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Tuning Particle Size in Helical Coil Channels

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***Abstract* — Precipitation, in recent years, has become a very important and researched about the topic for industries like the pharmaceutical. The need to understand this process so as to be able to control and obtain the desirable size, shape and surface area of Active Pharmaceutical Ingredient (API's) which would in turn lead to obtaining more suitable physiochemical properties thus becomes crucial. The aim of this article is therefore to study the factors which affect this particle size distribution (basically nucleation) and suggests the helical coil configuration to be more suitable for controlling the size. This is concluded by studying this same phenomenon in a semi batch reactor, effect of coiled configuration on residence time distribution and the mixing in curved tubes. Later, the theoretical work is supported by an experimental setup and the approach of the setup is discussed. Finally, we conclude by stating the results which could be derived from this set-up.**

***Keywords* — Helical coil, Precipitation, Active Pharmaceutical Ingredient, nucleation, semi batch reactor.**

I. INTRODUCTION

Precipitation or crystallization is the creation of the solid phase inside the solution or another solid phase. The generation of solid is due to the fact that the amount of the solid present in the solution is more than the equilibrium amount. Studies have shown that both the rate of precipitation and the particle size distribution depend significantly on the extent of micro-mixing ([3]). In laminar flow in straight pipes, diffusion is the dominant mechanism of mass transfer. In such conditions, mixing can be improved by introducing bends in the channels as explained by Dean Flow. ([6], [7])

Any perturbation of the flow that creates lateral movement will stretch the interface and increase the rate of mixing. According to the phenomenon of Dean Flow, addition of curvature to a microchannel induces two counter-rotating flow vortices orthogonal to the main flow direction which introduces an additional drag force on the micro-particle. We can control flow conditions in such a way that Dean Flow recirculates micro particles suspended within the primary flow until they settle in stable equilibrium positions. It has been shown ([8]) that particles can become focussed at one of two locations, either towards the top or bottom of the channel's cross-section at equal lateral displacements determined by the flow rate, as shown in the figure given below. In curved micro-channels, by varying channel dimensions, curvature of the flow channel, micro-particle dimensions and flow rate, we can focus micro-particles of different dimensions at different lateral locations within the flow ([8]). This thus increases mixing to a great extent ([9]).

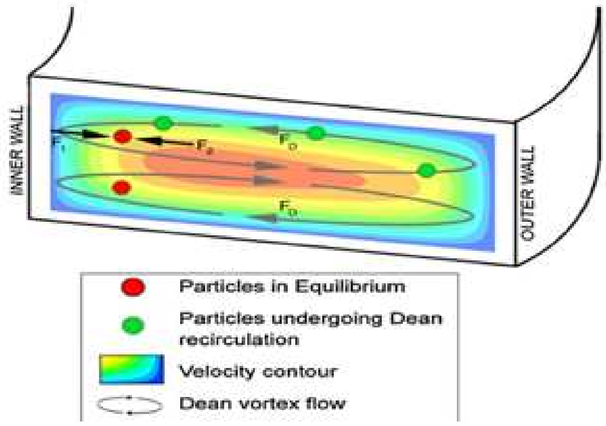

Fig. 1 Schematic cross section of a curved microchannel showing the two counter-rotating Dean vortices orthogonal to the main flow direction as a result of channel curvature, where F_D = Dean drag force, acting to recirculate non-equilibrated particles (*green*). The equilibrated particles (*red*) are subject to $F_1 = F_D + F_{WL}$, where F_{WL} = wall lift force and F_2 = Saffman lift force

(Source [9])

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The phenomenon of precipitation is not completely understood. The complexity lies in dealing with fluid flow, solid generation, diffusion and reaction kinetics at the same time. The number of the nuclei generated and the initial size depends on the process of nucleation but some processes like agglomeration, ripening and breaking create particles having different shape which makes the process analysis very difficult. ([2])

Nucleation means crossing of phase boundaries when the system reaches a thermodynamic unstable state temporarily. Growth follows nucleation and its rate and mechanism depends on the level of supersaturation. Agglomeration also results in the increase in the size of the particles. In this, two smaller particles collide and combine to form larger particles. Ripening is the process in which the system moves towards the more stable state. During the ripening less stable crystals are dissolved and more stable crystals are formed from the already formed crystals.

