

L-glutamic acid crystals of pure α form and uniform size distribution from continuous non-seeded reaction crystallization in slug flow

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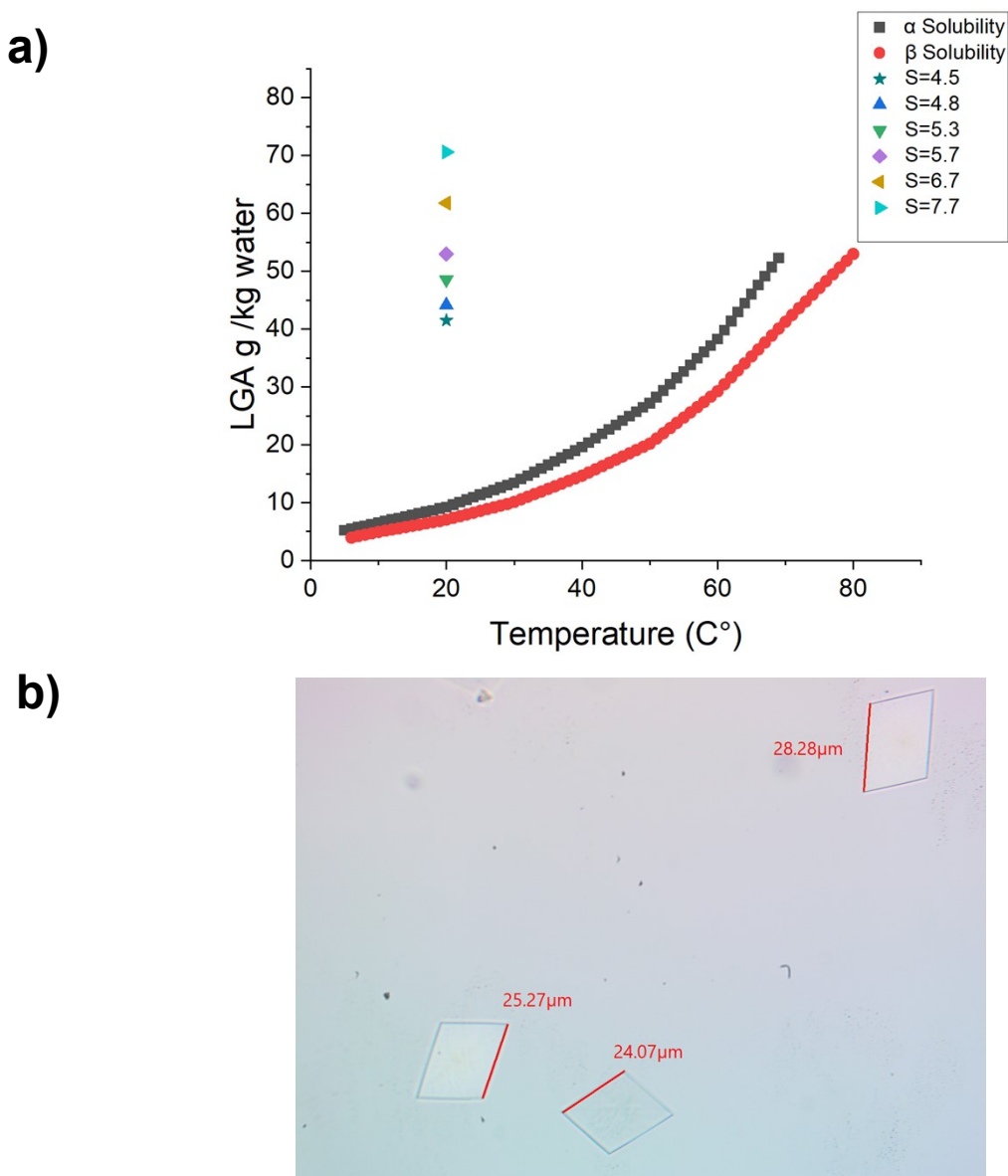


Figure S1 (a) Solubility¹ of α -form and β -form of L-Glutamic acid (LGA) crystals and starting supersaturations (Table 2) designed based on the α -form solubility. (b) Representative microscope images of LGA crystals at an initial supersaturation of 5.7 where the measurement technique of length is indicated.

Figure S2 Representative microscope images of LGA crystals (Table 3), from reactive crystallization in a stirred tank (semi-batch mode) at an initial supersaturation a) 4.5, b) 4.8, c) 5.3, d) 5.7, e) 6.7 and f) 7.7. The conditions are exactly the same as for Figure 2. Two images at two different sampling locations (top & bottom of flask) were shown for each condition, with experimental details in Table 2. The residence time is 12.3 minutes for (a-c), and 5.4 minutes for (d-e).

