

iGC-SEA

Inverse Gas Chromatography-
Surface Energy Analyzer



Surface Measurement Systems
World Leader in Sorption Science

For characterizing:

- Particles and powders
- Nanomaterials
- Films
- Fibers
- Composites and components
- Bulk solids



SURFACE ENERGY

The Key to Understanding Surface Properties

The factors which control the behavior and performance of many particulate solids, powders, fibers and films are often poorly understood. Such solids often display problems during manufacture, usage or storage across all industrial sectors.

Typically, particulate solids are subject to cursory characterization from a physical chemistry perspective, and often all that is known is the particle size or BET surface area of the solid. Contrast this with the detailed analytical chemical information, including the chemical structure and morphology as determined by NMR, FTIR, XRD, GC-MS and HPLC, which is routinely available. However, none of this information describes the thermodynamic state of the material. Researchers have now established that one of the most important properties of a powder, particulate material, film or fiber is its surface energy.

Surface energy γ , is the principle characteristic measured by the Inverse Gas Chromatography-Surface Energy Analyzer (iGC-SEA). The surface energy of a solid is analogous to the surface tension of a liquid and is a measure of attractive intermolecular forces in a solid.

It is the same intermolecular forces which are responsible for the attraction between powder particles and other solids, liquid and vapor molecules which can occur via long range van der Waals forces (dispersion forces) and short range chemical forces (polar forces). Thus, surface energy values (dispersive and polar) correlate to several key solid properties including wetting, dispersability, powder flowability, agglomeration, process-induced disorder, adhesion/cohesion, static charge, adsorption capacity and surface chemistry.

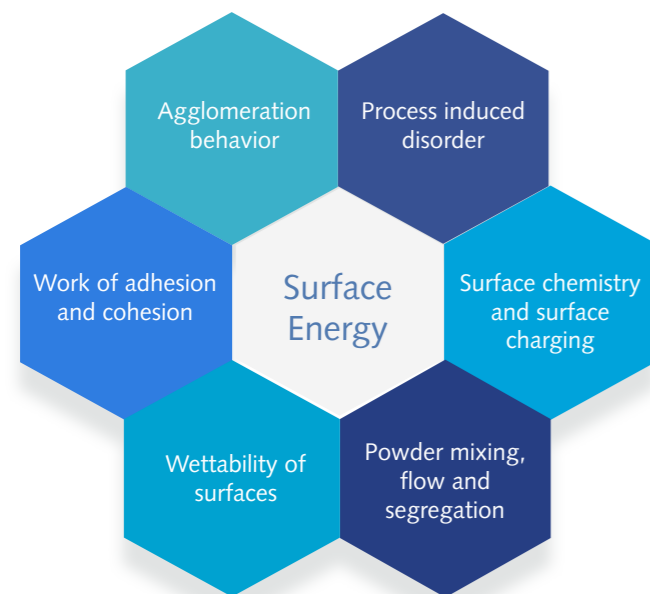
The iGC-SEA probes the solid surface interface by exposing the solid sample to vapor probes of known properties. The intermolecular forces that result from this interaction can be analyzed to quantify the total surface energy of the sample.



Experimental Technique for Measuring Surface Energy

There are a range of techniques available for measuring the surface energy of solid particulate materials. Though contact angle measurement is by far the most common method, it is rarely used for particle and other non-planar materials due to experimental limitations leading to inaccurate and unreliable results. Inverse gas chromatography is now the proven and preferred method for surface energy measurements, and surface energy heterogeneity in particular.

Inverse Gas Chromatography (iGC) is a gas-solid technique for characterizing surface and bulk properties of powders, particulates, fibers, films and semi-solids. A series of vapor pulses are injected through a column packed with the sample of interest. Unlike traditional analytical gas chromatography, iGC is a physical chemistry technique using vapor probes with known properties to characterize the unknown surface/bulk properties of the solid sample.



Understanding solid properties related to surface energy

The chart below shows different techniques and capabilities for measuring surface properties.

