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Experimental study about plugging in confined impinging jet mixers during the precipitation of strontium sulfate

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ABSTRACT

Precipitation in industry is frequently used for the manufacturing of fine particles. Stirred tank reactors are used for the production of these precipitates in most processes. Confined impinging jet mixers (CIJMs) are alternative precipitation apparatuses. Compared to stirred tanks, their use enables the production of higher product quality because of more advantageous, well-defined mixing conditions. Nevertheless, CIJMs have not yet found widespread application in industrial precipitation processes because of their tendency to plug. Fluid dynamic behavior of CIJMs depends on their geometry and is of importance for its plugging affinity. The plugging affinity is defined as the ratio between the pressure drop during precipitation and pressure drop during operation under non-precipitating conditions. In this paper, the plugging affinities of different CIJMs are investigated for the case of strontium sulfate precipitation. Strontium sulfate is known for its high plugging affinity. Hence, it is suitable for mixer plugging experiments. A search for conditions causing plugging is made by carrying out experiments at various flow rates and degrees of supersaturation. Three mixer types of similar basic dimensions are compared. Furthermore, the influence of ultrasound on the plugging affinity is investigated. It is found that ultrasound has some capability of protecting CIJMs against plugging.

1. Introduction

Precipitation is one of several possible unit operations in industrial process lines for manufacturing particles in the size range from several nanometers up to a few micrometers. Stirred tank reactors are generally used for the production of these precipitates, but confined impinging jet mixers (CIJMs) are promising precipitation apparatuses. However, CIJMs have not yet found widespread application in industrial precipitation processes. Approximately 95% of industrial processes produce precipitates in stirred tank reactors [1]. One reason for this is the plugging (clogging, blocking, obstruction) of mixing nozzles. Plugging is caused by nucleation and growth of precipitate on the internal surfaces of CIJMs or by high solid concentrations [2–4]. Therefore, CIJMs are used only on a laboratory scale. One scope of CIJMs for the past 50 years has been the investigation of fundamental mechanisms of particle formation [5,6]. A major task of CIJMs is to exclude mixing effects on the supersaturation buildup. Particle formation of fast processing precipitations can be faster than the mixing of reactants, especially at high supersaturations. Due to the very fast mixing with CIJMs ($<$ 5ms), it is possible to reach the supersaturation intended by excluding mixing effects on supersaturation buildup [7–14]. Product properties can also be controlled better due to the advantages of CIJMs compared to stirred tank reactors. As mentioned above, reactants can be mixed quickly, which leads to a high initial supersaturation due to the small mixing volume in CIJMs. Supersaturation is distributed more uniformly in the mixing zone of CIJMs than in the addition zone or bulk of a stirred tank reactor. The particles formed grow into small, narrow and unimodal particle size distributions due to the well mixed suspension [13,15,16]. Because precipitation is a common method for the production of nanoparticles [9,17–21] there is a need for controlling secondary processes like aggregation. With CIJMs it is possible to time the influence of additives on secondary processes more accurately than in a stirred tank reactor, because the addition of additives can be purposefully adjusted at desired points of time. Agglomeration of particles, for instance, can be suppressed by adding a surfactant, even shortly after reaching solid liquid equilibrium of the solution [22].

All the advantages of CIJMs compared to stirred tank reactors are not applicable in industry because of plugging. The fluid dynamic behavior in CIJMs is one reason for plugging and depends on the geometry of the CIJM [10,11,23,24]. In order to compare the

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Nomenclature

$\tilde{c}_0$	Initial molar concentration of reactant solutions (mol/l)
c^*	Mass concentration of $SrSO_4$ in solid liquid equilibrium (kg/m^3)
$\overline{D_P}$	Mean pressure ratio
D_P	Pressure ratio
Δp	Pressure (bar)
$\overline{\Delta p_S}$	Pressure during precipitation (bar)
$\overline{\Delta p_W}$	Mean pressure drop of water (bar)
Δp_W	Pressure of water (bar)
Re_{mix}	Reynolds number in the mixing zone
S_a	Activity-based supersaturation ratio
s	Travel length of syringe pistons (m)
t	Time (s)
u_{mix}	Flow velocity in the mixing zone (m/s)
$\dot{V}_{mix}$	Total volume flow rate in the mixing zone (ml/min)
V	Volume passed (ml)
ϕ^*	Volume content of $SrSO_4$ in solid liquid equilibrium (vol. (%))

