

# Inverse Gas Chromatography Surface Energy Analyzer

Purpose-built iGC instrumentation for advanced materials characterization

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## Surface Energy

Particulate solids, such as powders, fibers, and films, often exhibit issues during manufacturing, usage, and storage in various industrial sectors. To gain an understanding of the key factors that control their behavior and performance, surface energy  $\gamma$  is emerging as one of the most crucial solid material properties.

Discover how your materials are affected by:

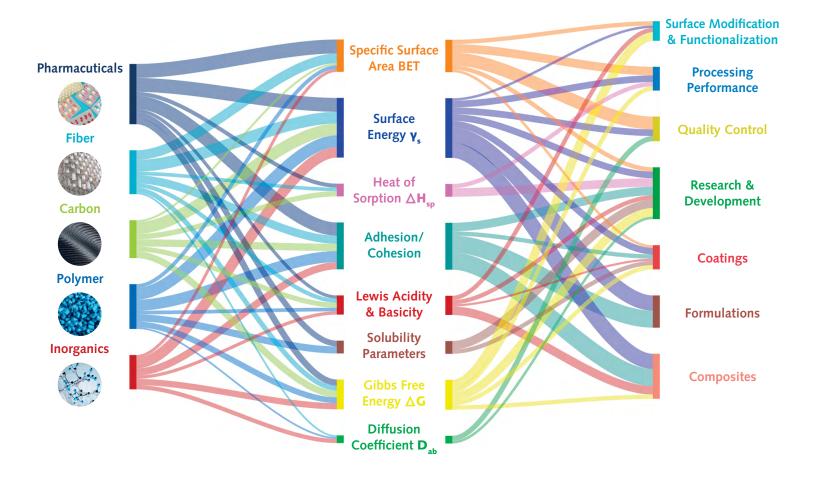


Surface energy  $\gamma$  is a measure of attractive intermolecular forces on a solid surface, similar to the surface tension of a liquid.

These intermolecular forces are responsible for the attraction between powder particles and other solids, liquids, and vapor molecules and can arise from long-range Van der Waals forces (dispersion forces) and shorter-range chemical forces (polar forces).

Surface energy values (dispersive and polar) are linked to several key solid properties, including wetting, dispersibility, powder flowability, agglomeration, process-induced disorder, adhesion/ cohesion, static charge, adsorption capacity, and surface chemistry.

But how do we determine the actual surface energy of a specific solid material? This is where the iGC-SEA comes into play. This powerful instrument employs Inverse Gas Chromatography (iGC) to determine Surface Energy with exceptional detail and accuracy via gas phase adsorption.



What is iGC?

**Inverse Gas Chromatography (IGC)** is a technique used to characterize the surface and bulk properties of materials by analyzing the interactions between a solid sample and various gases.

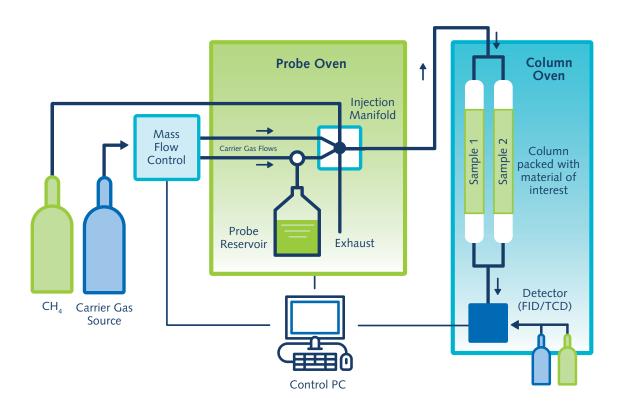
#### The Key to Understanding Surface Properties

### What is the **iGC-SEA**?

The iGC-SEA, or Inverse Gas Chromatography-Surface Energy Analyzer, is an instrument that operates on the principles of iGC. Inverse Gas Chromatography is a gas-solid technique used to characterize the surface and bulk properties of solid materials (For more details, see page 10). Due to its reliability and precision, IGC has become the preferred method for surface energy characterization in laboratories worldwide for particulate materials.