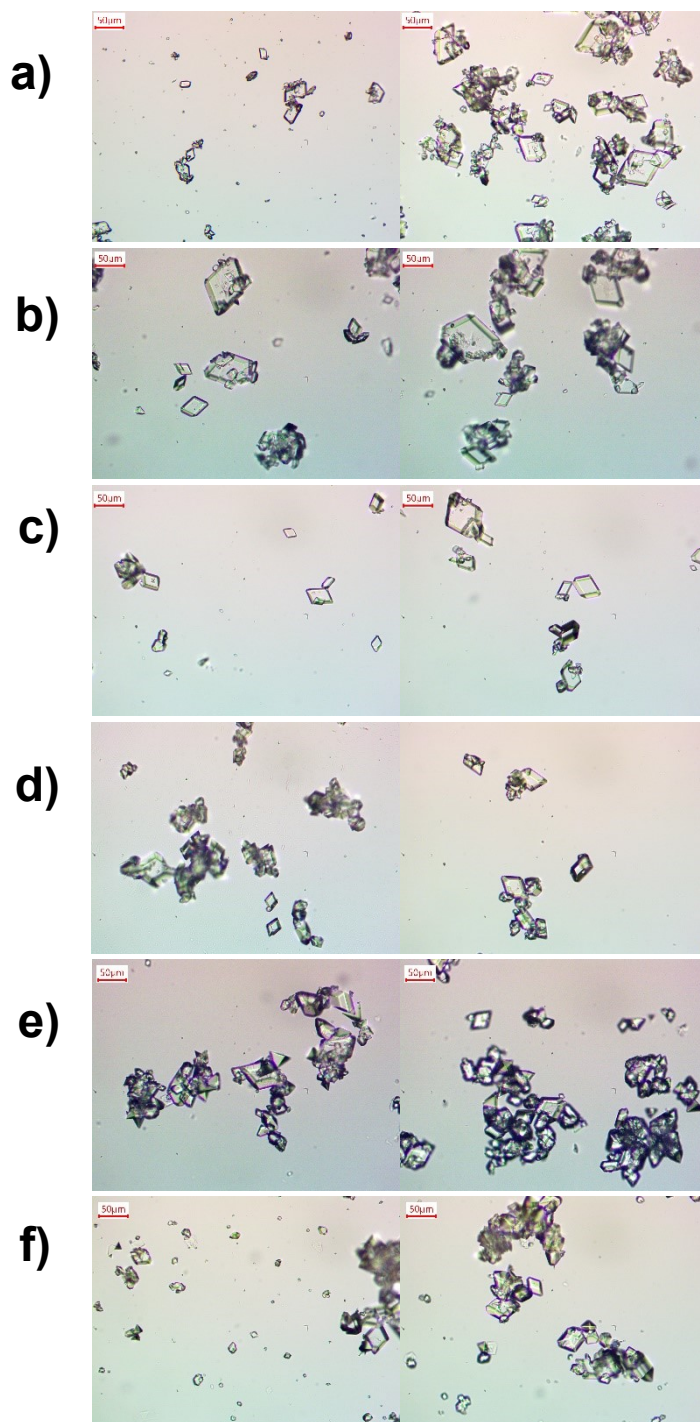
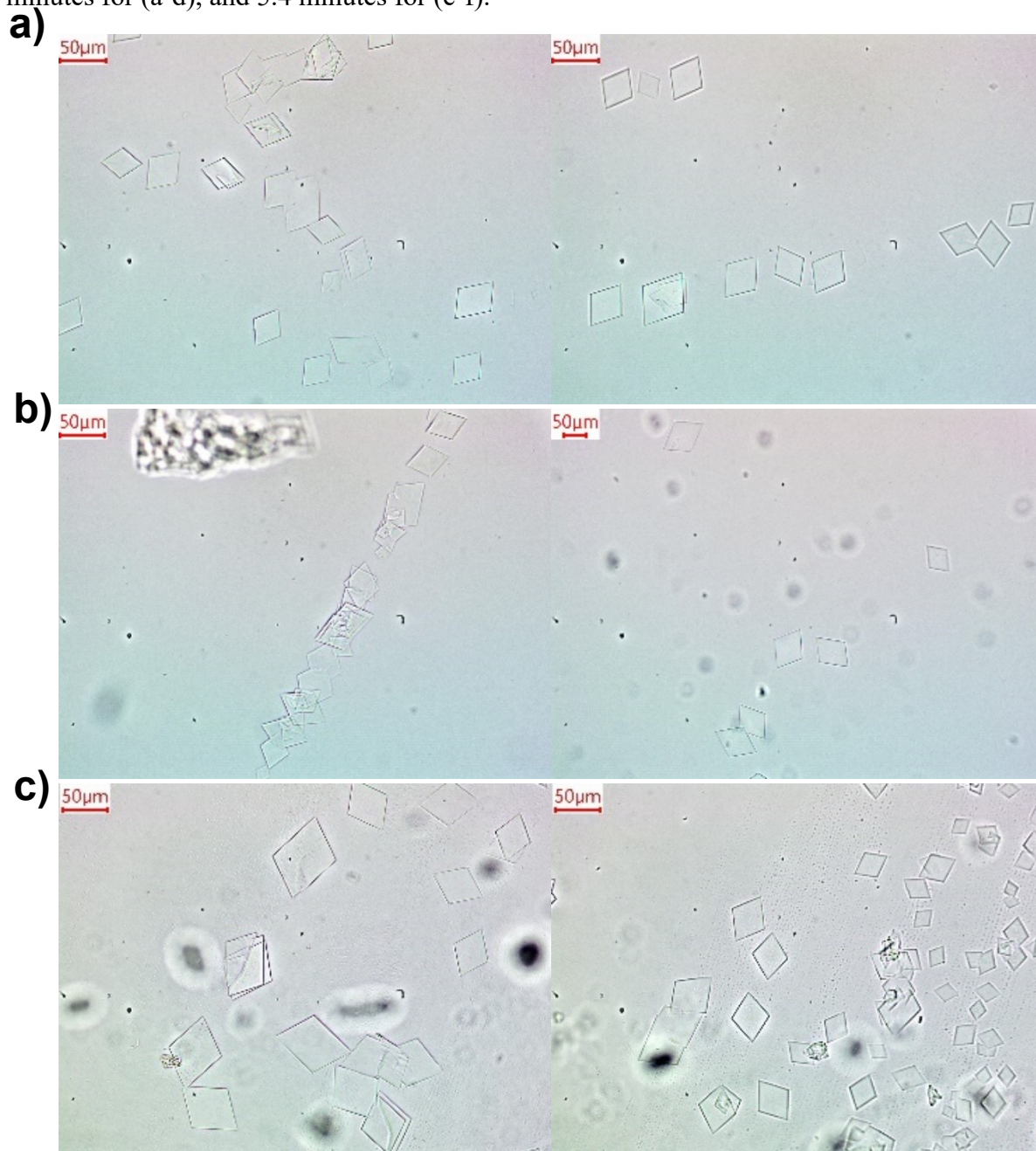