After the substances are properly mixed, a chemical reaction takes place following reaction kinetics and the product generated precipitates out depending on factors like critical supersaturation at the reaction temperature. ([3])

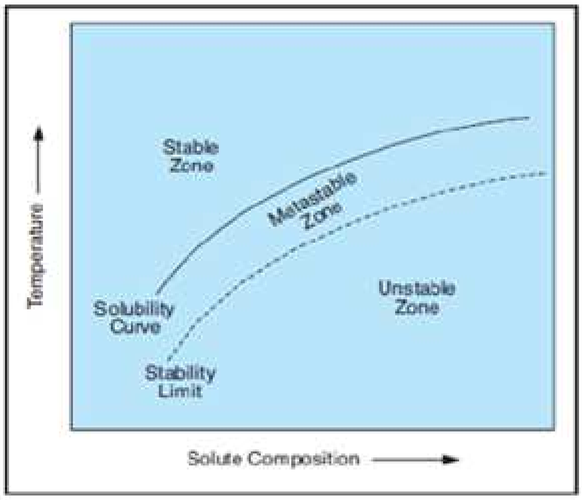

Fig2-Variation of solubility with temperature

(Source : [1])

The graph is showing 3 regions mainly:

Stable zone: No precipitation takes place as the solution is unsaturated.

Unstable zone: Precipitation is spontaneous and does not require the presence of nucleation sites.

Metastable zone: Supersaturated but the precipitation is not spontaneous and the presence of the nucleation sites is necessary for the precipitation to take place.

Diagrammatically, precipitation can be shown as given below. ([5])

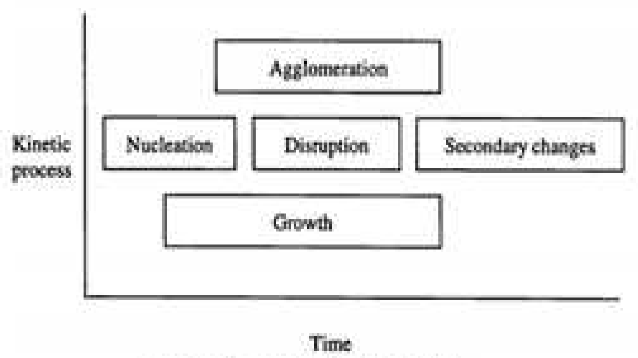

Fig3-The figure shows the order in which different phenomenon take place with respect to time that lead to precipitation.

(Source : [5])

Our aim here is to study the polydispersity of the particles formed, assuming complex processes like agglomeration, ripening and recrystallization are not taking place which is going to be true for short times. Thus, our main interest lies in studying the variation of particle size formed when efficient mixing leads to chemical reaction thus producing nuclei followed by its growth. We assume that all the nucleation sites are produced at time t=0 and after that we are always in the metastable region.

II. Literature Review

A lot of experiments and research have been done in the field of particle size distribution and on the ways helical coils affect the mixing of reactants.

A. *Particle size distribution for a semi-batch precipitation reactor.*

The particle size distribution (PSD) is one of the most important properties to control in processing steps like crystallization, precipitation and polymerization. The PSD obtained in the case of the below mentioned reaction exhibited significant variability, owing to variations in the individual particle phenomena such as nucleation, growth, aggregation, and breakage. Due to unavailability of sufficient secondary measurements for accurate inference of the entire PSD, complicating phenomena like aggregation and breakage was neglected and growth and nucleation kinetics were solely used for building a prediction model for PSD. ([4])

The reaction which was studied was:

$$Ca(OH) \;\; + Na_2CO_3 \;\rightarrow\; CaCO_3 \; + 2NaOH$$

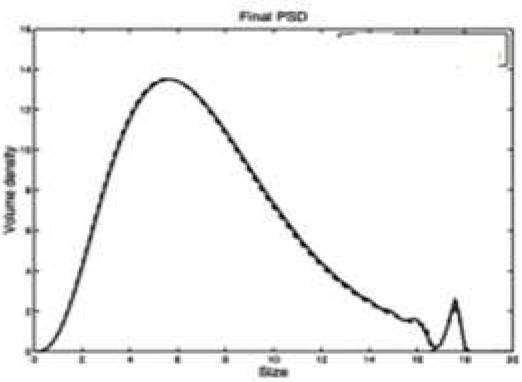

Fig4-This graph shows the particle size distribution in a semi batch reactor.

