

Effect of Particle Size Reduction on the Surface Energy of Sucrose

Codie Freeman | Jack Aston

Introduction

Surface energy governs powder behaviours including interparticle adhesion, flow, and caking tendency, directly influencing processability in pharmaceutical manufacturing.

Processes such as milling lead to particle size reduction, exposing fracture surfaces and generally increase **Brunauer–Emmett–Teller Specific Surface Area (BET SSA)**; this is also expected to increase **surface energy**. However, the mechanism of size reduction may independently alter particle morphology, surface characteristics and inter-particle interaction, including distribution of adhered fine particles, potentially confounding this relationship.

Three sucrose samples were produced via **two processing routes**: pestle and mortar grinding and sieving of granulated sucrose to yield Coarse (500–180 μm) and Medium (150–45 μm) fractions and sieving of industrially milled sucrose to yield a Fine fraction (45–30 μm).

Methods

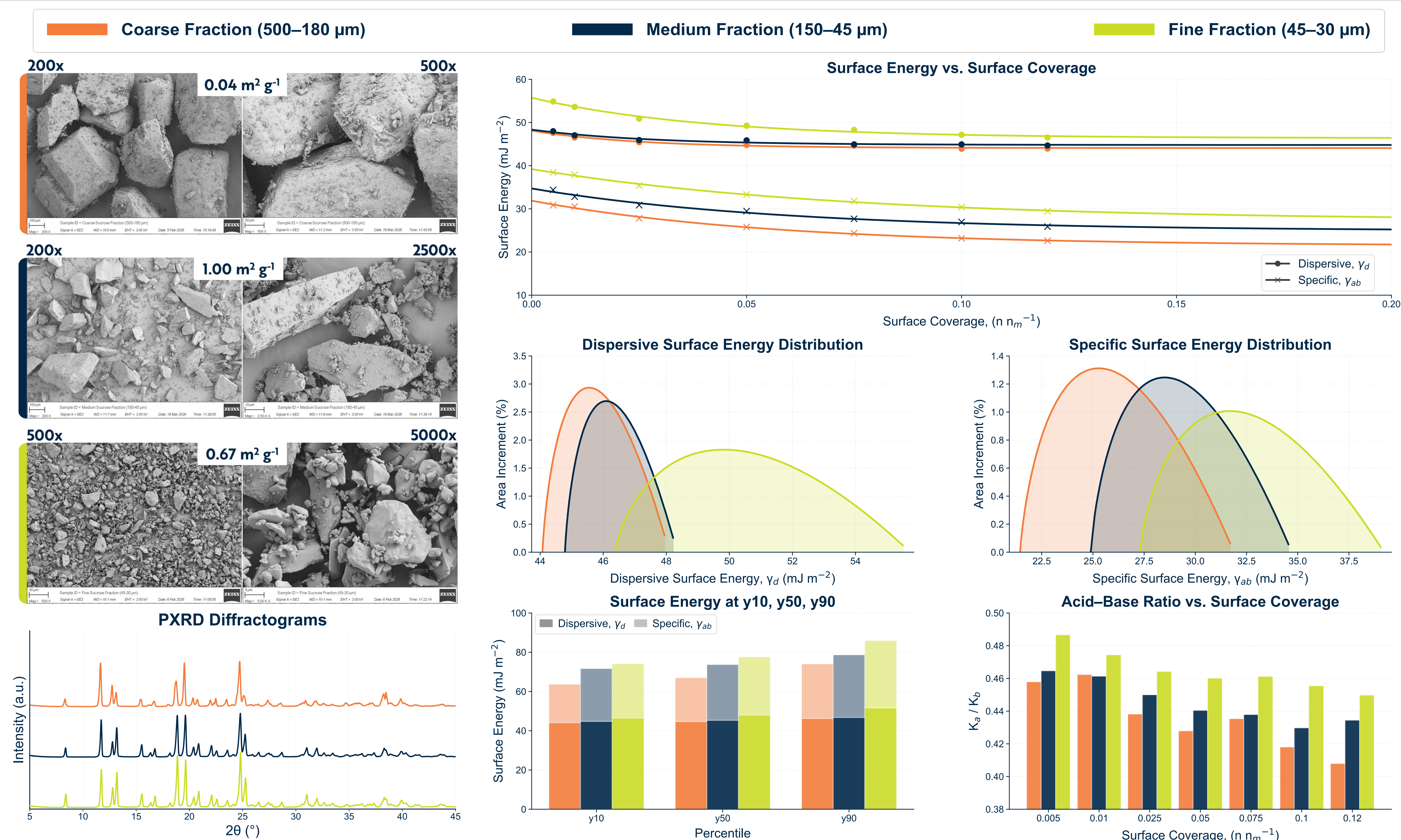
SEM images were generated using a **Zeiss Sigma Field Emission SEM** with a secondary electron (SE2) detector at 3 kV accelerating voltage.