Inverse Gas Chromatography (iGC)	Atomic Force Microscope (AFM)	Contact Angle (CA)	Wetting Balance
Ok for flat surfaces.	Ok for flat surfaces.	Excellent for flat surfaces.	Excellent for flat surfaces.
Excellent for particulates - repeatable, no-hysteresis or roughness effects.	Not well suitable for particulates - slow and poor data statistics.	Not suitable for particulates - swelling, hysteresis, dissolution, surface roughness.	Not suitable for particulates - swelling, hysteresis, dissolution, surface roughness.
Surface energy heterogeneity with a wide range of probe molecules.	Theory for determining surface energy can be complex.	Very few solutes possible.	Very few solutes possible.
Can measure vapor adsorption isotherms as well as surface energy.			

iGC-SEA for Measuring Surface Energy

What is iGC-SEA?

iGC-SEA or Inverse Gas Chromatography-Surface Energy Analyzer is an instrument that uses the iGC principle. The heart of its innovation is the patented injection manifold system which generates accurate solvent pulse sizes across a large concentration range, resulting in isotherms at unprecedented high and low sample surface coverages. This allows for the accurate determination of surface energy heterogeneity distributions. The fully automated iGC-SEA can be operated at different solvent vapor, flow rate, temperature, humidity and column conditions.

iGC-SEA has a unique data analysis software called Cirrus Plus, specifically designed to measure surface energy heterogeneity, isotherm properties and related physical characterization parameters. Further, bulk solid property experiments resulting from probe-bulk interaction and using solubility theory are now possible. It automatically and directly provides a wide range of surface and bulk properties of the solid samples and gives more accurate and reliable data than manual calculations.

iGC-SEA also has a humidity control option. The **impact of humidity** and temperature can be determined for the physico-chemical properties of solids such as as moisture induced T_g , BET specific surface area, surface energy, wettability, adhesion and cohesion.



iGC-SEA is used in characterizing particles, powders, fibers, films, nanomaterials, composites, components and bulk solids.

iGC-SEA APPLICATIONS

Industries that benefit from iGC-SEA:

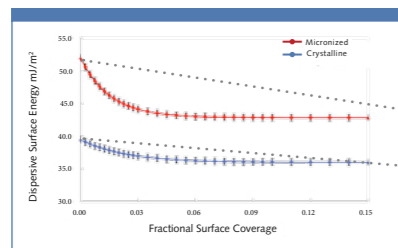
Aerospace
Building Materials
Chemical

Energy
Food
Industrial Materials

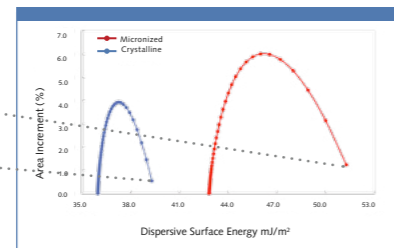
Nuclear
Pharmaceutical
Personal Care

Surface Energy Heterogeneity Profiling

Dispersive Surface Energy **Profiles**
Budesonide Samples



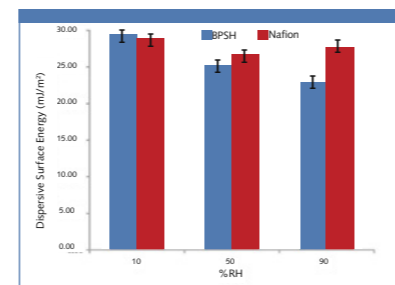
Dispersive Surface Energy **Distributions**
Budesonide Samples



The surface energy distribution is the integration of the surface energy profile across the entire range at surface coverage and is analogous in principle to a particle size distribution.

Dispersive, Polar and Acid-Base Surface Energy/Chemistry

Competitive sorption: Dispersive surface energy values for different proton exchange membranes as a function of background relative humidity conditions.



Gutmann acid (K_a) and base (K_b) values for different polymers along with their relative ranking on the triboelectric series.

Triboelectric Series Order	K_b / K_a
Polymethyl methacrylate	1.33
Polycarbonate	1.10
Acrylonitrile	1.09
Polybutadiene-Styrene	
Polypropylene	0.63
Polyvinylchloride	0.02

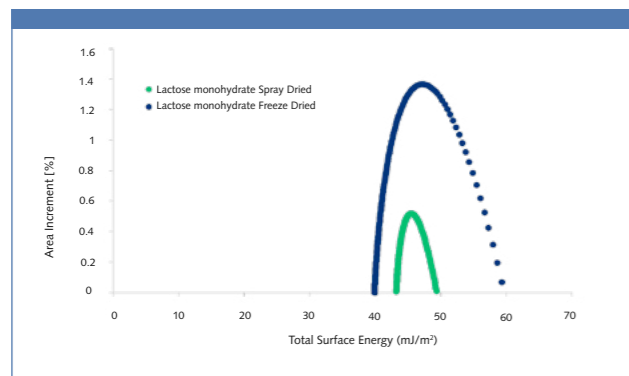
Works of Adhesion and Cohesion

Work of adhesion and work of cohesion values for different nanofiller-polyurethane composites correlate directly with composite mechanical properties. There is a good blending performance with a W_{ad}/W_{coh} ratio near 1.

