



Regular Article

Dynamics of aggregate size and shape properties under sequenced flocculation in a turbulent Taylor-Couette reactor

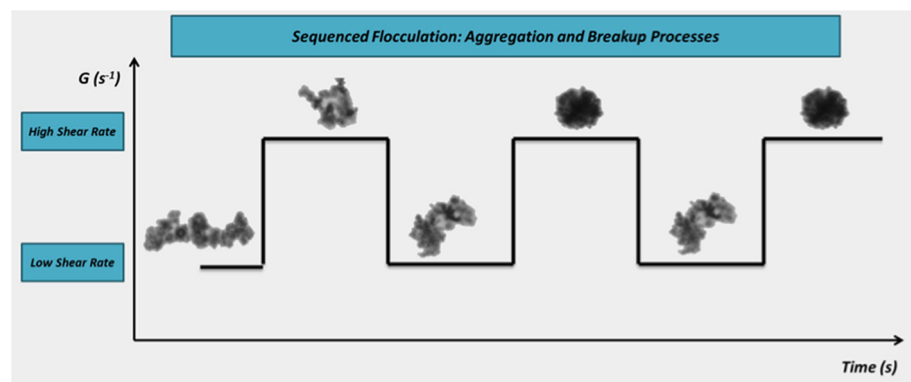


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GRAPHICAL ABSTRACT



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ABSTRACT

This paper concerns experimental investigation of the sequenced flocculation of latex particles in a Taylor-Couette reactor. The aim of this work was to investigate the evolution of both the size and the shape of aggregates under sequenced hydrodynamics. A number of studies have focused on the evolution of the aggregate size or size distribution during steps of growth-breakage-regrowth, but aggregates generally experience steps of breakage-regrowth on repeated occasions in real operating conditions (passages near the impeller or during the transfer processes, for example). The experiments conducted in this work consisted thus of an alternation of six steps with alternately low and high shear rates under turbulent conditions. The particle size distributions were monitored throughout the sequencing, and the circularity and convexity (shape parameters) distributions were measured, enabling a more precise description of the entire floc population, rather than a fractal dimension. While the aggregate size distribution was clearly controlled by hydrodynamics, the shape distributions continuously evolved during the sequencing. The main new finding of our work notes the independence between the aggregate shape and hydrodynamics. Indeed, after multiples steps of breakage-regrowth, regardless of the aggregate size distribution and hydrodynamics, the aggregate shape seemed to reach a unique steady-state morphological distribution.

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1. Introduction

Coagulation-flocculation is one of the most common processes used for the removal of fine particles in water. One of its well-known applications is water treatment, in which it facilitates

reduction of both the water turbidity and the amount of organic matter. The final properties of particulate systems result from aggregation processes during which primary particles stick together to form clusters and agglomerates or flocs whose size can finally reach several millimetres.

Consecutively to the aggregation of particles, various sizes and shapes of flocs can be observed in a suspension. In the case of water treatment, the flocculation step is commonly followed by a settling or filtration step whose efficiency can be greatly altered by the size, shape and aggregate density [1,2]. Producing aggregates with desired properties is thus crucial. Indeed, previous studies have shown that, depending on the aggregate shape, the settling velocity could be increased [3] or the resistance of the filtration cake could be impacted [4,5]. The coupled investigation of not only the size distributions of the aggregates but also their morphological characteristics is thus fundamental. The aggregate characterization can be made by either *in situ* or *ex situ* measurements. *In situ* measurements are generally performed by coupling a laser light sheet for illumination with a camera [6–10] whereas *ex situ* methods are based on confocal laser scanning microscopy [1,11–13] or sample analyses coupling a microscope and a CCD camera [7,14–17].

Most works concerning aggregate size and morphology focus on monitoring the size distributions (with time or at steady-state) and the fractal dimension (D_f). Settling, light scattering and image analysis are three common methods for determining the fractal dimension. The settling method gives a mass fractal dimension, a function of the aggregate volume [18,19]. The light scattering method consists of plotting the light intensity (I) scattered by the aggregate suspension as a function of scattering vector. Under certain conditions, the fractal dimension of the aggregates may then be deduced from the slope of the resulting curve ($I = f(Q)$) [11,19–23]. Finally, image processing enables connection of the area of the aggregate (A) with its perimeter (P) or its length (l) ($A \propto P^{2/D_f}$ or $A \propto l^{D_f}$) [19,23]. However, they all fail to represent the wide diversity within a population of aggregates. Indeed, the fractal dimension is not an individual characteristic but a global one [10,24–27]. This deficiency can be overcome by analyzing both the size and morphology distributions.