N_2 gas adsorption and PXRD analyses were conducted using a **Micrometrics Tristar 3020** and a **Bruker-AXS D2 Phaser**, respectively.

Surface energy analysis was conducted using a Surface Measurement Systems **Inverse Gas Chromatography – Surface Energy Analyzer**. Sucrose samples were packed into silanised glass columns to a total surface of approximately **0.5 m^2** and conditioned at **30 $^\circ\text{C}$** for 60 minutes under helium carrier gas (**10 sccm**).

Dispersive and specific surface energy heterogeneity profiles were determined using **Dorris-Gray** methodology and **Peak COM** parameter across a range of fractional surface coverages (**0.005–0.12 n nm^{-1}**).

Results



Discussion

BET SSA was expected to increase with decreasing particle size; the Fine fraction deviated from this expectation, attributed to a lesser level of adhered fine particles onto larger particle surfaces and differences in surface texture/roughness observed by SEM, highlighting that **milling method has significant impact beyond particle size alone**.

PXRD **confirmed the consistency of the crystalline phase** of the samples; peak intensity variation could be attributed to preferred orientation, with baseline characteristics of the Fine fraction suggesting possible increased amorphous contribution potentially from industrial milling.

Hand grinding produced comparable γ_d across granulated fractions, whilst having a greater impact on the γ_{ab} . The commercially milled sample exhibited noticeably higher surface energies and greater heterogeneity. This suggests type of **processing route has a greater influence** on dispersive surface energy than sieving and particle size alone.

Acid-base probe analysis exhibited greater basic character across all samples, suggesting sucrose surfaces possess higher concentration of electron-donating functional groups; a decrease in K_a/K_b ratio was observed with increasing surface coverage, suggesting **surface basic character is most pronounced at lower-energy sites**.

Conclusions

Particle size reduction was found to **impact both dispersive and specific surface energies** of sucrose across all samples.

Processing route and milling method were found to **directly impact surface energy values and characteristics** more significantly than particle size alone, with a greater impact observed on the dispersive surface energy; there was no direct trend between BET SSA and surface energies.

Surface energy characterisation of sucrose provides formulation insight beyond particle size alone, with processing-induced changes influencing **interparticle adhesion, blend homogeneity, and compactibility** in pharmaceutical manufacturing.

References

1. Farshchi, A. The effect of surface chemistry on the caking behaviour of sucrose crystals. *J. Food Eng.* 2025, 389, 112386.
2. Karde, V. Investigating sizing induced surface alterations in crystalline powders using surface energy heterogeneity determination. *Powder Technology*, 2022, 395, p645-651.
3. Dorris, C.M. and Gray, D.G. Adsorption of normal-alkanes at zero surface coverage on cellulose paper and wood fibres. *J. Colloid & Interface Sci.* 1980, 77, p353-362.
4. Della Volpe, C. and Siboni, S. Some reflections on acid-base solid surface free energy theories. *J. Colloid & Interface Sci.* 1977, 196, p121-136.