Sample	W_{ad}/W_{coh}	Tensile Strength at Break (MPa)
Polyurethane Alone	-----	61 ± 4
As Received Multi-walled Nanotube	0.55	60 ± 7
Oxidized Multi-walled Nanotube	0.49	56 ± 6
As Received Nanoclay	0.47	54 ± 11
Functionalized Nanoclay	0.86	71 ± 7

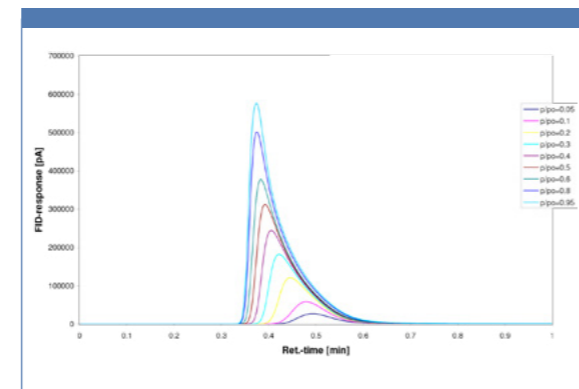
Spray Dry (SD) and Freeze Dry (FD) Product Evaluation

The SD product/sample has an energetically more homogenous surface due to a more uniform particle size and shape. The FD product/sample exhibits a wide variation of surface energy sites.

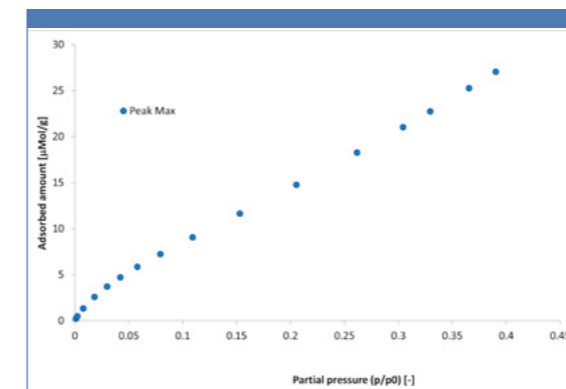


Adsorption Isotherms, Heats of Adsorption & Henry Constants

Series of pulses for a multiple injection experiment (variable concentration) on M745 with hexane at 303 K.

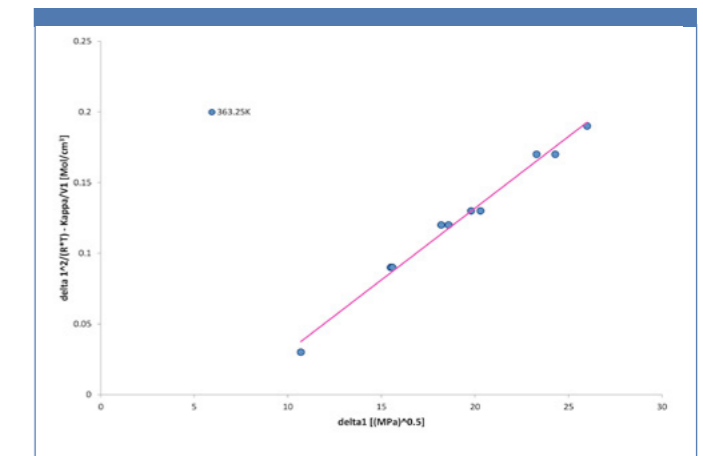


Peak maximum sorption isotherms of hexane on M745.



Solubility Parameters (Hildebrand and Hansen)

Hildebrand solubility parameter for Polymethyl Methacrylate. Value of $19.08 \text{ MPa}^{1/2}$ agrees with literature values ($17.4\text{-}21.3 \text{ MPa}^{1/2}$).



"iGC-SEA represents a huge advance in inverse gas chromatography and physico-chemical measurement."

APPLICATION NOTES

201 Gas phase diffusion studies of cyclohexane by infinite dilution inverse gas chromatography.

202 Determination of the dispersive surface energy of Paracetamol by pulse inverse gas chromatography at infinite dilution.

203 Heat of sorption studies on micro crystalline cellulose by pulse inverse gas chromatography at infinite dilution.

204 Determination of the glass transition temperatures T_g of maltose and its dependence on relative humidity by infinite dilution inverse gas chromatography.

205 The determination of the solubility parameter of different starch types by infinite dilution inverse gas chromatography.

206 Determination of permeability coefficients of alkanes in polyethylene powder by infinite dilution inverse gas chromatography.

207 Characterization of drug polymorphs by inverse gas chromatography.

208 The measurement of isotherms by pulse inverse gas chromatography.

209 An investigation of Chromosorb silicas as support materials for inverse gas chromatography.

210 Investigation of the influence of bleaching conditions on surface properties of standard hair samples by inverse gas chromatography.