Figure S3 Representative microscope images of α -form LGA crystals (confirmed with XRD data), from reactive crystallization in slug flow at an initial supersaturation of a) 4.5, b) 4.8, c) 5.3, d) 5.7, e) 6.7 and f) 7.7. The conditions are exactly the same as for Figure 3. Two images were shown for each condition, with experimental details in Table 2. The residence time in slug flow is 12.3 minutes for (a-d), and 5.4 minutes for (e-f).



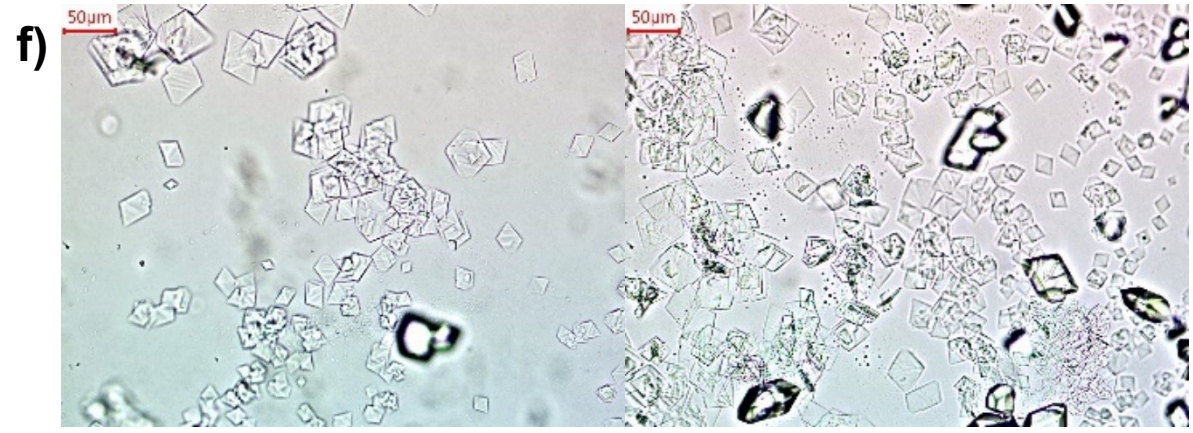
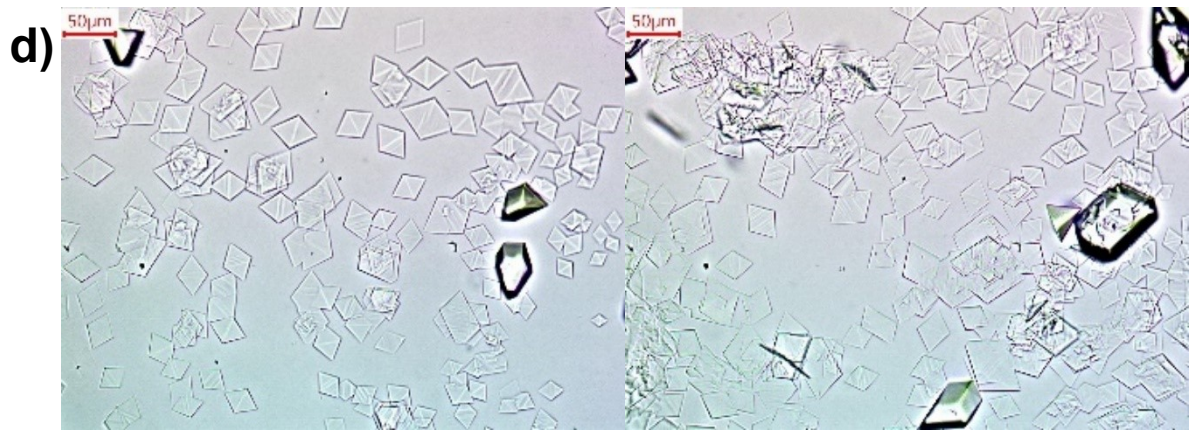


Figure S4 Microscope images of crystals from slug flow reaction/crystallization at an initial supersaturation of a) 4.5, b) 4.8, c) 5.3, d) 5.7, e) 6.7 and f) 7.7 after 50-second of stirring after slug flow. The XRD data for these crystals are in Figure S5.

