

# Isolation and rheo-structural analysis of amylose solutions

A route towards amylose wet fiber spinning

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Isolated amylose (AM), is considered as a resource for the fabrication of continuous fibers via a wet spinning procedure. This process involves the extrusion of a suitable AM solution through a spinneret in a coagulating bath to induce AM solidification. The resulting AM fibers could be further processed into carbon fibers (CFs).

The isolation of AM from potato starch entails starch dispersion, optional enzymatic debranching and selective precipitation of AM.<sup>[1-3]</sup> A pressurized autoclave was used for the high-temperature dispersion in 10 wt% DMSO.<sup>[4]</sup> Added microbial pullulanase hydrolyzed the  $\alpha$ -1,6-glycosidic side chains to yield linear AM although this enzyme also tended to hydrolyze the backbone to some extent.<sup>[1,5]</sup> AM was selectively precipitated from solution via the formation of an insoluble inclusion complex with n-butanol.<sup>[1]</sup> The effect of dispersion, debranching and precipitation on the structure and molecular weight of the starch species was monitored by Size-exclusion chromatography (SEC)<sup>[6]</sup> and wide-angle X-ray diffraction (WAXD).

Furthermore, the rheological behavior of different isolated amylose solutions in DMSO; a gift research sample, debranched AM and undebranched AM) was studied in order to assess their 'spinnability', an umbrella term encompassing the rheological properties required for polymer fiber spinning.<sup>[7]</sup> A critical parameter in this context is the overlap concentration  $c^*$ , the regime in which polymer coils are concentrated enough to overlap and above this point entanglements might occur in high-MW solutions. In addition, the shear, oscillation and extensional rheology of AM solutions were characterized under conditions relevant to fiber spinning. Shear thinning and solution elasticity contribute significantly to the stability of the fluid jet exiting the spinneret. Extensional rheology addresses aspects of the required balance between surface tension and extensional viscosity<sup>[7-9]</sup>

Debranching during AM isolation was found to be too detrimental to the AM molecular weight. Hence the resulting solution could not well entangle, leading to a low elasticity. Undebranched samples, on the other hand, retained more of their original MW during isolation leading to more elastic solutions.

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