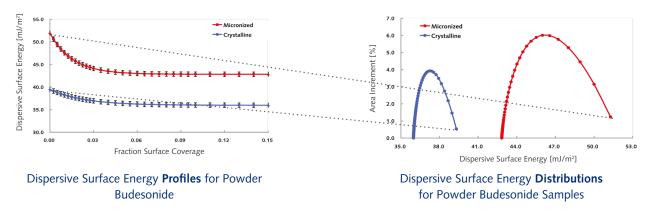
At the core of the iGC-SEA's innovation is the patented gas injection manifold system. This system generates accurate solute pulse sizes across a wide concentration range, producing isotherms at both high and low sample surface coverages. enables precise determination of surface energy heterogeneity distributions.

The iGC-SEA offers a humidity control option, allowing the assessment of humidity and temperature's impact on the physicochemical properties of solids, including moisture-induced Tg, BET specific surface area, surface energy, wettability, adhesion, and cohesion. The system can also conduct bulk solid property experiments employing probe-bulk interactions and solubility theory. Equipped with a purpose-built data analysis software suite, the iGC-SEA stands as an unparalleled instrument, providing a broad spectrum of accurate and reliable surface and bulk property measurements.



#### **iGC-SEA** schematic

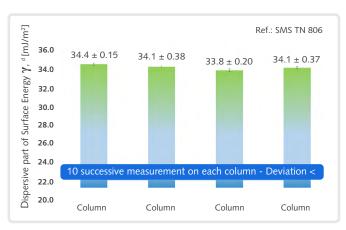
#### Surface Energy Heterogeneity Profiling



Analyzing surface energy profiles involves determining both dispersive and polar components as a function of the fractional surface coverage of the packed material. 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.

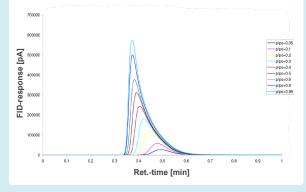
#### **Reliable SE Reproducibility**

The unique features of the iGC-SEA instrument enable unparalleled reproducibility in Surface Energy determination. An average standard deviation of less than 0.8% is observed on 10 successive measurements across 4 columns.

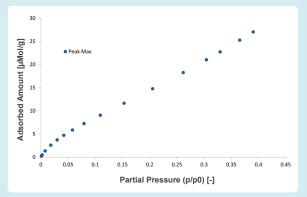


Technical performance of iGC-SEA on commercial Paracetamol (Sigma-Aldrich)

#### Adsorption Isotherms, Heat of Adsorption and 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

## Instrument Platform iGC-SEA Hardware

- Patented integrated gas phase injection systems with humidity generation
- ✓ Sample Volume:
  0 23.56 cm<sup>3</sup>

- 12 interchangeable reservoirs: Easy access drawers
- Sequential twin-sample
   column design
- ✓ 50 solvents database built-inability to add more
- \* Temp range dependent on instrument variant





- ✓ Sample column oven: 10 °C to 500 °C \*
- Flame Ionization Detector (FID): Adjustable gain
- ✓ Use of Nitrogen or Helium as carrier gas
- Purpose-built and fully integrated iGC
- ✓ H<sub>2</sub> & Organic
  Vapor Leak
  Detector

## Sample Columns



#### Film Cell Holder

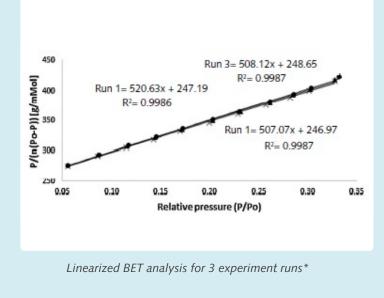


### **iGC-SEA** Advanced



- Analyzes surface energy and specific surface area for fine powders, fibers, and non-porous particulate solids.
- Fully-automated iGC system
- Patented headspace injection system with humidity generator
- User-friendly control & analysis software with CFR 21 Part 11 capabilities

