Adscientis
Smart Inverse Chromatography

Sized or unsized carbon fibres: what are the differences in terms of surface properties?

Focus on IGC applications

Introduction

Surface properties of carbon fibers are crucial for high performing composites. Dynamic adsorption of multiple gaseous probe molecules (Inverse Gas Chromatography) can characterize these properties most sensitively.

In practice, the provided datas guide product developers, production chemists and quality controllers towards optimization of products and processes. Indeed, IGC is a unique method to understand and quantify surface treatments and resulting interface effects between fibers and matrix.

This short application note focuses on carbon fibers, completely sized, unsized and partly sized ones and on the less-known concept of determining surface morphology and mobility.

Key points

- Carbon fibers, sized (A), partly sized (B) and unsized (C) are investigated using IGC under ideal infinite dilution (IGC-ID).
- A n-alkanes homologous series provides dispersive component of the surface energy (γ_s^d) [1].
- Cyclic and branched alkanes (cyclooctane, isooctane) provide quantitative information about nanoroughness and soft features thanks to the IM and RIM concepts [2].

Results

Fig.1

- The n-alkanes series provides nice straight lines ($r^2 \geq 0.9999$) inducing accurate surface energy determinations.
- The steep slope of unsized sample C indicates a stronger interaction with n-alkanes expressed as a high disperse surface energy of 78.4 mJ/m^2.
- Isooctane (A, B, C) and cyclooctane (A, B) are below the n-alkanes indicating a size or compatibility exclusion effects.
- Cyclooctane interacts stronglier with the sized sample A than the n-alkanes (ΔGa above n-alkanes).
- Such relative positioning of isooctane and cyclooctane (A) is typical for soft materials (liquids, coating, sizing, polymer above Tg…).

Sample	Slope [kJ/mol]	γ_s^d [mJ/m^2]	r^2
A	2.43 ± 0.01	32.3 ± 1.3	1.0000
B	2.71 ± 0.01	40.3 ± 1.7	1.0000
C	3.79 ± 0.03	78.4 ± 4.2	0.9999

Fig.2

- Figure 2 is a typical application of the IM and RIM concept.
- Sample A is in the area of soft surfaces (yellow domain). The molecular probes detect the sizing.
- Sample B is in the green domain corresponding to surfaces having hard and soft surface parts. The molecular probes are detecting both hard (fiber) and soft (sizing) surfaces.
- Sample C is in the blue domain corresponding to hard and clean surfaces. The molecular probes are only detecting size exclusion effects.

References:

*[1]. « Adsorption of n-alkanes at zero surface coverage on cellulose paper and wood fibers »; G. M. Dorris, D. G. Gray; Journal of Colloid and Interface Science **77**(1980), 353-362.*

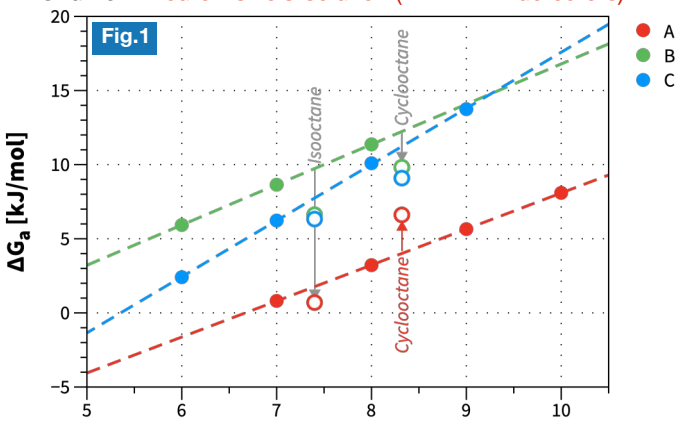
[2]. IM and RIM concept note Download the IM and RIM concept note (pdf)

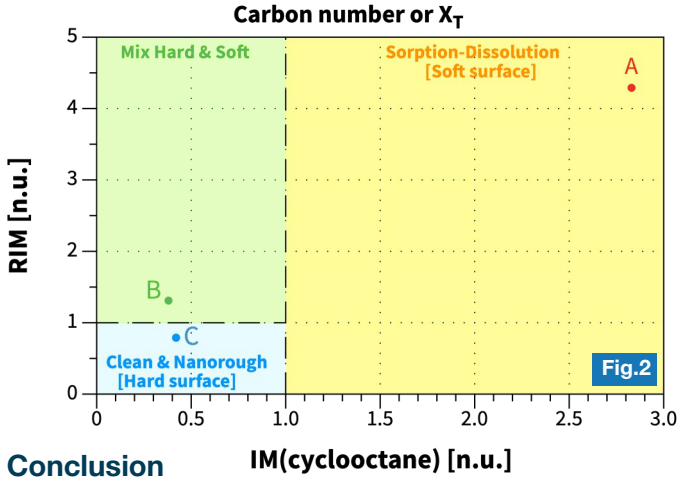
[3]. NeuronIC brochure Download the neuronIC brochure (pdf)

Operating conditions

neuronIC

Samples: Carbon fibers [3]
Sample weights: between 1.6 and 3.0g (sample dependent)
Measurement temperature: 30°C
Dead time determination: methane
Probes: n-hexane, n-heptane, n-octane, n-nonane,n-decane, isooctane and cyclooctane (Polar probes: not used here)
Injected probe amounts (vapours): some nmoles (ID)
Carrier gas: Helium at 10.0 mL/min
Instrument: neuronIC IGC solution (with 2 FID detectors)





Conclusion

Three different carbon fiber samples are investigated with IGC-ID. These measurements were performed using a representative large bundle of carbon fibers packed in a column, and as such, these results are statistically more meaningful than single fiber wetting measurements. Moreover, IGC-ID measurements are relatively easy and fast via a dual-channel IGC equipped with a fully-automated Adscientis neuronIC Intelligent IGC system [3].

This system can easily and accurately differentiate sized (A), partly sized (B) and unsized (C) carbon fibers. Indeed, the determined γ_s^d values showed meaningful and significant differences, ranging from a low of 32.3 (A) to 78.4 mJ/m^2 (C). Moreover, the IM (Morphology Index) and RIM (Relative Morphology Index) concept clearly sets the fibers samples in the correct domain with A (sized) in the soft surface domain, B (partly sized) in the mix domain and C (unsized) in the clean and nanorough domain.

This Application Note offers a good example of the utility of the IGC-ID technique in accessing changes in the surface energetics of treated and sized carbon fiber surfaces which can be carefully monitored and optimized to insure desired polymer matrix adhesion related performance specifications.