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Liquid–liquid dispersion in a continuous oscillatory baffled reactor – Application to suspension polymerization

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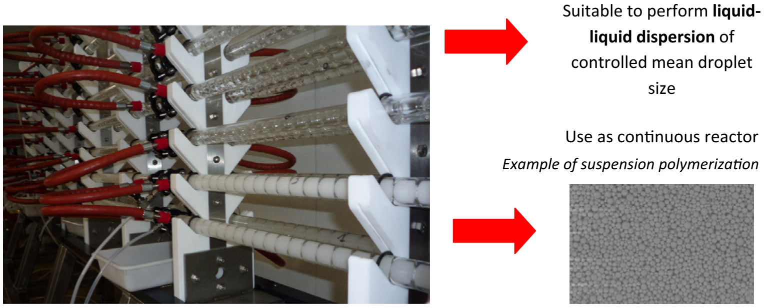
HIGHLIGHTS

- We study the batch to continuous transposition for process based on liquid–liquid dispersion.
- Liquid–liquid dispersion in a continuous oscillatory baffled reactor is thoroughly studied.
- Oscillation is the main parameters responsible for droplet breakage.
- Net flow Reynolds number does not affect the mean droplet size but allows the control of the residence time.
- Vinyl acetate suspension polymerization can be carried out continuously, including the sticky step, in continuous oscillatory baffled reactor.

GRAPHICAL ABSTRACT



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ABSTRACT

Reducing energy costs, improving safety, minimizing waste are the current aims of chemical engineering. Process intensification in fine chemistry has been extensively studied but less work refers to heterogeneous reactions involving two liquid phases. This paper focuses on batch to continuous suspension polymerization transposition and especially on the liquid–liquid dispersion step. The main features of suspension polymerization reaction are based on (i) the initial liquid–liquid dispersion requiring a controlled size and narrow distribution and (ii) on the control of the final particle size during the agglomeration step by avoiding fouling which is a bottleneck for continuous flow. For the continuous transposition, liquid–liquid dispersion and reaction studies are carried out in continuous oscillatory baffled reactor (COBR) which is a multipurpose reactor. Its suitability to overcome the bottlenecks is demonstrated through the investigation of the oscillating and flow conditions or the dispersed phase holdup. A controlled droplet size distribution can be achieved. Oscillation is the main parameter responsible for droplet breakage. The suitable conditions to obtain stable dispersion are determined. For one of the first times, COBR is used for suspension polymerization.

1. Introduction

Liquid–liquid dispersions involve the droplets generation of controlled droplet size and distribution. They are implied within

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Nomenclature

A	amplitude [m]
C_D	standard orifice coefficient
D_{ax}	axial dispersion coefficient [m^2 s^{-1}]
D_o	annular opening diameter [m]
d_{32}	mean Sauter diameter [m]
D	internal column diameter [m]
D_h	hydraulic diameter [m]
f	frequency [s^{-1}]
h	baffle spacing [m]
L	reactor length [m]
N	number of baffles per unit length [m^{-1}]
N_{tot}	total number of baffles in the column
U,u	global velocity [m s^{-1}]
U_0	net flow velocity [m s^{-1}]
U_p	pulsation velocity [m s^{-1}]
Q_c	continuous phase flow rate
ΔP	pressure drop [Pa]
T	transparency factor [dimensionless]
Q_d	dispersed phase flow rate [m^3 s^{-1}]
t	time [s]

Greek symbols

ε	mean energy dissipation rate due to oscillatory flow [W kg^{-1}]
ε_n	mean energy dissipation rate due to net flow [W kg^{-1}]
ρ_c	continuous phase density [kg m^{-3}]
ρ_d	dispersed phase density [kg m^{-3}]
σ	interfacial tension [N m^{-1}]
υ	kinematic viscosity [m^2 s^{-3}]
Φ	holdup

