 'C' must be positive, that the selected P/P_0 range must be limited to where the term $n(1 - P/P_0)$ continually increases with P/P_0, and the P/P_0 value corresponding to the monolayer capacity should fall within the selected range.

Applying the Rouquerol Criteria

In order to apply these criteria with ease, the 'Rouquerol-Plot' is utilized. Figure 1. The maximum of this plot (blue-marked measurement point) provides a very easy-to-recognize upper limit for the acceptable P/P_0 range for use in determining BET surface area. Relative pressures past this point should not be used. Usually activated carbon and zeolite with micropores deliver Type I adsorption isotherms. In the following Fig. 1 the nitrogen sorption measurement (top left) of activated carbon is shown. The data is then used to produce a Rouquerol Plot, where a upper limit can be easily identified (right). Finally, the corresponding BET plot is presented and shows the upper limit for the respective material (bottom left).

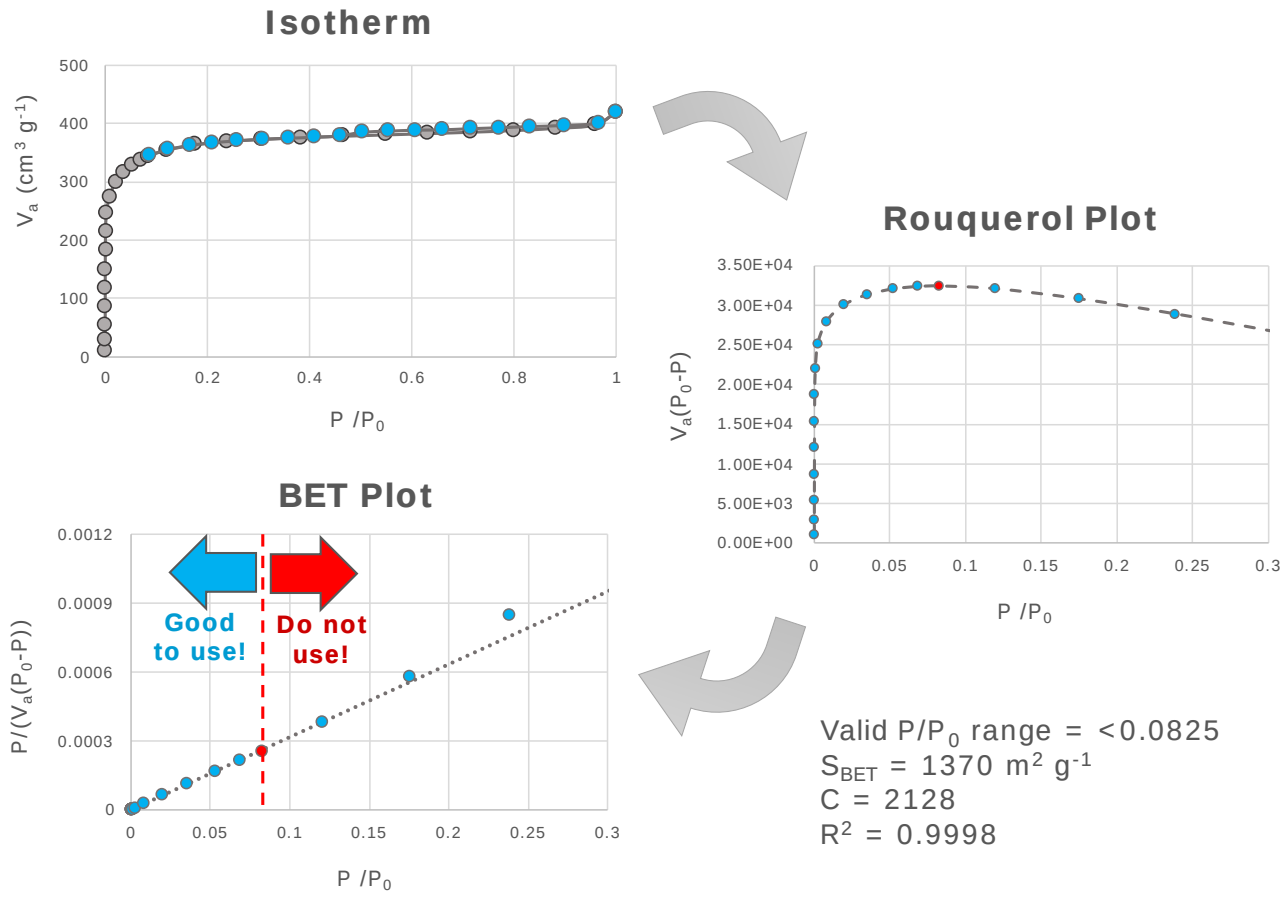


Fig. 1 – Rouquerol-plot (left) and BET-plot of activated carbon in relative pressure range according to Rouquerol *et al.* (right).

