iGC-SEA

Inverse Gas Chromatography-
Surface Energy Analyzer

Surface Measurement Systems
World Leader in Sorption Science

For characterizing:

- Particles and powders
- Nanomaterials
- Films
- Fibers
- Composite components
- Pharmaceuticals

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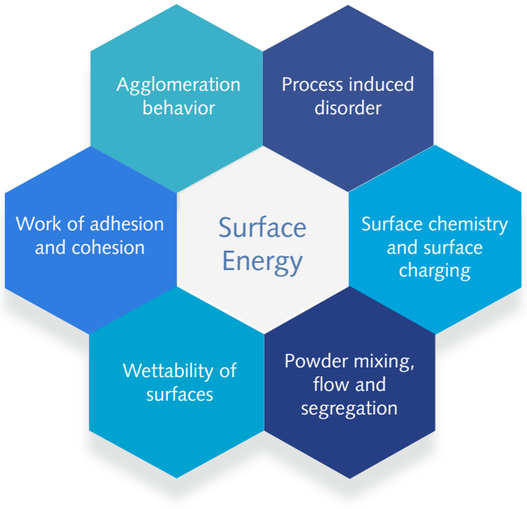
SURFACE ENERGY

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.

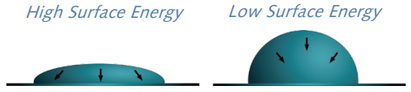
Surface energy Υ, is the principle characteristic of solids measured by the Inverse Gas Chromatography-Surface Energy Analyzer (iGC- SEA). The surface energy of a solid is analogous to the surface tension of a liquid and it is a measure of attractive intermolecular forces on a solid surface.



Understanding solid properties related to surface energy

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 chromatogrphy 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.

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.	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 area.			

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.

iGC-SEA has a unique data analysis software, specifically designed to measure surface energy heterogeneity, isotherm properties and related physical termodynamic 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, thereby the **impact of humidity** and temperature can be determined for the physico-chemical properties of solids such as as moisture induced Tg, BET specific surface area, surface energy, wettability, adhesion and cohesion.

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

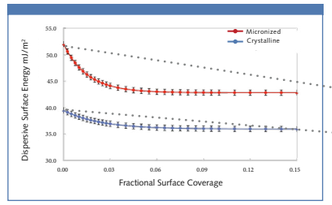
iGC-SEA APPLICATIONS

Industries which benefit from iGC-SEA:

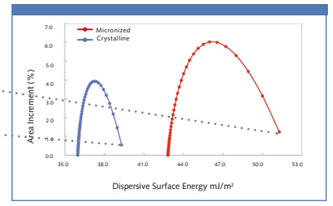
Aerospace
Building Materials
Chemical
Energy
Food
Composite Materials
Nuclear
Pharmaceutical
Personal Care

Surface Energy Heterogeneity Profiling

Dispersive Surface Energy **Profiles**
Budesonide Samples



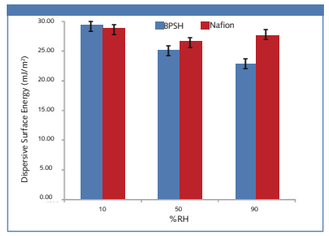
Dispersive Surface Energy **Distributions**
Budesonide Samples



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 and Acid-Base Surface Energy/Chemistry

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 metharcrylate	1.33
Polycarbonate	1.10
Acrylonitirile	1.09
Polybutadiene-Styrene	
Polypropylene	0.63
Polyvinylchloride	0.02