(Source: [4])

B. *Effect of coiled configuration of residence time distribution*

To improve the performance of flow reactors and heat exchangers we desired to achieve uniform reaction conditions or lower temperature gradients. Narrower residence time and thermal time distributions can be obtained by increasing the mixing between the fluid elements.

Experiments have revealed that bending of helical coils causes a significant narrowing of the residence time distributions even for low values of Dean Number. The parameters which are effective in narrowing the residence time distribution in bent coils are: number of bends, bend angle, and bend spacing ([11]). It has been found out experimentally that increase in the coil pitch broadens the RTD while the increase in the cross-sectional ellipticity narrows the RTD. ([13])

C. *Mixing In curved tubes*

In curved tubes increase in Reynolds number produces increased levels of secondary flow, which produce enhanced mixing, whereas in straight tubes as the Reynolds number increases, residence time for diffusion to take place decreases which thus leads to a decrease in the mixing efficiency. Thus a balance of mixing mechanisms due to molecular diffusion and secondary flow exists in helical tubes. It was observed that at low Reynolds numbers, i.e., of the order of 0.1, curved tube mixers are not very attractive because of the low intensity of the secondary flow, but at higher values of the Reynolds number, of the order of 10–100, curved tube present an efficient solution. Therefore curved tube mixers may provide an alternate solution for static mixers in the Reynolds number range of 10 to 100. It was also observed that at a low curvature ratio (i.e., λ=5) the secondary flows are stronger and mixing is higher as compared to the higher curvature ratio (i.e., λ=10). ([10])

It has been proved that the performance of coiled chemical reactor lies between the performance of plug and laminar reactors. ([12])

Going through the past and ongoing researches in this field, we found out that there is scope in conducting experiments to study the particle size distribution in helical coils. Experiments have been conducted on PSD for batch reactors and also a lot of research is done on the geometry of coils. Thus we aim at combining both the areas and coming up with satisfactory results.

III. Objectives

- Obtain the particle size distribution curve at different concentration and flow rate conditions inside a helical coil channel
- To compare the efficiency of mixing of reactants in the helical coil channels to that in batch reactors

IV. Approach

To study the polydispersity of particles formed in the helical channels we must take into consideration, the critical concentration of solubility. We have to first ensure that we are always in the metastable region when growth is taking place as otherwise the number of nuclear sites will not be a constant and reaching to a general result in this case would not be feasible. For this we first determine the maximum concentration of the reactants which would satisfy the above conditions. This concentration can be estimated by the dilution method ([1]).

The concentration of the products depend upon the extent of mixing. We cannot directly tune the mixing conditions, but by varying the flow rate and concentration of reactants, we try to understand their combined effect on mixing.

Once determined, we set a specific temperature and choose an appropriate flow rate. The concentration of the reactants is then varied below the critical concentration and the particle size distribution is plotted.

Finally, we plot the graph for different flow rates.

Our reaction of interest is:

$$ZnNO_3 + NaOH \rightarrow ZnO + Side\ products$$

The particle size distribution of calcium carbonate has thoroughly been studied in the past in semi batch reactors and thus the results we obtain in case of helical coils can be compared to this.

Hence we will obtain experimental results on the variation of all the parameters and this graph could be extrapolated to get values at conditions which are experimentally not possible. For example, if we need to assume metastable region at concentrations higher than the critical concentration.

V. Conclusion

The experimental setup and procedure as discussed above may be used to obtain results of high importance and which could be used for a better understanding of the process. The results which could be concluded by following the above methods are:

- Effect of flow rate on final particle size distribution in helical coils
- Effect of reactant concentration on final particle size distribution in helical coils
- Understanding the influence of flow rate and concentration of mixing in helical coils

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