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Surface analysis of functionalized substrates for the nucleation and crystallization of pharmaceutical molecules

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Content

Nucleation represents the core of a variety of natural processes, ranging from ice crystal formation to protein aggregation, which can occur homogeneously or heterogeneously, depending on whether aggregation mechanism involves the presence of single or multiple phases. Recently, the role of external surface features on crystallization of active pharmaceutical ingredients (APIs) is being investigated by relating surface chemistry and morphology to nucleation kinetics and polymorph selection.¹

Surface functionalization using SAMs has been largely investigated and applied to different substrates and constitutes one of the most robust methods available to obtain well controlled functionalized surfaces.^{2,3,4} In the present study, the use of glass substrates functionalized with self-assembly of trimethoxysilanes differing for their head group chemistry for the nucleation and crystallization of small pharmaceutical molecules was investigated. Silane anchoring was achieved via wet chemistry-based route whilst systematic characterisation of morphology and chemistry was carried on in order to determine the influence of different parameters (Figure 1). Assessment of effective functionalization was carried out by means of contact angle and surface Z-potential analyses, whilst the surface chemistry of functionalized glass was probed using XPS and ToF SIMS. Lastly, AFM was adopted for the characterization of surface topography. High-quality monolayers carrying thiol, methacrylate and glycidyl groups were successfully synthesized whereas in the case of amino-terminated silanes surface roughness dramatically increased and correlation between ideal and experimental elemental ratios characterizing the monolayer was not achieved. Nucleation and crystallization of biopharmaceutical molecules was also carried out by studying aspirin and paracetamol crystallization out in a thin-film solution deposited onto SAMs. Crystallization outcome was studied according to kinetic and thermodynamic aspects by optical microscopy, whilst crystal orientation and form were evaluated by means of X-Ray Diffractometry (XRD). Finally, preliminary results on nanostructured surfaces will be also presented.