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Experimental analysis of coagulation of particles under low-shear flow

Jordi Colomer[a,*], Francesc Peters[b], Cèlia Marrasé[b]

[a]Grup de Física Ambiental, Department of Physics, University of Girona, Campus de Montilivi, 17071 Spain
[b]Institut de Ciències del Mar (CSIC), Pg. Marítim de la Barceloneta 37-49, 08003 Barcelona, Spain

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Abstract

The aggregation and breakup of particle flocs were investigated by monitoring the size distribution of a suspension of aggregates, with diameter d_o, under shear flow created by two mixing systems. The aggregation behavior was studied in 63 experiments under various conditions of induced shear rate and particle volume concentration for particle aggregates smaller than the Kolmogorov scale. Despite small shear rates being characteristics of natural systems, only experiments with comparatively high shear rates have been conducted to date. Because of this reason, in this study, the shear rates were chosen to mimic those found in natural systems. In the first set of experiments the aggregate size, d, was analyzed by changing the mean shear, $\overline{G}$ (ranging from 0.70 to 27.36 s^{-1}) created in a tank with a grid oscillating through the whole suspension volume. In the second set of experiments, a spherical flask was placed in an orbital shaking table where $\overline{G}$ ranged from 0.45 to 2.40 s^{-1}. In all the cases there was an increase of d at increasing $\overline{G}$. The dependence on d was found to be identical for the particle volume concentrations investigated, $\phi = 0.2, 0.8, 2, 4, 6, 8$ and 10×10^{-5}, with the stable aggregate size shifting towards aggregate growth as particle volume concentration increased. These results demonstrate that shear provided a means to keep the particle number count high for collisions to occur but it is small enough that the aggregation–breakup balance is dominated by aggregation.

Keywords: Particle coagulation; Concentration; Shear; Oscillating grid; Orbital shaker; Turbulence

1. Introduction

The intensity, extent and duration of the relevant mixing processes and the concentration of suspended particles are key parameters to understand the fate of microorganisms and inorganic particles in a body of water. It has been recognized that the intensity and the spatial and temporal characteristics of turbulence are important factors determining the dynamics of particles, especially for particles smaller than the scale of the smallest eddies dissipating turbulent energy (Kiørboe et al., 1994; Reynolds, 1994; Huppert et al., 1995; Li et al., 2004).

Among systems to generate sheared flows such as paddles, impellers and couette devices, this study focuses on laboratory experiments conducted in mixing boxes, where a characterizable turbulence is generated by (a) a vertical oscillation of a horizontal grid moving inside a container or (b) a container moving in an orbital shaker table. When the fluid is laden with particles, they are in a continuous process of aggregation and disaggregation until eventually a steady state is reached with a given

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*Corresponding author. Tel.: +34 972 418372; fax: +34 972 418098.
E-mail address: jordi.colomer@udg.es (J. Colomer).

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average aggregate size. Shear facilitates to some extent aggregation, but as shear rate increases it causes limiting growth to aggregates (Spicer et al., 1996; Spicer and Pratsinis, 1996a; Serra et al., 1997; Yukselen and Gregory, 2004). Floc structure and particle concentration of primary particles are important since they determine floc size and density (Spicer and Pratsinis, 1996a; Spicer et al., 1998; Mikkelsen and Keiding, 2002; Chakraborti et al., 2003; Selomulya et al., 2004).

The increase in floc size is found to be especially relevant in lakes and seas where aggregates can account for the removal of particles as they form. Seasonal changes in the intensity of turbulent mixing in a lake may produce shifts in phytoplankton succession and the population composition of the algal assemblage (Berman and Shteinman, 1998). Turbulence can also be highly significant to phytoplankton by affecting their swimming motion (Karp-Boss et al., 2000) and to grazing on bacteria (Peters et al., 2002). Biological rates related to the ingestion of particles or the uptake of dissolved substances are, on average, favored by turbulence although there is considerable variability related to growth, taxa and organism sizes (Peters and Marrasé, 2000). Flocs comprised of dead and living cells of green algae were found to form as a result of shear. At the same time shear produced growth inhibition (Gervais et al., 1997; Hondzo et al., 1998; Hondzo and Lyn, 1999; Juhl et al., 2001). Finally, turbulent motion has been found to affect bacterial growth and respiration (Bergstedt et al., 2004; Malits et al., 2004).

