Surface Measurement Systems
World Leader in Sorption Science

iGC-SEA

Inverse Gas Chromatography-Surface Energy Analyzer

Surface Characterization

For materials such as:

Particles and powders

Nanomaterials

Films

Fibers

Composite components

Pharmaceuticals

ACCURATE. PRECISE. AUTOMATED.

Surface Energy

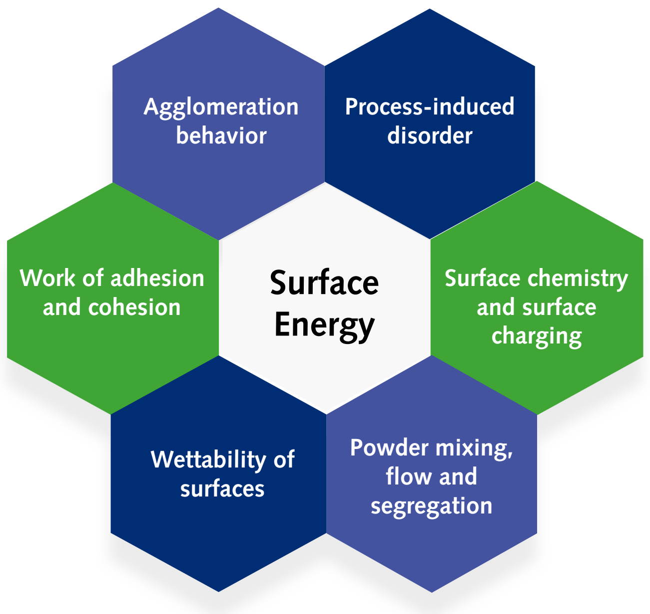
The Key to Understanding Surface Properties

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 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 particulate 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.

Contact angle for droplets on surfaces

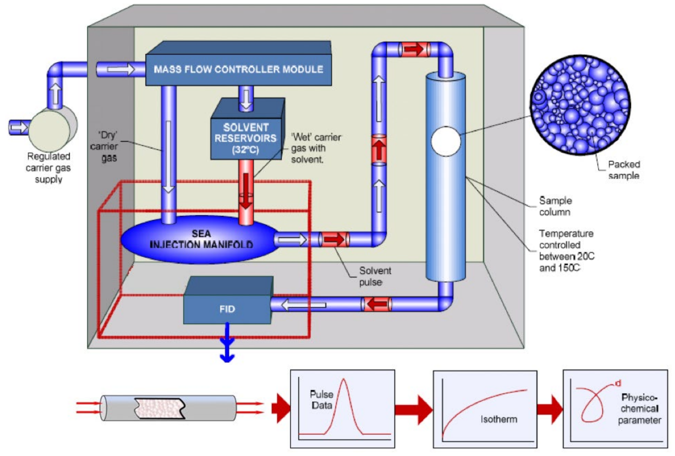
Inverse Gas Chromatography (iGC) is a gas-solid technique for characterizing surface and bulk properties of powders, particulates, fibers, films and semi-solids. A series of vapor pulses are injected through a column packed with the sample of interest. Unlike traditional analytical gas chromatography,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

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 humidity control option, thereby the **impact of humidity and temperature** can be determined on the physico-chemical properties of solids such as moisture induced T_g, BET specific surface area, surface energy, wettability, adhesion and cohesion.

The system also has a unique data analysis software suite, specifically designed to analyze surface energy heterogeneity, isotherm properties and related physical termodynamic parameters. Further, bulk solid property experiments resulting from probe-bulk interactions 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 schematic

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. Excellent for particulates - repeatable, no-hysteresis or roughness effects. Surface energy heterogeneity. Can measure vapor adsorption isotherms as well as surface area.	Ok for flat surfaces. Not well suitable for particulates - slow and poor data statistics. Theory for determining surface energy can be complex.	Excellent for flat surfaces. Not suitable for particulates - swelling, hysteresis, dissolution, surface roughness. Very few solutes possible.	Excellent for flat surfaces. Not suitable for particulates - swelling, hysteresis, dissolution, surface roughness. Very few solutes possible.