#### Surface Area Analysis Case Study

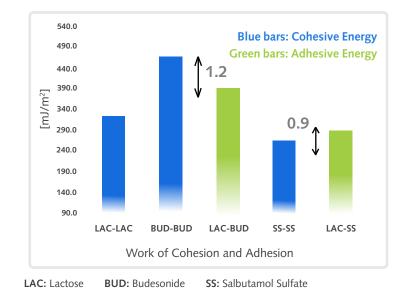


Sample	Variety		Fibre processing			
Cellulose	BioMid®	Dry jet-wet spinning				
		International, South Korea) KK60 (Thailand)		process Water retting		
Kenaf	KK60 (Th					
	Linseed flax		Mechanical decortication			
Flax	Linseed fl	ax				
Flax eproducibility BE	(Canadia			scrutching		
eproducibility BE	(Canadia	n)	by	scrutching		
eproducibility BE	(Canadia T experiment.	n)	by	scrutching		
eproducibility BE BET specific surfa	(Canadia T experiment. ace area (Octane)	n) (m²g <sup>-1</sup> ) at 3	by and 0	scrutching 0% RH		
eproducibility BE BET specific surfa Specimen	(Canadia T experiment. ace area (Octane) Run 1	n) (m <sup>2</sup> g <sup>-1</sup> ) at 3 Run 2	by s 30 °C and 0 Run 3	scrutching )% RH Mean	Std (%)	

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

#### **Predict Blending Performance for DPI Formulations**

The surface energy derived Cohesion-Adhesion Energy 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 behavior of resultant interactive mixtures at certain excipient proportions.



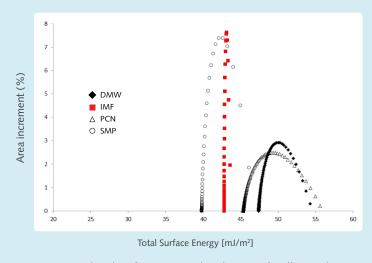
	$W_{coh}$	$W_{adh}$	CAB with lactose		
BUD	490	400	$W_{coh} > W_{adh}$		
SS	270	290	$\rm W_{coh} \leq \rm W_{adh}$		
Formulation		Content uniformity RSD (%)			
SS+LAC		4.2			
BUD-	⊦LAC	28.1			

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

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

#### **Investigating Flow Behavior of Powders**

The iGC-SEA is a valuable tool, providing insight on powder flowability using surface energy analysis. More energetically homogeneous powders (narrow energy curves, see graph below) display improved flow behavior. A standard technique for measuring powder flowability corroborates the IGC analysis findings, showing that Infant Milk Formula (**■**) has better flowability.



**Brookfield Flow Tester** Flow function Classification GEA TEST 1/slope [s] Mean± SD Mean± SD DMW Easy-flowing  $23^{a} \pm 2.8$ 4.93<sup>a</sup> ± 0.26 Free-flowing IMF  $23^{a} \pm 0.7$ 10.50<sup>b</sup> ± 1.29 PCN  $103^{b} \pm 8.5$  $4.15^{a} \pm 0.25$ Easy-flowing SMP  $21^{a} \pm 0.7$ 9.19<sup>b</sup> ± 0.19 Easy-flowing

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

Normalized surface energy distribution of milk powder\*

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

## **iGC-SEA** Nova



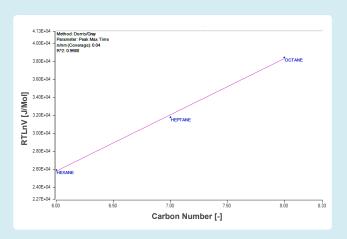
- New high temperature oven: 30 °C 500 °C
- *In-situ* preconditioning and surface energy analysis within a single instrument
- Fully-automated iGC system
- Patented head space injection system with humidity generator
- User-friendly control & analysis software with CFR 21 Part 11 capabilities

#### Carbon Black Surface Characterization for Energy Storage

Vulcan XC 72 is a carbon black with high electrical conductivity for diverse applications, such as batteries, fuel cells, conductive paper, and catalyst support. This material provides exceptional conductivity even at low loading levels and plays a crucial role as a support in anode and cathode electrodes, particularly in polymer electrolyte membrane fuel cells (PEMFC). BET SSA: 128.8 m<sup>2</sup>/g.