In this study, we experimentally address the importance of turbulence intensity on floc aggregation and breakup with primary particles of a given small size where the only relevant mechanism in the collision frequency is the shear stress. In general, turbulence levels are designed to maximize the effects of both particle concentration and shear rates on particle coagulation. Special attention is made to reduce the intensity of turbulence in the experiments compared to those used in other similar experiments (Spicer and Pratsinis, 1996b; Serra and Casamitjana, 1998; Liem et al., 2000; McAnally and Mehta, 2000). This results in a closer match of turbulence assessed in the laboratory and naturally occurring field turbulence. As pointed out by Hondzo and Lyn (1999), since shear rate (proportional to energy dissipation, ε, by $G=(\varepsilon/v)^{1/2}$, with v the kinematic viscosity) largely determines the steady-state diameter of any particle population, laboratory results are relevant to natural systems. Grid mixing systems have characteristics to maximize mixing intensity (therefore high particle contacts) while minimizing floc breakup rate (Liem et al., 2000). It has been found to be an alternative mixing device to traditional impeller systems, with an excellent performance for particle removal for flocculation mixing experiments (Liem et al., 2000). Also, containers placed in orbital shakers have been extensively found to account for the effects of turbulence on microorganisms (Savidge, 1981; Berdalet, 1992; Duetz and Witholt, 2001).

Since small shear rate conditions are dominant in aquatic environments (turbulence ranges from 10^{-6} to $10^{0}\,\mathrm{cm^2\,s^{-3}}$ in terms of dissipation of energy, or from 10^{-2} to $10^{1}\,\mathrm{s^{-1}}$ in terms of mean shear) here, low shear rate conditions will be carefully studied. Generally, previous studies have focused on $\overline{G}$ larger than $20\,\mathrm{s^{-1}}$ because of technical constrains (Spicer et al., 1996; Serra et al., 1997; Liem et al., 1999, 2000) and did not account for the coupling between the low shear rate and particle concentration-dependent regimes of the aggregation/breakup processes. As the median is less than the Kolmogorov microscale $\eta=(v^3/\varepsilon)^{1/4}$ it is likely that the breakage mechanism is erosion (Biggs and Lant, 2000) and validates the use of G as an appropriate parameter for turbulence characterisation (Mikkelsen and Keiding, 2002).

2. Materials and methods

2.1. Particles

We used monodisperse sulfate polystyrene latex particles (Interfacial Dynamics Corporation, Portland, Oregon, USA) of 2.1 μm in diameter (standard deviation of 0.037 μm) as primary particles. Because of the sizes of the particles, Brownian motion would be relevant in the first states of the aggregation process. The particles had a density of $1055\,\mathrm{kg\,m^{-3}}$. Thus, during coagulation experiments, a density-matched aqueous solution, created by adding 99.5% purity NaCl to ultrapure water (Milli-Q-water, Millipore, Bedford, MA), was used to reduce the Debye–Hückel length to a value of $10^{-8}\,\mathrm{m}$. Density matching prevented particles from settling out of suspension during and after an experiment and reduced gravity and turbulent acceleration-induced coagulation of different sized aggregates. The density difference within the resulting salty water and particles was low enough ($<10\,\mathrm{kg\,m^{-3}}$) that the calculated ratio of settling velocity to turbulent integral scale velocity was negligible for all experimental conditions. Therefore, coagulation due to differential settling of particles was negligible.

2.2. Turbulence generating devices

In the set of oscillating grid experiments, the device was similar to that described in Peters and Gross (1994). Grids were made of stainless steel coated with a plastic polyamide. Movement was provided by four independently controlled motors, each attached to a frame holding two grid shafts. Thus, replicate containers (working height of 0.153 m and diameter of 0.129 m) for up to four different levels of grid movements could be used. Grids moved up and down inside 2 L plexiglass containers. Oscillating frequencies were of 1.49, 1.51, 3.37 and 15.05 r.p.m. and oscillating amplitude was 10 cm for the lowest frequency setting and 14 cm for the others.

In this case, the bulk turbulent parameters, i.e., turbulent kinetic energy dissipation rate, ε, and mean shear rate, $\overline{G}$, can be calculated based on the drag force F_{D} produced by the mixing device as follows:

$$F_{\mathrm{D}} = \tfrac{1}{2}\overline{U_{\mathrm{R}}^{2}}C_{\mathrm{D}}A_{\mathrm{S}}\rho, \tag{1}$$

$$\bar{\varepsilon} = \frac{E}{\rho VT}, \tag{2}$$

$$\overline{G} = \sqrt{\frac{\bar{\varepsilon}}{v}}, \tag{3}$$

where $\overline{U_{\mathrm{R}}}$ is the relative mean velocity between mixing device (in this case, the grid) and fluid, C_{D} is the grid drag coefficient, A_{S} is the grid solid area, ρ is the fluid density, E is the energy generated by the grid in one cycle, V is the fluid volume and T is the period of the oscillating grid. As pointed out by Liem et al. (1999) in the case of vertically oscillating grid mixing, the average mean horizontal velocity, $\overline{U}$, and the average mean vertical velocity, $\overline{V}$, at any cross-section within the container are practically zero and in this case, the energy E in Eq. (2) can be calculated as

$$E = 4\int_{0}^{T/4} F_{\mathrm{D}}\overline{U_{\mathrm{R}}}(t)\mathrm{d}t, \tag{4}$$

where $\overline{U_{\mathrm{R}}}(t) = U_{\max}\sin(\omega t)$ with $U_{\max}$ the vertical grid speed calculated according $U_{\max} = \omega A/2$, with ω the angular frequency of the grid and A the stroke length of the grid.