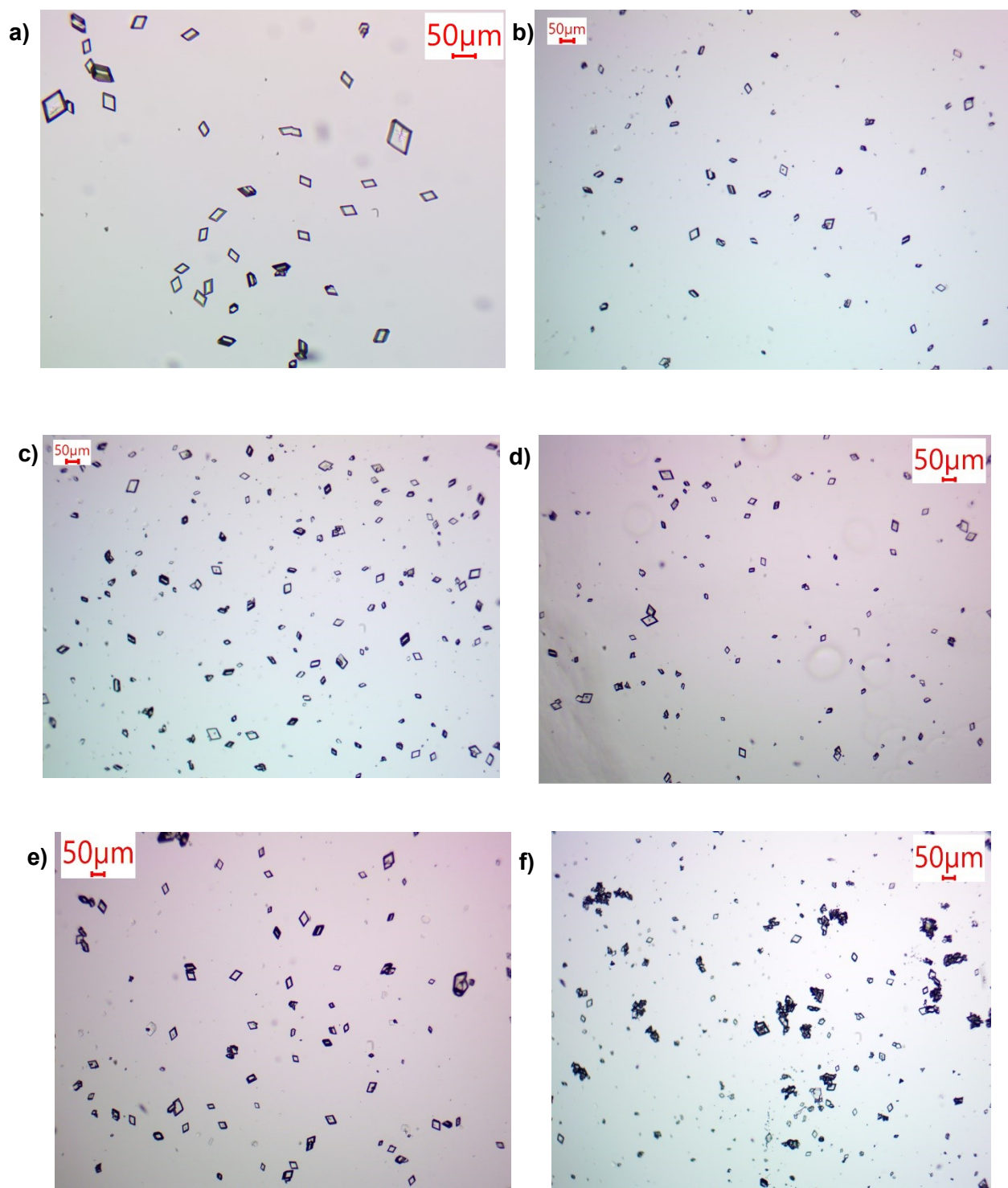


Figure S5 (a) XRD pattern of product crystals from stirred tank, semi-batch mode (Figure 2) at initial supersaturation of 4.5, 4.8, 5.3, 5.7, 6.7 and 7.7 confirming product crystals are pure α form². The crystal structures of α - and β -LGA were obtained from the Cambridge Crystallographic Data Centre (CCDC), with CCDC codes of LGLUAC02 and LGLUAC01 included for comparison.