Surface Energy Heterogeneity Profiling

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

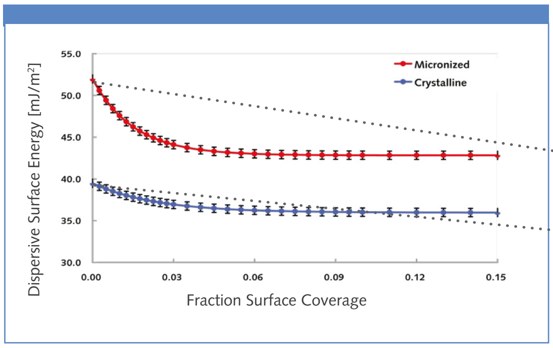


Figure 1. Dispersive Surface Energy **Profiles** Budesonide Samples

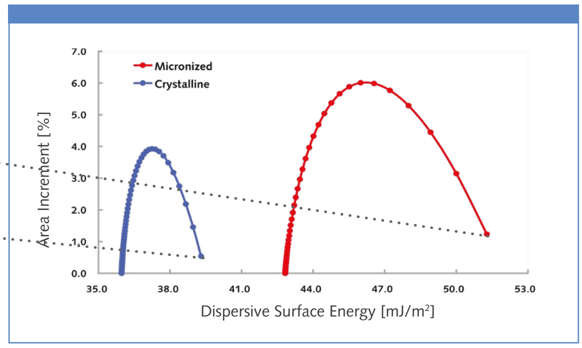


Figure 2. Dispersive Surface Energy **Distributions** Budesonide Samples

Surface Energy Reproducibility

When determining the dispersive component of the surface energy at infinite dilution on 4 columns with 10 replicas on each column, an average standard deviation of less than 0.8% is observed.

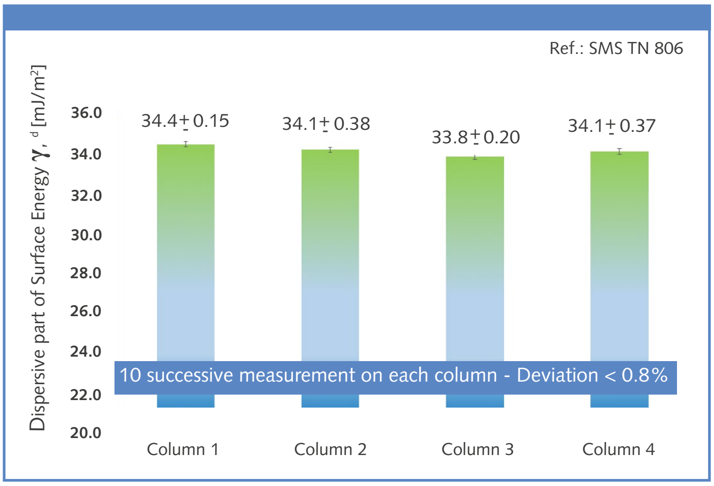


Figure 3. Technical performance of iGC-SEA on commercial Paracetamol (Sigma-Aldrich)

Adsorption Isotherms, Heats of Adsorption and Henry Constants

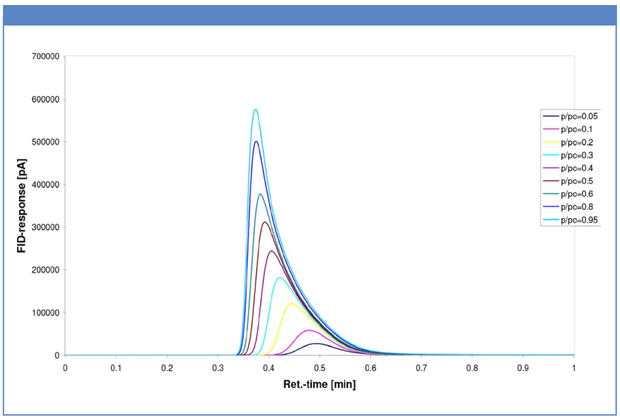


Figure 4. Series of pulses for a multiple injection experiment (variable concentration) on *M745 with hexane at 303 K

