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Experimental/numerical approaches for the investigation of nucleation, growth and aggregation mechanisms for the precipitation of Na_2SO_4 in supercritical water

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ABSTRACT

Supercritical water oxidation processes (SCWO) have been developed as an alternative technology to treat toxic and/or complex chemical wastes with a very good efficiency. However, one main limitation of SCWO processes comes from the precipitation of inorganic compounds. When dealing with supercritical water conditions (T ≥ 374°C, P ≥ 22.1 MPa), the polarity of water drops and inorganics, such as salts, are no longer soluble. This precipitation phenomenon results in a solid salt deposition in the reactor, which leads to clogging and interruption of the continuous process. Although this limitation is well known, little information are available in the literature regarding the precipitation mechanism and the salt particle properties. Great efforts have been put to solve this precipitation problem, mainly dealing with engineering through the development of new reactor designs. In this context, we propose to study the salt precipitation from another strategy, focused on the understanding of the precipitation phenomenon in supercritical water, using one common salt: disodium sulfate (Na_2SO_4) [1].

Precipitation occurs through the following mechanisms: nucleation (primary or secondary), the growth and finally aggregation and breakage of the particles. We propose in this work to investigate separately the nucleation and the growth of the crystallites and the final aggregation of the particles. These phenomena are studied both numerically and experimentally in order to provide interesting data for modeling and validation. As a first step, a numerical modeling of the precipitation in supercritical conditions is performed by taking into account all the implied physical phenomena: thermodynamic, hydrodynamic (CFD) and nucleation & growth [2]. Crystallite sizes formed after precipitation are measured with *in situ* wide angle X-ray scattering (WAXS) in sapphire capillary for comparison and determination of the growth rate of the particles.

In the second part, this work intends to study the formation of salt aggregates. For that, a specific and dedicated experimental set-up is presented and succeeds in recovering salt powders from supercritical precipitation. Several analyses are performed on the salt samples to obtain aggregate sizes, morphologies and size distributions. Based on the experimental results and the previous results of nucleation and growth mechanisms, a numerical modeling of the salt precipitation and aggregation is reported to acquire information on the possible aggregation mechanism.

References

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[2] Voisin, T., Erriguible, A., Philippot, G., Ballenghien, D., Mateos, D., Cansell, F., Iversen, B.B., Aymonier, C. Investigation of the precipitation of Na2SO4 in supercritical water (2017) Chemical Engineering Science, 174, pp. 268-276.