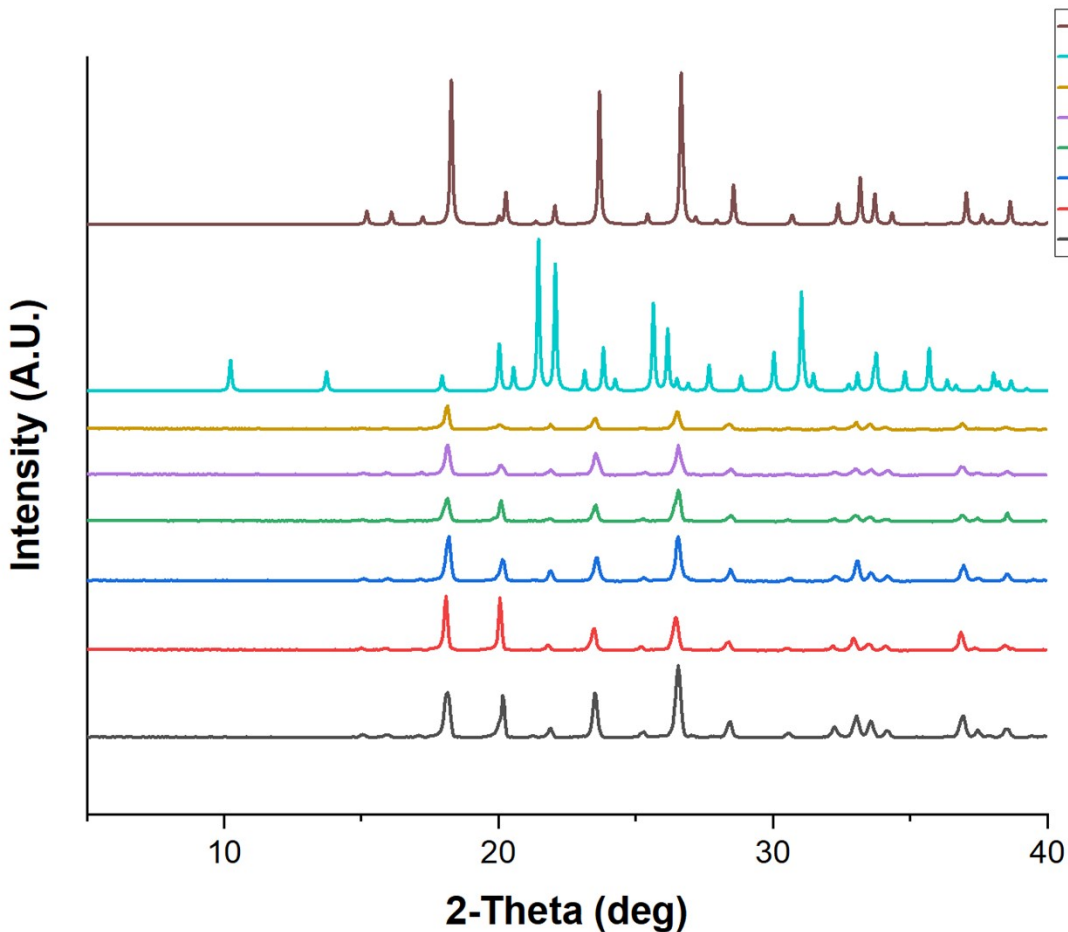


Figure S6 (a) XRD pattern and (b) DSC spectra of product crystals from slug flow (Figure S4) at an initial supersaturation of 4.5, 4.8, 5.3, 5.7, 6.7 and 7.7. Both confirm product crystals are in pure α form². The crystal structures of α - and β -LGA were obtained from the Cambridge Crystallographic Data Centre (CCDC), with CCDC codes of LGLUAC02 and LGLUAC01 included for comparison.