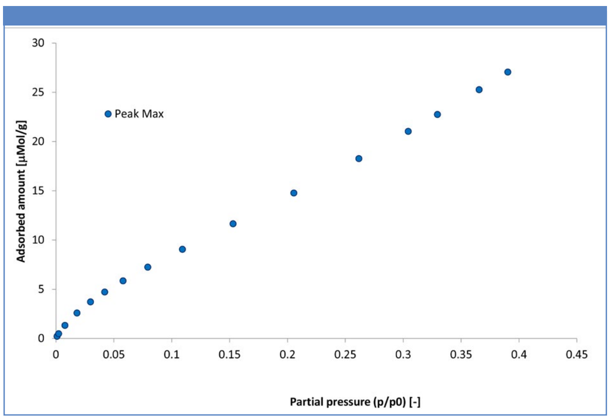


Figure 5. Sorption isotherms of hexane by pulse injections on *M745

*The M745 is an **α**-alumina, which is used as a certified reference material. Reference: SMS Application Note 208.

DPI Formulation Case Study

The surface energy derived Cohesion-Adhesion balance (CAB) can be used to predict blending performance. As shown, the CAB model can effectively predict the interactive powder mixing behavior of small particles along with the compatibility/ flow behaviour of resultant interactive mixtures at certain excipient proportions.

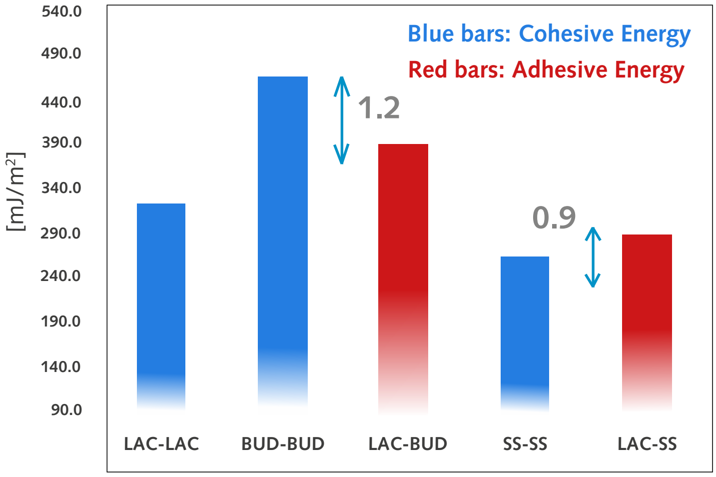


Figure 6. Work of Cohesion and Adhesion

Table 1.

	W_{coh}	W_{adh}	CAB with lactose
BUD	490	400	$W_{coh} > W_{adh}$
SS	270	290	$W_{coh} \leq W_{adh}$

Table 2.

Formulation	Content uniformity RSD (%)
SS+LAC	4.2
BUD+LAC	28.1

(Data by R.Price, Univ. of Bath, UK)

LAC: Lactose, BUD: Budesonide, SS: Salbutamol Sulfate

Similar study: "Applying surface energy derived cohesive–adhesive balance model in predicting the mixing, flow and compaction behaviour of interactive mixtures" (European Journal of Pharmaceutics and Biopharmaceutics 104 (2016) 110–116).

Flowability & Distribution Case Study

Total surface energy distribution of milk powders (Figure 7) and their flowability (Table 3). Energetically more homogenous powders show better flowing behavior. Demineralised whey (◆), Infant Milk Formula (■), Phosphocasein (△) and Skimmed milk powder (○).