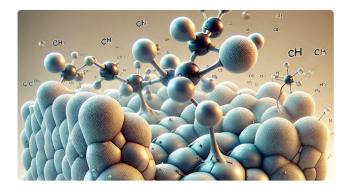


	Humidity: 0%RH		
Results:	Sample mass: 8mg		
Experiment Temperature:	523K	573K	
Dispersive Surface Energy (y <sub>d</sub> <sup>50</sup> ):	139 mJ/m <sup>2</sup>	240.1 mJ/m <sup>2</sup>	

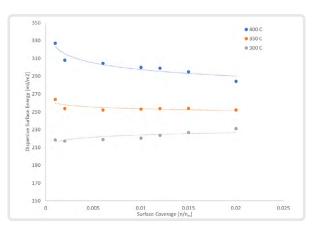


Dorris-Gray Method

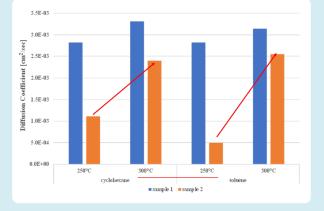
#### **Dispersive Surface Energy of Zeolite 13x**



The yd surface energy of Zeolite 13X at 300°C and 350°C reveals homogeneity, with a consistent and uniform surface energy distribution. However, when the temperature rises to 400 °C, some degree of heterogeneity begin to appear.



Dispersive Surface Energy of Zeolite 13x

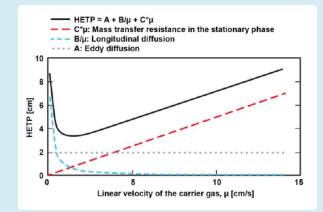


#### High Temp Diffusion Coefficient

Diffusion and mass transfer are critical in the design and optimization of catalytic processes, operations processing of materials, and in adsorptive separation. The van Deemter model is a continuation of the plate theory and involves the dynamic response of HETP as a function of the average linear velocity of the carrier gas (see Figure 1), distinguishing the three types of diffusion types: eddy diffusion, longitudinal diffusion, and mass transfer resistance.

Diffusion of Microporous Catalysts

Comparing the changes in diffusion coefficients as a function of temperature, the values are higher at 300°C than 250°C for both vapors on both samples. This is expected since higher temperatures lead to faster diffusion rates. When comparing the cyclohexane and toluene results on the same sample, the cyclohexane diffusion coefficients are higher for both samples and temperatures.

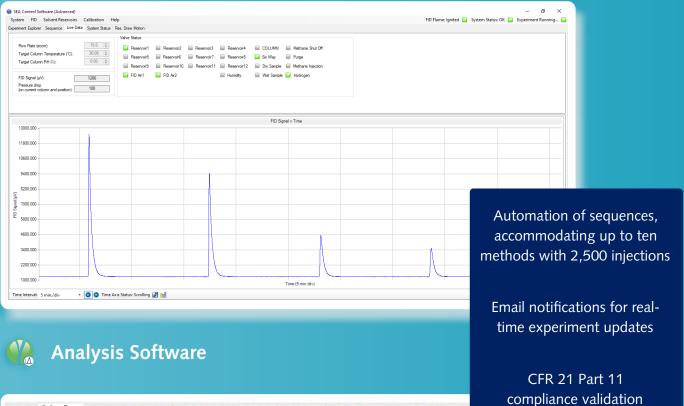


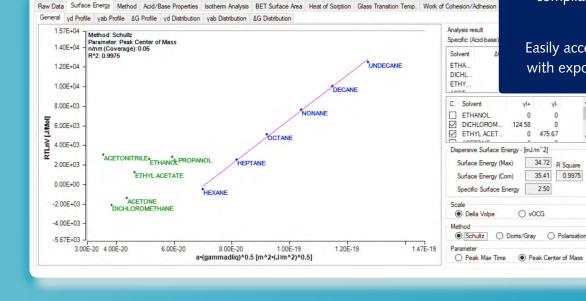
Van Deemter Plot: The 3 Components of the van Deemter Equation