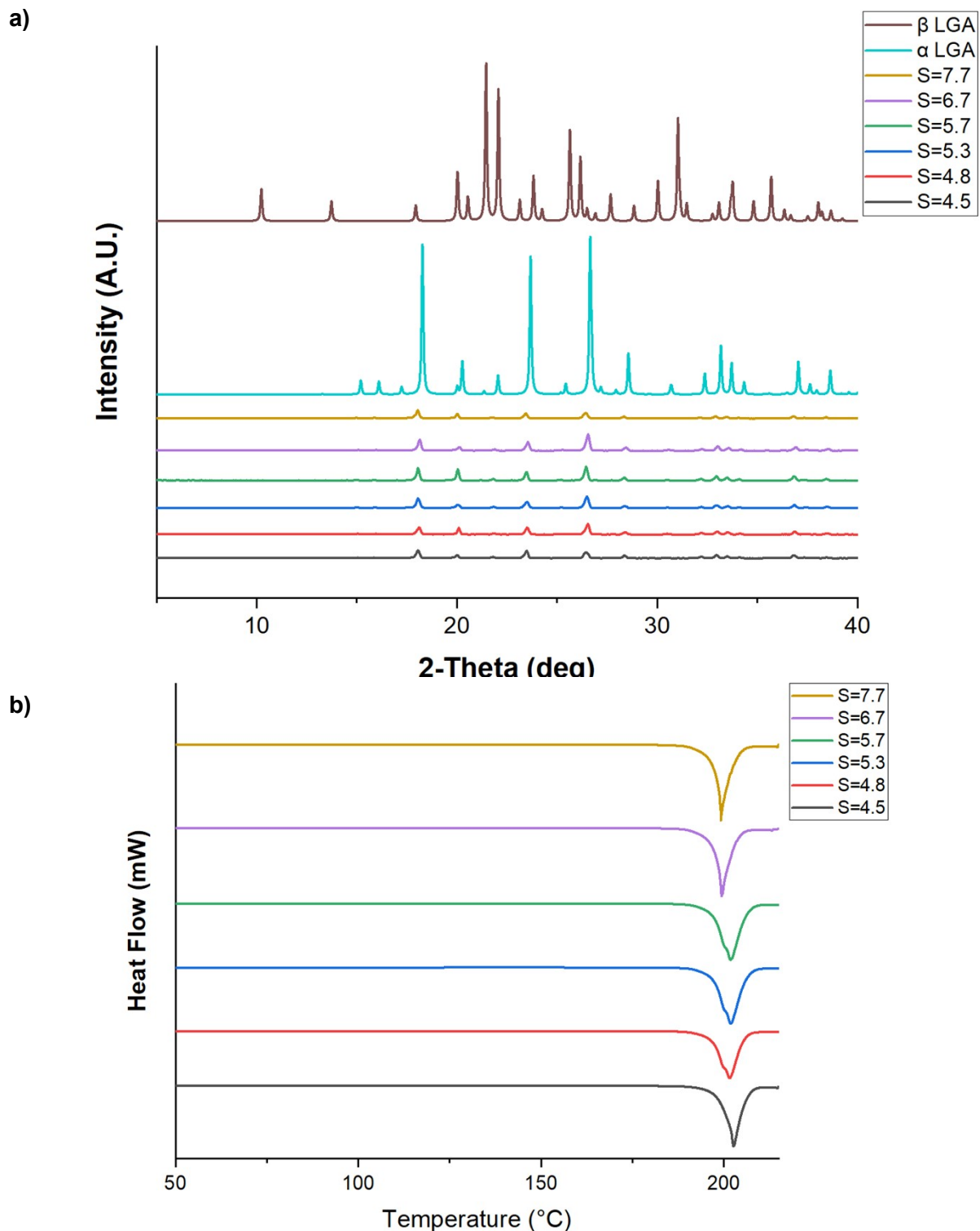


Figure S7 Microscope images and (b) XRD pattern of product crystals from stirred-tank reactor/crystallizer in batch mode. Reactants were mixed following stoichiometry of reaction in Eq 1 for MSG a) 0.47, b) 0.6 and c) 0.8 to have starting concentrations at initial supersaturation of 4.5, 5.7, and 7.7 respectively, the same as Fig. 2a, S2a, 2d, S2d, 2f and S2f. d) XRD pattern for (a-c).