Dimensionless numbers

Pe	Peclet number
Re_n	net flow Reynolds number
Re_o	oscillatory Reynolds number
Re_{oh}	hydrodynamic oscillatory Reynolds number
We_h	hydrodynamic Weber number

different processes including liquid–liquid extraction [1], crystallization [2–4] or polymerization to control final particle or latex properties [5,6]. For such separation techniques or reactions, it is crucial to control accurately the dispersion because it is directly related to efficiency of processes. Among the process intensification route, homogeneous reactions have been deeply investigated as well as nonpolymer reactions [7]. Batch to continuous transposition is often reported to increase the product yield and quality, decrease the use of reactants (catalysts), solvent or other materials, improve safety and allow the work at extreme operating conditions. Few studies focus on batch to continuous transposition of liquid–liquid reactions and especially on two-phase polymerization reactions [8,9]. Consequently, in this paper, suspension polymerization is considered. Traditionally performed by thermally initiated radical polymerization in batch stirred tank reactors, the continuous transposition is based on the final product properties and kinetics observation. The initial properties of the liquid–liquid dispersion will strongly impact the properties of the final polymer beads or pearls [10]. Suspension polymerization can then be divided into three different steps [11,12]: (i) a liquid–liquid dispersion step, (ii) the reaction steps in which the polymerizing droplets can agglomerate leading to the final polymer particle size and (iii) a step enabling to reach a conversion between 80% and 90% without change in the polymer particle size. Fig. 1 sums up the steps and underlines the limitations of the suspension polymerization reaction.

The first bottleneck in the continuous transposition remains in the droplets production. The initial dispersion requires controlled mean droplet diameter and droplet size distribution. It has to be stable enough to prevent from coalescence and destabilization in course of polymerization. Different continuous processes are available to create liquid–liquid dispersion including membrane [13], rotor–stator [14], static mixer [15–17], colloid mills [14,18], high pressure homogenizer [19], mixer-settler [20] and pulsed column [21,22]. A remarkable feature of continuous liquid–liquid dispersion processes are their ability to create quickly droplet of controlled size. Whereas in batch stirred tank reactors, the emulsification time, defined as the time needed to reach stabilized mean droplets sizes, is typically about 15–30 min [17,23–26], this time can be a limiting step when it comes to continuous process owning residence time ranging from microsecond, to millisecond, to second [15–19].

Table 1 provides some examples of mean droplet size correlation including their range of applications depending on the chemical parameters (viscosity, densities, and interfacial tension), equipment parameters (porosity, pore diameter, pipe diameter, stirrer dimension…) and the operating conditions (flow rate, shear rate, pulsation…). Their limitations are underlined. Balances between hydrodynamic conditions and physico-chemical parameters are different from batch in continuous processes because breakage, coalescence phenomenon and interface stabilization act on different time scales. It is important to understand the mechanisms which display the droplet size in this type of continuous liquid–liquid contactors. These different contactors can thus be used to generate droplets and be associated with different

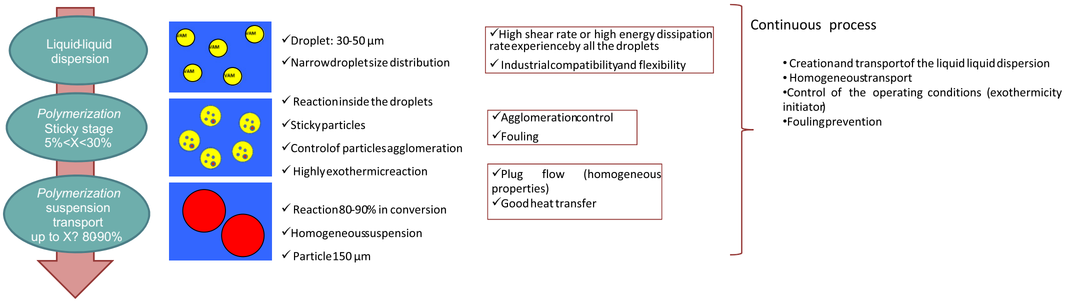

Fig. 1. Steps and limitation for continuous suspension polymerization.