Gas Adsorption Analyzers

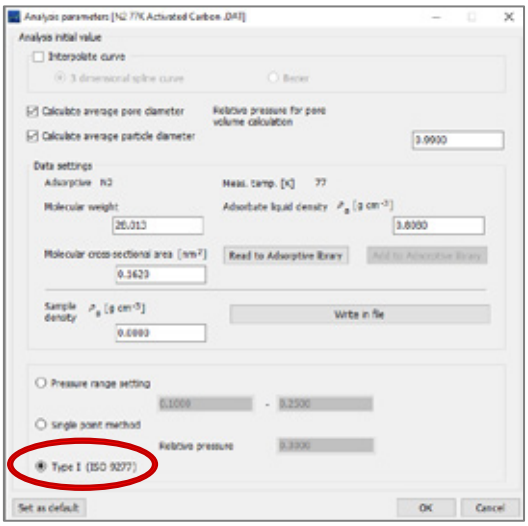
Modern instrumentation makes these measurements quite feasible for all varieties of research and industry. Microtrac MRB's BEL product line constitutes a series of manometric/volumetric and dynamic flow measurements. These systems are capable of collecting data automatically and efficiently.

Fig. 2 – A selection of gas adsorption analyzers from Microtrac capable of collecting high quality measurements.

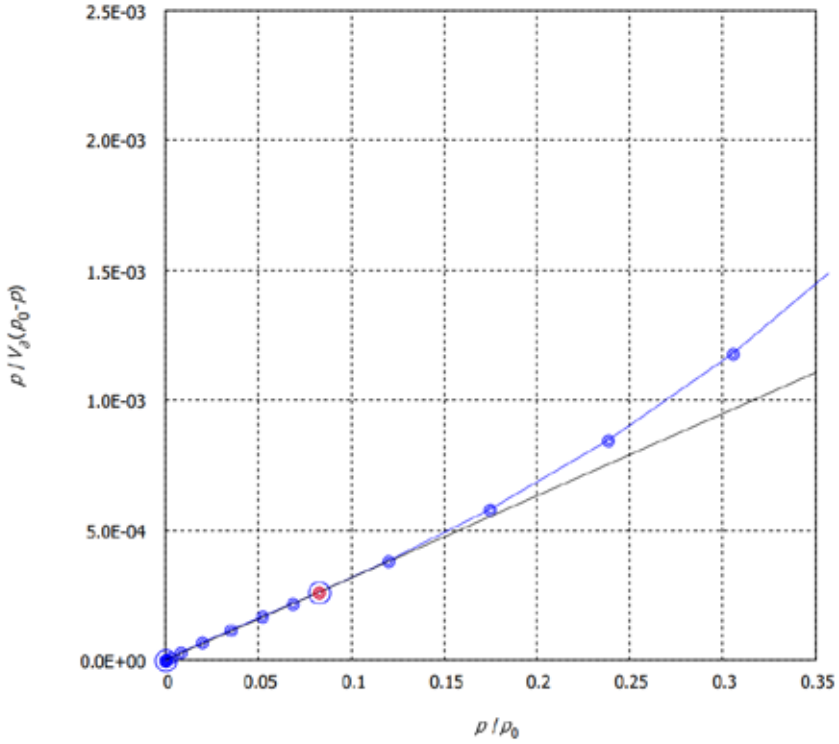
Our analysis software, BELMaster7, can automatically determine the upper limit per the Rouquerol plot, so anyone can easily determine the proper BET range in type I materials.

To use this function, follow these steps:

1. Select the isotherm to be analyzed
2. Click on ‘Analysis’ à ‘BET plot’
3. Select the generated BET plot
4. Click on ‘Settings’ à ‘Analysis parameters settings’
5. Select Type I (ISO 9277), then ‘OK’



The BET range limit will be marked in in a color that stands out, regardless of the selected data color.



Industrial Standard Methods

Standard methods are important for validating industrial processes. What follows are a list of some important industrial standards relating to the collection of specific surface areas for microporous materials. All the listed methods are capable of being collected using instrumentation here at Microtrac MRB. As there are a vast number of accepted standard methods, as well as new ones being accepted continuously, there will certainly be soe missing from this list. If you don't see exactly what you're looking for, or the standard method of your choice isn't on the list but you're unsure if it would still apply, please reach out to us at info@microtrac.com and we'll be happy to work with you to find out if adsorption is right for you.

ASTM D4365
Standard Test Method for Determining Micropore Volume and Zeolite Area of a Catalyst

ISO 9277
Determination of the specific surface area of solids by gas adsorption — BET method

ISO 15901-3
Pore size distribution and porosity of solid materials by mercury porosimetry and gas adsorption — Part 3: Analysis of micropores by gas adsorption

DIN 66135-1
Particle measurement technology - Micropore analysis using gas adsorption - Part 1: Basics and measurement methods

DIN 66135-2
Particle characterization - Micropore analysis by gas adsorption - Part 2: Evaluation by isotherms comparison

DIN 66135-3
Particle measurement technology - Micropore analysis using gas adsorption - Part 3: Determination of the micropore volume according to Dubinin and Radushkevich

DIN 66135-4

Particle size analysis - Micropore analysis by gas adsorption - Part 4: Determination of pore distribution according to Horvath-Kawazoe and Saito-Foley

Summary

When it comes to analyzing micropores, great care must be taken. Fortunately, methods such as the Rouquerol criteria exist to help validate data. The BET method is very widely used, and unfortunately the Rouquerol plot is not as widely considered. Hopefully, this document has provided you an entry point into the utility of this method.

If you're unsure of the application or how this technology could help your research or industrial needs, please contact info@microtrac.com.

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