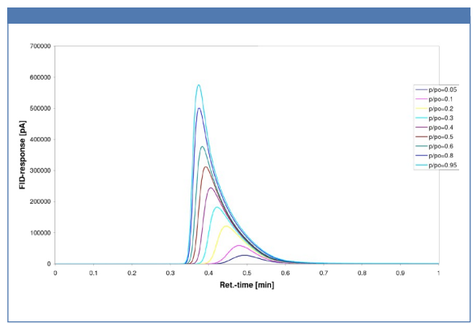
Works of Adhesion and Cohesion

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

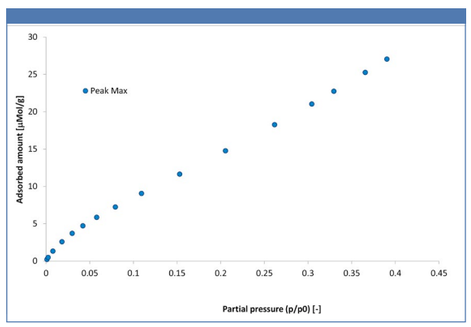
Sample	W_{ad}/W_{coh}	Tensile Strength at Break (MPa)
Polyurethane Alone	-----	$61^{+}4^{-}$
As Received Multi-walled Nanotube	0.55	$60^{+}7^{-}$
Oxidized Multi-walled Nanotube	0.49	$56^{+}6^{-}$
As Received Nanoclay	0.47	$54^{+}11^{-}$
Functionalized Nanoclay	0.86	$71^{+}7^{-}$

Adsorption Isotherms, Heats of Adsorption & Henry Constants

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

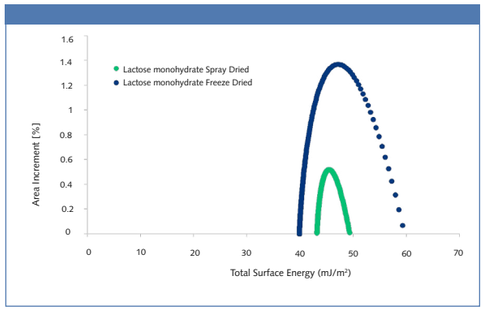


Sorption isotherms of hexane by pulse injections on M745.



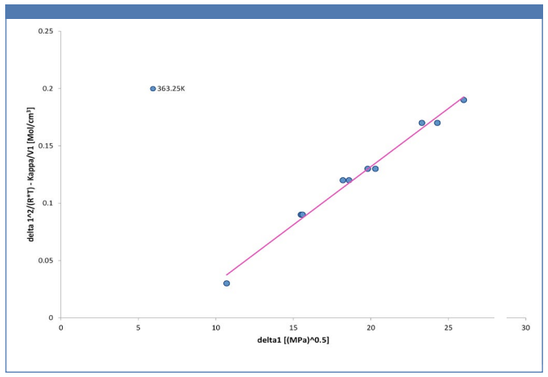
Spray Dry (SD) and Freeze Dry (FD) Product Evaluation

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.



Solubility Parameters (Hildebrand and Hansen)

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



"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 Tg 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 sterate" *(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 granualtion.

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 gas phase injection system with a 1:4000 injection volume ratio.

12 solvent reservoirs:
Easy accesss drawers.
Temperature-controlled for vapor stability

Flame Ionization Detector (FID):
Adjustable gain

Fully integrated design

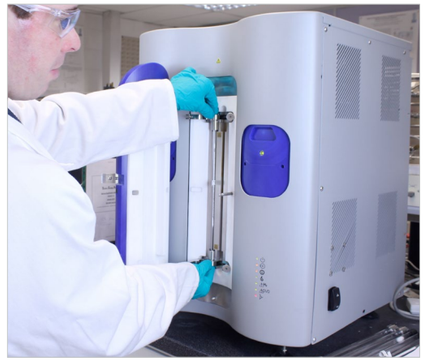
H_2 & Organic Vapor Leak Detector

2-sample column design:
Higher throughput

Sample column oven:
20°C to 150°C
Fully integrated

Minimized bench requirements:
System Dimensions:
W:~490mm
H:~650mm
D:~564mm
System Weight: 60kg

Optional:
Film/monolithic sample holder
Background humidity control



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

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

carbon fiber *cotton* *hair* *granules* *powder* *medical metal implants*

iGC-SEA Data Analysis Software

iGC-SEA data analysis software 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
- Competitive sorption measurement
- Acid-base chemistry analysis

Advanced features include:

- Glass Transition Temperature
- Hildebrand and Hansen solubility parameters
- Crosslink density
- Work of adhesion/cohesion determination
- Heats of adsorption/sorption measurement

UK (Head Office)
Unit 5 Wharfside, Rosemont Road,
Alperton, London, HA0 4PE, UK
Telephone: +44 (0) 208 795 9400

USA (Regional Office)
2125 28th Street, SW, Suite 1
Allentown, PA, 18103
Telephone: +1 610 798 8299

Email: info@surfacemeasurementsystems.com
www.surfacemeasurementsystems.com

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