It is commonly observed that, depending on the hydrodynamics, the aggregate size can be greatly affected [28–31]. An increase in the applied shear rate generally leads to an aggregate diameter decrease associated with a tightened distribution and an increased fractal dimension. In stirred tanks or during the transfer processes, for example, aggregates are submitted to various shear rates due to the heterogeneity of hydrodynamics. Those shear rate fluctuations have led some authors to analyse the impact of steps of breakage and regrowth on the aggregate size and morphology [26,32]. These works often show that, in simple cycled shear flocculation cases (a step of growth immediately followed by a step of breakage and a step of regrowth), the diameter of the aggregates after a step of regrowth is generally lower than the diameter before the breakage step. Moreover, it has been shown that the fractal dimension value is higher after the regrowth step than after the first flocculation step, which is representative of more compact aggregates. Cycled shear flocculation thus seems irreversible [24,28]. However, although several authors have investigated the case of floc breakage and regrowth, few have studied solid-liquid suspension behaviour after more than one step of breakage to find out if the irreversibility phenomenon continues [6,33,34]. Those studies have generally provided data concerning the aggregate sizes but nothing about their morphology. More recently, some works have reported on morphological image analysis with the aim of better understanding the effect of hydrodynamics on floc properties [9,35–37].

In this context, the present work provides new results obtained during hydrodynamic sequencing experiments in a Taylor-Couette reactor under turbulent conditions. The objective of this article is to investigate the impact of several steps of breakage and regrowth under varying hydrodynamic conditions on both the size and the shape of latex aggregates. An *in situ* monitoring of the aggregate sizes has been conducted using a particle size analyser, providing access to the volume size distributions of aggregates with time. After each shear rate change, a sample of the suspension was analysed with a microscope coupled with image analysis software, providing several shape parameter values for each aggregate.

2. Experimental setup

2.1. Materials and devices

Spherical polystyrene latex particles from Polysciences Inc. with a diameter of 0.2 μm and a high level of surface sulphate groups were used for the experiments. All of the experiments have been conducted with a volume fraction of latex primary particles of 3.5×10^{-5} . NaCl was chosen as a coagulant. A salt solution was prepared with a concentration of 82 g of NaCl per litre. This concentration is higher than the critical coagulation concentration, and its value was chosen to have the same density between the particles and the solution ($\rho = 1055 \text{ kg m}^{-3}$), thus avoiding differential sedimentation phenomena during the experiments. Two litres of the salt solution were prepared several hours before the experiments using demineralized water, placed in a beaker and submitted to vigorous stirring to remove air bubbles, which could bias the results afterwards. All the experiments were carried out at room temperature, which ranged from 20 to 25 $^\circ\text{C}$.

After the stirring, the solution was poured into the Taylor-Couette reactor. This apparatus is composed of two concentric cylinders: an inner cylinder with a radius (R_i) of 10 cm and an outer cylinder with a radius (R_o) of 11.5 cm and a height (H) of 20 cm. The outer cylinder is fixed, while the inner one can rotate at various angular velocities (Ω), which can be controlled via a monitor. The reactor used during the experiments has been previously described in the literature [9].

The total volume of the reactor is approximately 2 L, and the solution fully fills the gap between the cylinders. The reactor is also equipped with tangential outlets, allowing the suspension to be sampled without damaging the aggregates. There are also two apertures on the top of the reactor to perform a direct sampling of the suspension using a syringe whose tip has been cut to widen it, thus yielding a sufficient diameter (approximately 3 mm) to avoid breaking the latex aggregates formed during the experiments.

A Taylor-Couette reactor was chosen in this study because the hydrodynamics and flow regimes inside such a reactor are well characterized and it is possible to create a controlled shear flow [38]. The flow regime in such a reactor is characterized by the Taylor and Reynolds numbers, the expressions of which are respectively given in Eqs. (1) and (2).

$$Ta = \frac{R_i \Omega^2 (R_o - R_i)^3}{\nu^2} \quad (1)$$

$$Re = \frac{R_i \Omega (R_o - R_i)}{\nu} = \left(\frac{Ta R_i}{(R_o - R_i)} \right)^{\frac{1}{2}} \quad (2)$$

In which Ω is the angular velocity [rad s^{-1}] and ν is the kinematic viscosity [$\text{m}^2 \text{s}^{-1}$]. The experiments have been carried out for a rotation speed of the inner cylinder ranging from 18 to

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