THE microReport

micromeritics®
The Science and Technology of Small Particles™

MICROMERITICS INSTRUMENT CORPORATION

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In This Issue

CERTIFIED ISO9001 MANUFACTURER NORCROSS, GA

www.micromeritics.com

Micromeritics Version 3.0 Software for the ASAP 2020 Surface Area and Porosity Analyzer

Enhanced features for Fuel Cell and Hydrogen Storage researchers.

Version 3.0 provides options specifically designed to help fuel cell and hydrogen storage researchers easily obtain hydrogen adsorption isotherms. Enhancements allow users to collect isotherms by specifying absolute pressure targets, which are commonly used for hydrogen adsorption studies. The ASAP 2020 report system has also been expanded to provide Pressure Composition Isotherms. Tabular reports have been updated to include weight percent adsorbed. All of these upgrades are included to simplify the collection of hydrogen adsorption isotherms and also provide final reports which quickly provide more information to the researcher.

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ASAP 2020 V3.0

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The ASAP 2020 Version 3.0 software also includes substantial upgrades for researchers synthesizing mesoporous materials.

The report system has been updated to provide new models and methods for mesopore characterization. Version 3.0 includes Density Functional Theory models for both microporous and mesoporous materials, and the traditional t-plot and BJH models have been enhanced for these materials. The additional reports are included to further improve the quality of pore size distributions obtained from the ASAP 2020.

Application Deadline for the Initial Instrument Grant is

September 30, 2006

Instrument Grant Program Launched

Micromeritics has recently launched an Instrument Grant Program intended to provide particle characterization instruments to non-profit universities and research organizations for the purpose of fostering and supporting meritorious research projects.

Micromeritics is now accepting applications for its newly instituted Instrument Grant Program. Researchers at non-profit universities and institutions will now have the opportunity to acquire the use of expensive particle characterization instrumentation that might not have been otherwise available.

Types of instrumentation that will qualify as Donated Equipment include particle size analyzers, gas adsorption analyzers, mercury porosimeters, gas pycnometers, and chemisorption instrumentation. Since the nature and scope of the instruments that may be requested will vary, it is anticipated that the value of awards will vary also.

Major recipients of a Program Grant of the Donated Equipment may include individual researchers or a group of researchers within the same department or from several departments at one institution. In addition, the Donated Equipment may be shared by two or more institutions provided the grant application demonstrates the feasibility of such shared use.

For a detailed grant description, application requirements, application, and submission information, see our web-site, *www.micromeritics.com.*

The President of the company has appointed a special committee to review the merits of all applications. A maximum of one instrument/integrated system will be awarded per calendar quarter. Program Grants are also limited to a maximum of one instrument/integrated system per application. Applications may be submitted at any time in accordance with the application instructions. Applications are good for a period of one year from the date of submission.

Micromeritics Releases an Extensive Bibliography Referencing More Than 6,000 Scientific Papers

For those interested in the physical characteristics of powders and solids for fundamental research, product development, quality assurance and control, production, or process control applications, this bibliography is an indispensable tool.

In this series of peer-reviewed papers, various particle characteristics such as surface area, particle size, pore volume, pore size, absolute density, envelope density, bulk density, catalytic activity, and active surface area are determined for a wide variety of materials. Each of these technical papers references one or more Micromeritics products. The list by no means represents a complete and exhaustive search of the literature, but includes those papers that are available from various sources on the internet.

We have often been asked for references to papers in which a scientist uses a Micromeritics instrument to study the same or similar materials as the new user intends to study. This document will satisfy that need. This bibliography also serves as a powerful reference resource for all those performing, involved in, or dealing with particle characterization.

This bibliography is available for download on our website: www.micromeritics.com in Adobe® Acrobat® PDF file format, which easily can be searched by key words and phrases using the 'Find' feature of Adobe's free Acrobat Reader®.