211 An investigation of minerals used in asphalt by inverse gas chromatography.

213 Determination of thermodynamic parameters by frontal inverse gas chromatography.

214 The determination of the permeability and the activation energy of diffusion of drug powders by infinite dilution inverse gas chromatography.

215 A sorption study on microporous materials by finite dilution inverse gas chromatography.

216 Characterization of surface properties of glass fibres by inverse gas chromatography.

227 Determination of Acid-Base Component of the Surface Energy by Inverse Gas Chromatography.

302 An Overview- Characterization of strong solid-vapour interactions by inverse gas chromatography.

303 An Overview of iGC-SEA - A new instrumental technique for characterizing the physico-chemical properties of polymers.

304 An Overview of Characterization of alumina and related surfaces by inverse gas chromatography.

PUBLICATIONS

"Surface characterization of standard cotton fibres and determination of adsorption isotherms of fragrances by IGC" (*Surface and Interface Analysis*, DOI: 10.1002/sia.5811)

"Effect of milling on particle shape and surface energy heterogeneity of needle-shaped crystals" (*Pharm Res* (2012) 29:2806-2816 DOI: 10.1007/s 11095-012-0842-1)

"Measuring surface roughness of pharmaceutical powders using vapor sorption methods" (*AAPS PharmSciTech* (2010) DOI: 10.1208/s12249-010-9571-0)

"Use of surface energy distributions by inverse gas chromatography to understand mechanofusion processing and functionality of lactose coated with magnesium stearate" (*European Journal of Pharmaceutical Sciences* 43 (2011) 325-333)

"Influence of fines on the surface energy heterogeneity of lactose for pulmonary drug delivery" (*International Journal of Pharmaceutics* 388 (2010) 88-94)

"Determination of surface heterogeneity of D-Mannitol by sessile drop contact angle and finite concentration inverse gas chromatography" (*International Journal of Pharmaceutics* 387 (2010) 79-86)

"Inverse gas chromatographic method for measuring the dispersive surface energy distribution for particulates" (*Langmuir* 2008, 24, 9551-9557)

"Analysis of surface properties of cellulose ethers and drug release from their matrix tablets" (*European Journal of Pharmaceutical Sciences* 27 (2006) 375-383)

"Inverse Gas Chromatography of As-Received and Modified Carbon Nanotubes" (*ACS- Langmuir Article*, DOI: 10.1021/la900607s)

CASE STUDIES

603 Correlating drug-binder adhesive strengths measured by using Inverse Gas Chromatography with tablet performance.

605 The effect of primary particle surface energy on agglomeration rate in fluidised bed wet granulation.

To know more about iGC-SEA applications, publications and case studies, please email info@surfacemeasurementsystems.com.

INSTRUMENT PLATFORM

Surface Measurement Systems continues as the world leader in inverse gas chromatographic instrumentation as illustrated by the iGC-SEA. Designed and manufactured in-house with extensive customer participation and feedback, the iGC-SEA meets and exceeds the need for a stable, reliable and easy to use surface energy heterogeneity and bulk property sorption solution.

iGC-SEA Hardware

Unique variable injection system with a 1:4000 injection volume ratio.

12 solvent reservoirs:

Easy access drawers.

Temperature-controlled for vapor stability

Flame Ionization

Detector (FID):

Adjustable gain.

Fully integrated design:

Provides easy access to the sample column oven and solvent reservoirs.

2-sample column design:

Higher throughput.

H₂ & Organic Vapor Leak Detector

Sample column oven: 20°C to 150°C

Fully integrated.

Optional:

Film/monolithic sample holder

Background humidity control



Minimized bench requirements:

System Dimensions:

W:~490mm

H:~650mm

D:~564mm

System Weight: 80kg



iGC-SEA straight column design - simple sample packing and column loading



Columns with samples (L) and film cell (below)



Cirrus Plus: iGC-SEA Data Analysis Software

Cirrus Plus is the iGC-SEA data analysis software. It harnesses unsurpassed experimental flexibility, delivering extensive and user-friendly data analysis alongside one-click report generation. Cirrus Plus enables routine system operation and data analysis minimizing operator interaction time.

Standard features include:

- Isotherm determination/BET/Henry constant
- Surface energy analysis
- Surface heterogeneity mapping
- Work of adhesion/cohesion determination
- Competitive sorption measurement
- Heats of adsorption/sorption measurement
- Acid-base chemistry analysis

Advanced features include:

- Glass Transition Temperature
- 1-D and 3-D solubility parameters
- Crosslink density
- Work of cohesion and adhesion

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