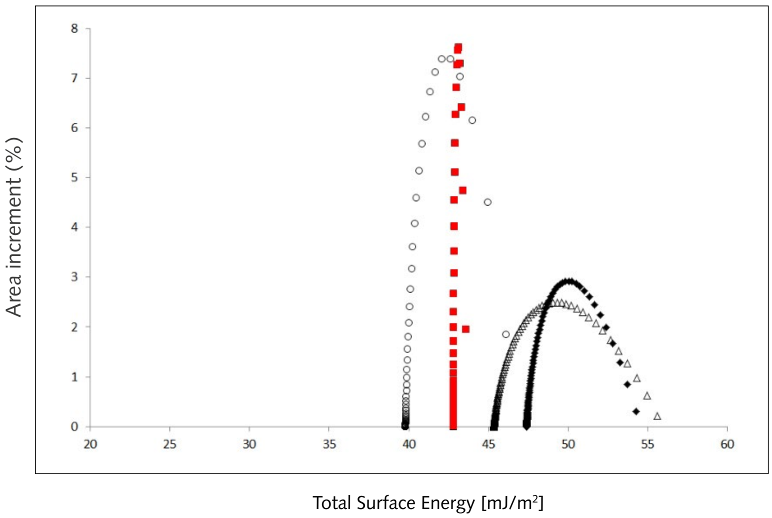


Figure 7. Total surface energy distribution of milk powder*

Table 3.*

	GEA TEST [s] Mean±SD	Brookfield Flow Tester: Flow function 1/slope Mean±SD	Brookfield Flow Tester: Classification
DMW	23[a] ± 2.8	4.93[a] ± 0.26	Easy-flowing
IMF	23[a] ± 0.7	10.50[b] ± 1.29	Free-flowing
PCN	103[b] ± 8.5	4.15[a] ± 0.25	Easy-flowing
SMP	21[a] ± 0.7	9.19[b] ± 0.19	Easy-flowing

DMW: Demineralised whey powder, IMF: Infant milk formula powder, PCN: Phosphocasein powder, SMP: Skim milk powder. *GEA Powder Flow Method A23a (1978).*

*Reference: "Relationships between surface energy analysis and functional characteristics of dairy powders" (Food Chemistry 237 (2017) 1155–1162).

Specific Surface Area Analysis Case Study

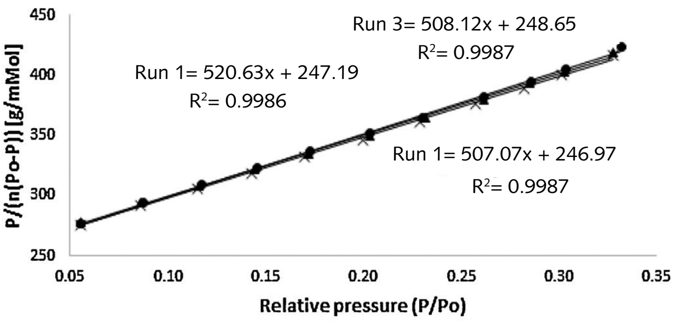


Figure 8. Linearised BET analysis for 3 experiment runs*

Table 4.*

Commercial natural fibres studied

Sample	Variety	Fibre processing
Cellulose	BioMid® (ENC International, South Korea)	Dry jet-wet spinning process
Kenaf	KK60 (Thailand)	Water retting
Flax	Linseed flax (Canadian)	Mechanical decortication by scrutching

Table 5.*

Reproducibility BET experiment.

BET specific surface area (Octane) (m^2g^{-1}) at 30 °C and 0% RH					
Specimen	Run 1	Run 2	Run 3	Mean	Std (%)
BioMid®	0.546	0.545	0.543	0.545	0.1
Kenaf	0.503	0.494	0.501	0.500	0.5
Flax	1.373	1.423	1.440	1.412	3.5

*Reference: "Inverse gas chromatography for natural fibre characterisation: Identification of the critical parameters to determine the Brunauer–Emmett–Teller specific surface area" (Journal of Chromatography A, 1425 (2015) 273–279).