Michigan Catalysis Society 2006 Giuseppe Parravano Memorial Award Winner

Levi T. Thompson, Jr., Professor of Chemical Engineering, University of Michigan, has been selected as the winner of the 2006 Michigan Catalysis Society Parravano Award for Excellence in Catalysis Research and Development.

Prof. Thompson was chosen for outstanding contributions to the development of novel carbide and nitride catalysts, and for his passion in mentoring and educating undergraduate and graduate engineering students, instilling in them enthusiasm for catalysis research.

Dr. Thompson has been a customer and user of Micromeritics gas adsorption instrumentation for more than 10 years. He has utilized the ASAP 2000 series instruments for physisorption and chemisorption and the AutoChem series for temperature-programmed experiments on catalysts.

Searching for the Optimum Adsorption Temperature by the use of the Temperature-Programmed Adsorption (TPA) Technique

By: Dr. Simone Yunez, Laboratorio de Caracterización de Catalizadores, INTEVEP, Research Centre of Petróleos de Venezuela, Los Teques, Venezuela email: zamoraju@pdvsa.com Yunezs@pdvsa.com

A temperature-programmed adsorption technique has been applied at INTEVEP, Petróleos de Venezuela, to investigate the optimum temperature for H_2 chemisorption on cobalt-supported catalysts. It has been seen that selecting the appropriate adsorption temperature makes a large difference on the measured value of the dispersion. Moreover, this technique can be extended to any adsorption system regardless of its adsorbate and adsorbent components.

Introduction

Selection of the optimum chemisorption temperature for a specific adsorbate in catalysis has been and still is a matter of investigation. Usually this temperature is selected to be near ambient (35 °C) for convenience. Thus, the selected temperature could be far from the appropriate temperature at which the chemisorption process is activated. Using a wrong adsorption temperature could yield lower values for the dispersion of the active metals on catalysts. Hence, the use of the temperature-programmed adsorption (TPA) technique would lead to the correct activation temperature and, therefore, to the correct values for the different parameters of the catalyst. A series of cobalt-loading-supported-silica catalysts has been studied. The unique purpose was to determine the optimum H_2 chemisorption temperature, and therefore to relate the volume of the chemisorbed hydrogen to the dispersion and the active metal on the catalyst. A temperature-programmed desorption and reduction (TPD/TPR) Micromeritics instrument, the AutoChem 2920, has the capability of reducing the initial temperature below ambient. The instrument was coupled to an Omnisorb Mass Spectrometer to follow the H_2 signal to ensure the correctness of the thermal conductivity detector (TCD) response. A mass of about 0.2 g of the original sample has been taken for each experiment. First, the sample was reduced following a TPR profile with a mixture of 5% H_2 in argon. The ramping temperature was 10 °C/min up to the calcination temperature (450 °C). The reduction process was then kept isothermal until the TCD response returned to baseline, to avoid any possible sintering of the active phase. Just after reduction was completed, the sample temperature was reduced to –10 °C under a continuous flow of the reducing mixture (5% H_2 in argon), which was also used for the subsequent experiments. Once the TCD signal returned to baseline indicating the equilibration of the experiment and possible saturation with H_2 of the sample, the sample temperature was then programmed to increase 10 °C/min up to 200 °C. At this point, any possible activity (adsorption and/or desorption of H_2) by the sample was recorded by

both the TCD and the Mass Spectrometer.

Results

The TPA profiles showed a fairly symmetric peak at about 100 °C. On the other hand, the Mass Spectrometer, which was connected at the exit of the TCD, demonstrated that this peak corresponds to the consumption of H_2 by the catalyst. This same experiment was repeated several times to ensure reproducibility of the peak position. All experiments gave almost identical TPA profiles.

Now having established the optimum temperature for H_2 chemisorption for these particular catalysts by TPA, complementary experiments were necessary to ensure that 100 °C was really the optimum temperature at which H_2 uptake is maximum. Four samples from the same original batch were taken for these new experiments using the Micromeritics ASAP 2010 high vacuum system to obtain the adsorption isotherms. The sample was first reduced at 450 °C for 4 hours, and then submitted to high vacuum for 4 hours to ensure the complete elimination of any excess H_2 remaining after reduction.