Our proprietary software includes a seamless system control software and robust data analysis software featuring advanced tools tailored for research-focused users.



#### **Control Software**





Easily accessible raw data with export functionality

vl-

0

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35.41 0.9975

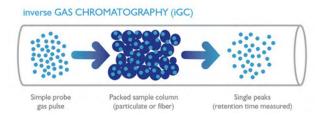
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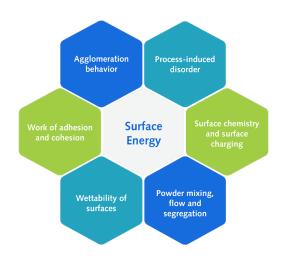
0 475.67

#### The Inverse Gas Chromatography Technique

The iGC-SEA is a purpose-built Inverse Gas Chromatography (IGC) system designed to measure surface energy and various physiochemical properties. In this process, the sample is packed into a column and positioned within a controlled environment where temperature and humidity are regulated. During an IGC experiment, different vapor probe molecules are introduced to account for both the dispersive (non-polar) and acid-base (polar) components, allowing the determination of intermolecular force strength during vapor-sample interactions.

Once the surface energy of a solid has been determined, these values can be correlated to several key solid properties, including wetting, dispersibility, powder flowability, agglomeration, process-induced disorder, adhesion/ cohesion, static charge, adsorption capacity, and surface chemistry.





#### Surface Measurement Systems, Your iGC Specialists

- · We are the producers of world's first purpose-built IGC instrument
- We are pioneers in vapor sorption instrumentation with over 30 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.
- We ensure outstanding instrument performance.
- The iGC-SEA is accompanied by a complete Windows® software for experimental control and analysis.
- Industries using iGC-SEA: building materials, personal care, chemical, pharmaceutical, energy, food, and more.





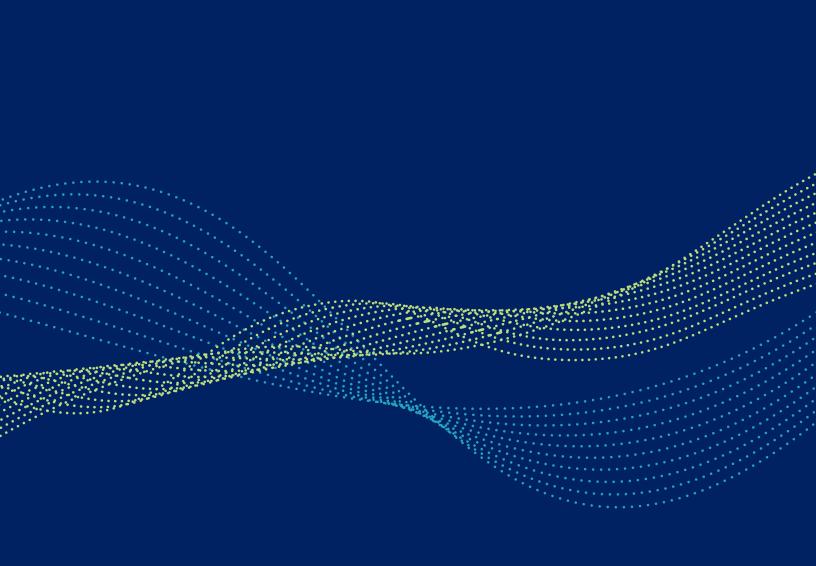
WINNER Innovation Award FUTURE

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