plugging affinities of various CIJMs, the precipitation of strontium sulfate is assumed to be a suitable chemical system for testing because it forms a gelatinous phase within milliseconds at high supersaturation. Kucher [25] described the formation mechanism of strontium sulfate in detail. At high supersaturation ($S_a \geq 15$), the gel consists of needle-like crystals with lengths in the range of 50 μm. This morphology seems to be metastable. After some seconds to few minutes, these crystals ripened to the stable morphology due to a second nucleation event. Thereby, the suspension loses its gelatinous structure and an ordinary suspension is formed. Solid liquid equilibrium is reached after all crystals reached the stable morphology. Kucher and Kind [26] reported that it was not possible to precipitate strontium sulfate with the Y-mixer they used, because the mixer plugged within seconds. The pumps of the experimental setup were not able to exhaust the mixing zone from the blockage. In this study, we propose a more convenient experimental setup to carry out strontium sulfate precipitations in CIJMs. We hypothesize that the tendency for plugging is influenced by the CIJM geometry. Different CIJM types, Y-mixer, T-mixer and two-jet vortex (TJV) mixer, have a similar basic geometry for an independent comparison of the plugging affinity. The pressure drop in CIJMs is measured online during precipitation. Precipitation is carried out at high supersaturations, which lead quickly to blockages under regular conditions. Therefore, an experimental setup is required that can compensate for high pressure drops. Data acquisition with a high sampling rate is also needed in order to record sudden changes of the pressure drop while the CIJM is plugging.

It is known that plugging is a big problem in terms of solid liquid separation and transport of suspensions through porous media [27–29]. There are hints in the literature, that it is possible to

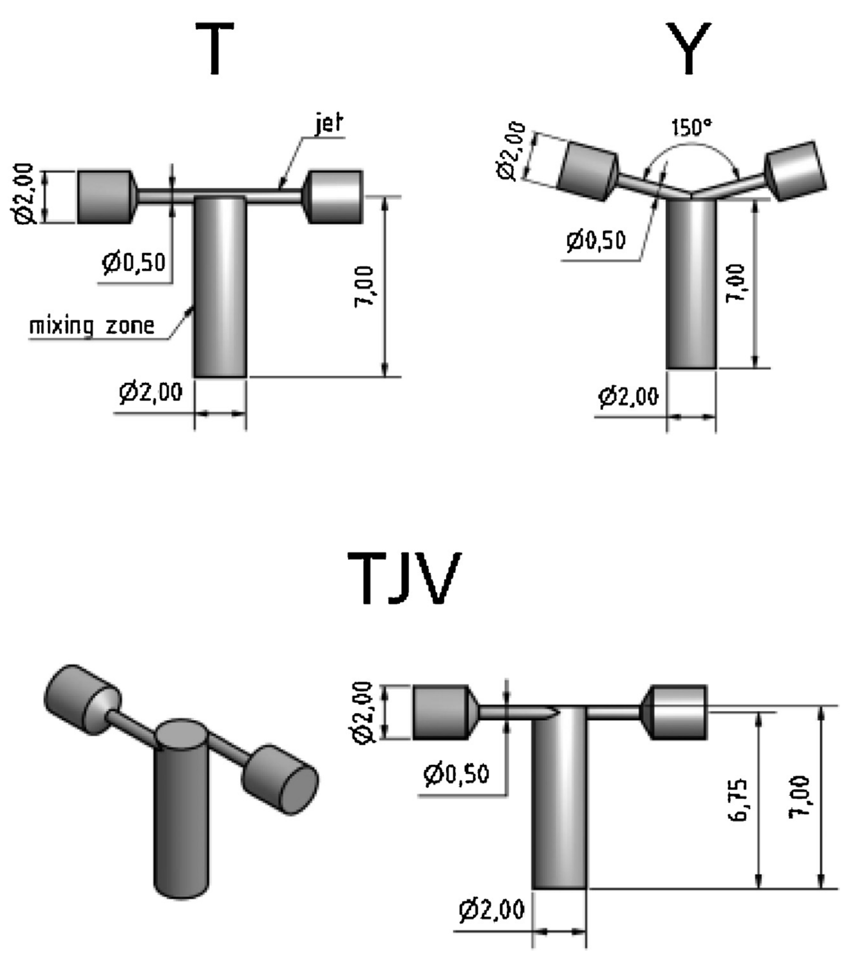

Fig. 1. CAD sketches of three CIJM types.