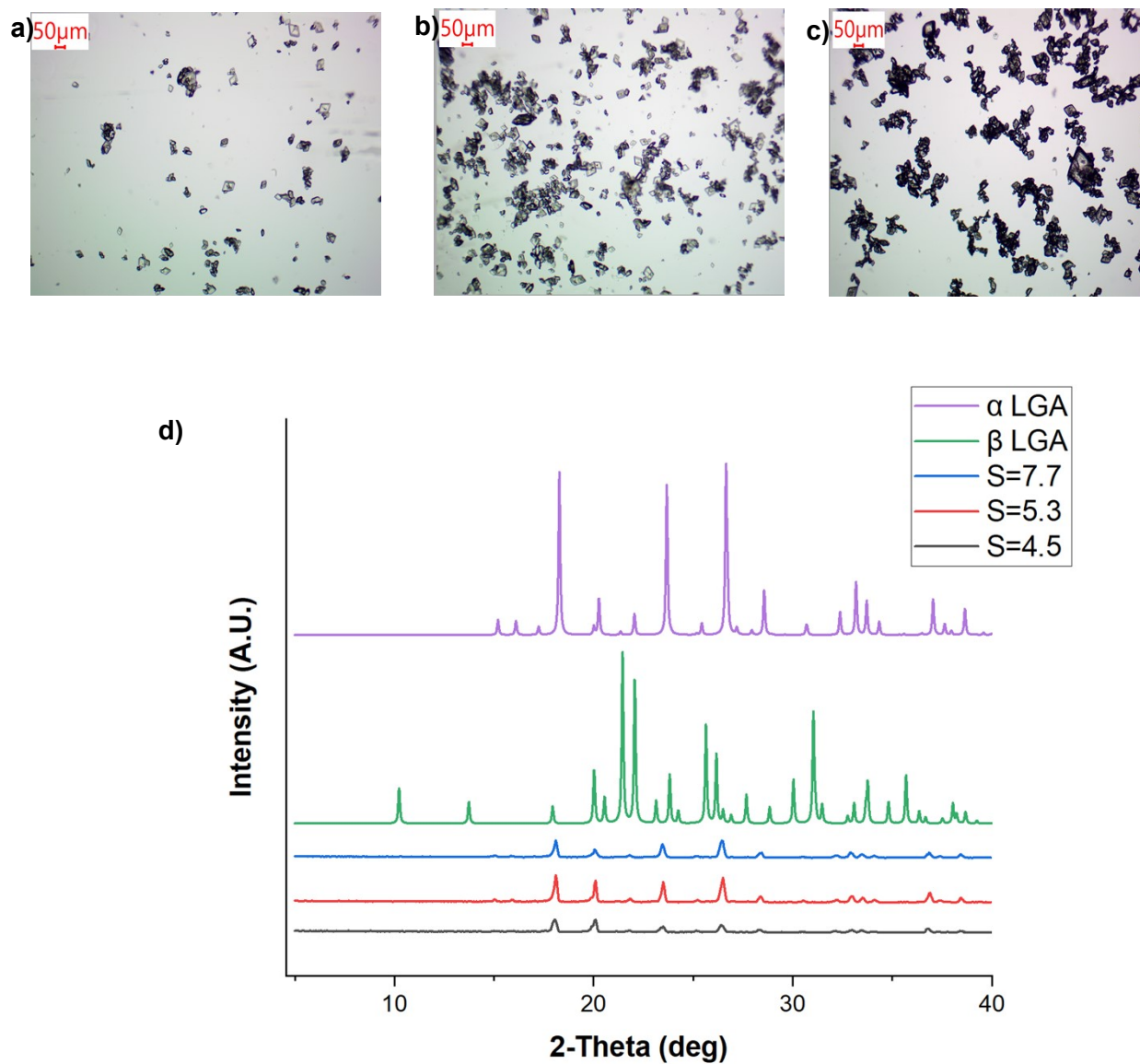
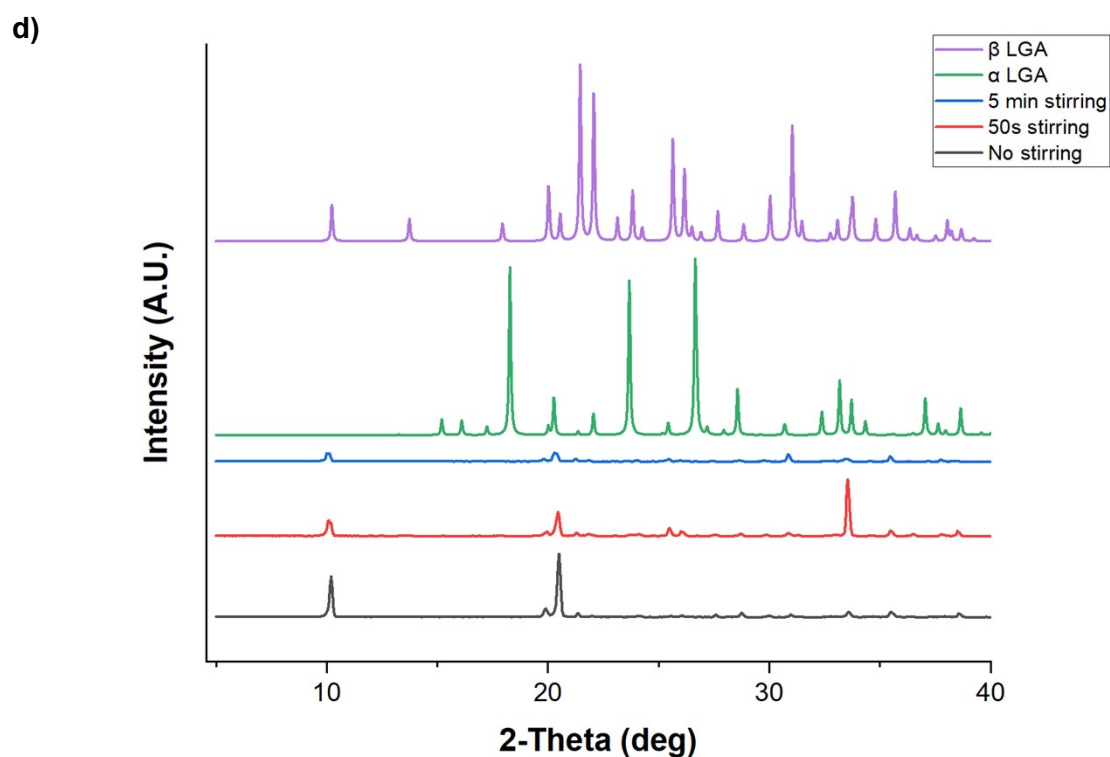
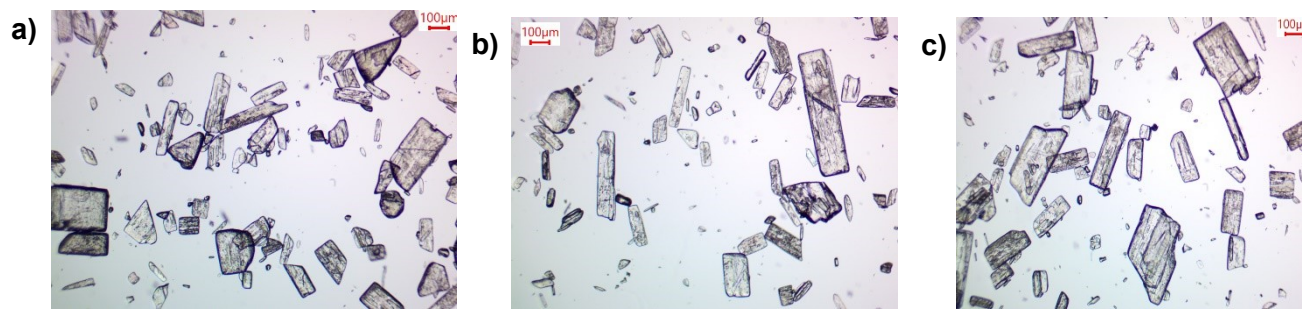


Figure S8 Microscope images of slurry of 0.025g/mL solution of commercial L-glutamic acid powders (β -form) in stirred tank, after mechanical stirring for a) 0 second, b) 50 seconds, and c) 5 minutes. d) XRD pattern for (a-c). All showing no polymorphic transformation from β to α form within the experimental time frame.



References

- 1 Y. Tahri, E. Gagnière, E. Chabanon, T. Bounahmidi and D. Mangin, *J. Cryst. Growth*, 2016, **435**, 98–104.
- 2 J. Aarthi, P. Dhanasekaran, T. S. Senthil and N. M. Ganesan, *Cryst. Res. Technol.*, 2018, **53**, 1700190.