Application Notes

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

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

226 Surface Energetic Heterogenienty of Carbon-based Nanomaterials

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

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

Publications & Case Studies

"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)*

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

Duralliu A, Matejtschuk P, Williams DR, 2019, "Measuring the specific surface area (SSA) of freeze-dried biologics using inverse gas chromatography" *(European Journal of Pharmaceutics and Biopharmaceutics, Vol: 142, Pages: 216-221)*

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

iGC-SEA Hardware

460 mm (W) x 530 mm (D) x 650 mm (H)

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

Sample column oven:
20 ^{o}C to 150 ^{o}C

H_2 & Organic Vapor Leak Detector

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

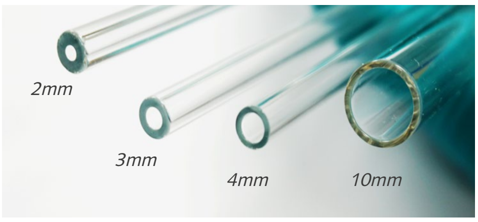
Flame Ionization Detector (FID):
Adjustable gain

Propose built and fully integrated iGC

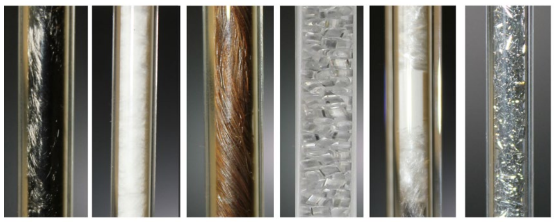
Use of Nitrogen or Helium as carrier gas

Sequential 2-sample column design

Glass columns' internal diameter



Typical samples packed in iGC columns



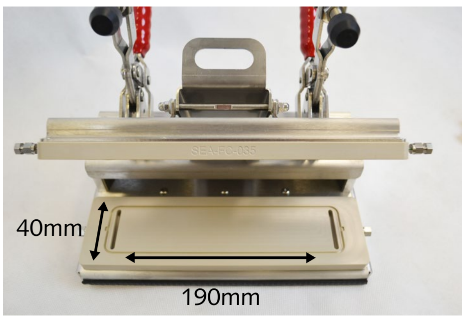
carbon fiber | *cotton* | *hair* | *granules* | *powder* | *medical metal implants*

Optional:
Film/monolithic sample holder

Measuring area:
40 x 190 x .01mm

Background humidity control

Film cell (L) and with paper as sample material (R)



Instrument Platform

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. SEA Analysis enables routine system operation and data analysis mimimizing operator interaction time.

Standard features include:

- Isotherm determination/BET/Henry constant
- Surface energy analysis
- Surface heterogeneity mapping
- Acid-base chemistry analysis

Advanced features include:

- Glass Transition Temperature at Background Humidity
- Hildebrand and Hansen solubility parameters
- Work of adhesion/cohesion determination
- Heats of adsorption/sorption measurement

Supports CFR 21 Part 11

Why Surface Measurement Systems?

- Producer of world's first and only commercial iGC instrument
- Invented the DVS Technology with over 25 years of continuous innovation
- Every instrument is built upon the knowledge and experience of our industry leading sorption scientists
- Our service team provides uncompromising support to our customers and partners
- Outstanding instrument performance
- Most complete and intuitive Windows® software for experimental control and analysis
- Industries benefitting from iGC-SEA: Aerospace, Building Materials, Personal Care, Chemical, Pharmaceutical, Energy, Food, Composite Materials among others.
- Winner of Innovation Award 2018 and ISO 9001:2015 Compliance

Surface Measurement Systems
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QAS INTERNATIONAL ISO 9001:2015 REGISTERED COMPANY Certificate No. CA10727

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