Each reduced sample was tested for H_2 chemisorption at different analysis temperatures; 35, 70, 100, and 140 °C. The adsorption isotherm was obtained by increasing the H_2 pressure from 10 mmHg to atmospheric pressure. The uptake of H_2 at the monolayer was determined from the extrapolation of the adsorption isotherm to the y-axis (absolute pressure in mmHg). Results of these experiments are plotted in Figure 1. The dispersion and the active metal surface area were obtained according to the classical methods of calculation.

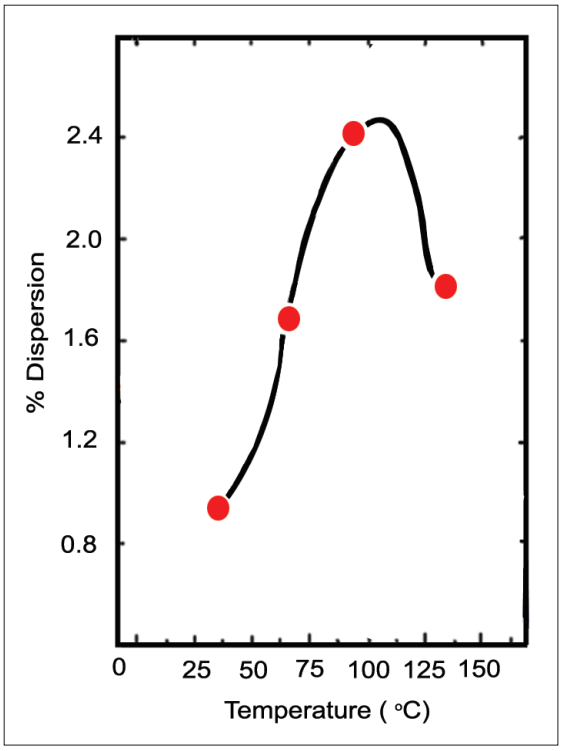

Figure 1 shows the variation of the % dispersion as function of the analysis temperature.

Conclusion

It can be concluded from these results that the TPA technique is a very powerful tool for use in determining the appropriate analysis temperature for chemisorption. The technique could be used and applied for any process of this kind regardless of the adsorbate and adsorbent in use. Combination of the volumetric, the flowing and the Mass Spectrometer systems should yield definitive results in many other applications in catalytical processes. A catalyst characterization laboratory should combine as many different techniques as possible to ensure complete knowledge of the catalyst properties. This will permit the activity and selectivity of the catalyst to be known long before being loaded into reactors and high expenses incurred.

MICROMERITICS ANALYTICAL SERVICES The Particle Testing Authority

Micromeritics Analytical Services and MVA Scientific Consultants celebrated the one-year anniversary of their strategic alliance on May 18. The strategic alliance allows both companies to better serve customers by offering a more complete solution for their application. MVA Scientific Consultants provides critical analysis and support for a variety of public and private sector interests through the use of electron and light microscopy. The firm's staff of world-class specialists offers over 100 years of combined experience in particle identification and source determination, materials characterization, and surface metrology.

Tim Vander Wood (left), Executive Director of MVA Scientific Consultants presents a micrograph to Preston Hendrix, President of Micromeritics Instrument Corporation, celebrating the one-year anniversary of their strategic alliance.

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New Techniques and Services

Micromeritics Analytical Services has added a high-pressure, static gas adsorption analyzer capable of measuring gas adsorption isotherms up to 10 atm. Another new high-pressure system is capable of measuring microreactions up to 100 atm, using flowing gases and a TCD/mass spec detection system. Both of these techniques allow customers to better understand how their materials will behave under real-world conditions.

Web Site Updates

Particletesting.com has a new look and a new page dedicated to the Application of the Month. Each month we will be selecting one sample which is unique and out of the ordinary. Check out June's selection; a fully intact squirrel skull.

New Information

Micromeritics Analytical Services has released a new presentation which describes the benefits of outsourcing analytical testing services and the range of services offered by Micromeritics Analytical Services. This presentation can be downloaded from the web site www.particletesting.com/downloads.aspx, or a CD can be mailed to you.

Mark your calendars.

Micromeritics Analytical Services will be exhibiting at the 2006 AAPS National Meeting and Exposition. The exhibition and meeting will be held in San Antonio, TX October 30 – November 2.

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Micromeritics Instrument Training Courses

Training is provided for most Micromeritics instrumentation at the time of installation. This training presents all the information required for a new operator to quickly become proficient operating the instrument. In cases where personnel changes occur or more advanced training is required, Micromeritics conducts a variety of classes for many of our instruments. These courses are held at our headquarters in suburban Atlanta, Georgia. The courses include:

Detailed Operational Procedures
Items covered are effective sample file creation, use of analysis parameters, and manual sample entry. You´ll learn how to utilize the full power and flexibility of the operating software.

Automatic Analysis
Develop correct analysis procedures to optimize collection of accurate, reproducible data. Much of the class time is spent performing analyses in a controlled, tutorial environment.

Systems Utilities
Discover all of the instrument software utilities which help you manage sample information files and directories, protect data, and select system options.

Troubleshooting
Learn techniques that enable you to locate and quickly resolve instrument problems.

Report Generation and Comprehension
Learn to configure reports and obtain more useful information, as well as improve comprehension of the reports produced.

User Maintenance
Practice routine maintenance procedures which improve operation, reduce downtime, and increase data accuracy.

Theory Overview
Learn about the scientific theory upon which each instrument is based and how it applies to the critical factors relevant to successful sample preparation and analysis performance.

Enrollment
Training courses last from 2 to 3 days and are designed to provide hands-on, performance-based instrument knowledge. Small classes guarantee close individual attention. Included in the course materials are a Study Guide, an instrument Operator´s Manual, and other handout materials. Certificates of Completion are also awarded to all trainees.

Training 2006

Saturn DigiSizer® 5200
August 15 - 17

SediGraph 5120
August 29 - 31

AutoPore
September 12 - 14

Gemini V
September 19 - 20

AutoChem II
October 31 - November 2

TriStar
November 7 - 9

For additional information or to register for the class of your choice, contact the Micromeritics Training Department at 770.662.3607. Early registration is recommended since class space is limited.

See our website for a complete course schedule.
www.micromeritics.com

UOP employees attending the recent TriStar training course.

Left to Right:
Phillip Jackson
Doug McCoy
*Ana Vazquez - Medina**
Darryl Florence
Peter Rumfola
Ginger Murray
Robin Bankard
George Gordon
**Trainer*

Events

American Chemical Society Meeting and Exposition
September 10 - 14
The Moscone Center Booth # 224
San Francisco, CA

Materials Science & Technology 2006 Conference and Exposition
October 15 - 19
Cinergy Center Booth # 224
Cincinnati, OH

2006 AAPS Annual Meeting and Exposition
October 15 - 19
Henry B. Gonzalez Convention Center Booth # 941
San Antonio, TX

ICE 2006
November 1 - 3
Ernest N. Morial Convention Center Booth # 336
New Orleans, LA

See our web-site for a complete Event schedule www.micromeritics.com

Attention Authors

We welcome articles and information concerning particle technology applications performed with Micromeritics instrumentation. Everything from a single plot with operating conditions to an in-depth article on physisorption, chemisorption, etc. with supporting plots will be considered. If your material is published in The microReport, you will receive a copy of Analytical Methods in Fine Particle Technology by Paul A. Webb and Clyde Orr.

Send your article to:
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The microReport
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Include your title, return address and phone number. Contributions cannot be returned, but each will be acknowledged.

How To Reach Us

Micromeritics offers over 50 sales, service, and distribution offices throughout the world. For additional information, a free product demonstration, or the location of the office nearest you, call or write:

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Subscriptions are free to particle technology users and can be started simply by writing to The microReport editor.

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