In Eq. (1) the grid drag coefficient C_{D} is calculated based on the literature (White, 1974; Peters and Gross, 1994) as a combination of the grid geometry and the rod Reynolds number (R_{d})

$$C_{\mathrm{D}} = 1 + 10R_{\mathrm{d}}^{-2/3}, \tag{5}$$

$$R_{\mathrm{d}} = \frac{\overline{U_{\mathrm{R}}}d}{v}, \tag{6}$$

where d is the diameter of the grid and v is the kinematic viscosity. The respective levels of turbulence levels calculated following (1)–(6) give $\overline{G}$ of 0.70, 1.10, 3.30 and 27.36 s^{-1}.

The second apparatus consisted of a standard spherical flask (with flat bottom) of 4 L (3 L of fluid) placed on a orbital shaker, which oscillated with an orbit of 3 cm. In this case the energy dissipation, in the analogy of stirred tanks with fluid mixed by impellers (Spicer and Pratsinis, 1996b; Biggs and Lant, 2000), may be characterized by

$$\bar{\varepsilon} = \frac{P_{\mathrm{o}}N^{3}D^{5}}{V}, \tag{7}$$

where P_{o} is defined as the orbital shaker power number, N is the orbital shaker speed, V is the fluid volume and D is the diameter of the orbit. P_{o} is obtained from Zirbel et al. (2000) who quantified levels of fluid velocity and shear stress in flasks using digital particle image velocimetry. Although few data are presented, for an orbit of 2.54 cm, 125 mL Erlenmeyer flasks containing a volume of 60 mL and oscillation rates of 75 r.p.m. and 120 r.p.m., it is possible to determine P_{o} to be 9.96×10^{-5} given the measured values of ε by these authors . Then, according to (3) and (7), for a volume V of 3 L, and N between 60 and 184 r.p.m. $\overline{G}$ in this experiment ranges from 0.45 to 2.4 s^{-1}.

2.3. Coagulation experimental procedure

The particle volume fraction was set to $\phi = 0.2, 0.8, 2, 4, 6, 8$ and 10×10^{-5}, corresponding to mass concentrations of 2.1, 8.4, 21, 42, 63, 84, and 105 mg L^{-1}. This range was chosen as wide as possible and only limited for technical reasons. Samples were withdrawn from the bulk of the tank. This was done gently with a pipette of large orifice (diameter of 4 mm) in order to avoid aggregate breakage when sampling.

Four preliminary experiments at $\phi = 2 \times 10^{-5}$ were performed for aggregation of particles in the oscillating grid tank with sample points spaced 0.05 m in the vertical. No differences in the particle diameter attained by the particle suspension at each sampled point were found. Other studies using these apparatus have focused on characterizing the turbulence generated and demonstrate that the apparatus provides optimal characteristics for flocculation in water treatment processes (Liem et al., 2000) . We assume then that in the turbulent domain created by the grids, particle aggregates experience a continuous change of their fluid neighborhood because of crossing from one eddy to another (Atkinson and Wolcott, 1990; De Silva and Fernando, 1994). So for in all of experiments a single point was measured, 0.05 m below the water surface. In the experiments with the spherical flask placed in the shaker table samples were taken at the center of the fluid volume.

Floc diameter was analyzed from the samples using a Coulter Counter using a 70 μm nominal aperture diameter, which provided measurements over a particle diameter range of 1.4–42.0 μm. The particle counter determines the number and size of particles suspended in an electrically conductive liquid. Changes in the resistance between two electrodes immersed in the liquid are caused by particles passing through a small aperture lying between the two electrodes (Andreadakis, 1993).

The resistance linked to voltage pulses is proportional to particle size and the median size is calculated from the population distribution. It should be mentioned that the Coulter Counter is considered to represent the solid volume of aggregates (Serra and Logan, 1999) and for particle size distributions of bacteria and for microsphere aggregates differs to the actual length of aggregates (either measured by image analysis or light scattering). Serra and Logan (1999) report actual length of aggregates 2.22–2.45 larger than solid length of aggregates for aggregates of 10 μm (solid length).

Finally, the time evolution of the particle aggregation is quantified by the nondimensional time, t^*, which depends on the shear, $\overline{G}$, and on the initial particle volume fraction, ϕ, as $t^* = t\bar{G}\phi\cdot$ It is chosen because this nondimensional time represents a normalized number of collisions taking place in the system (Oles, 1991; Serra et al., 1997).

3. Results and discussion

Aggregate formation rate increases with increasing energy dissipation. The growth in the aggregate size was faster at higher shearing rates in the beginning of the aggregation process (Fig. 1). As the characteristic diameter, d, we use the median of the size distribution (with a confidence of 95%) with respect to aggregate volume. For all cases, at larger t^*, the breakup of aggregates is more pronounced until it balances the aggregation, and the steady state is then reached.

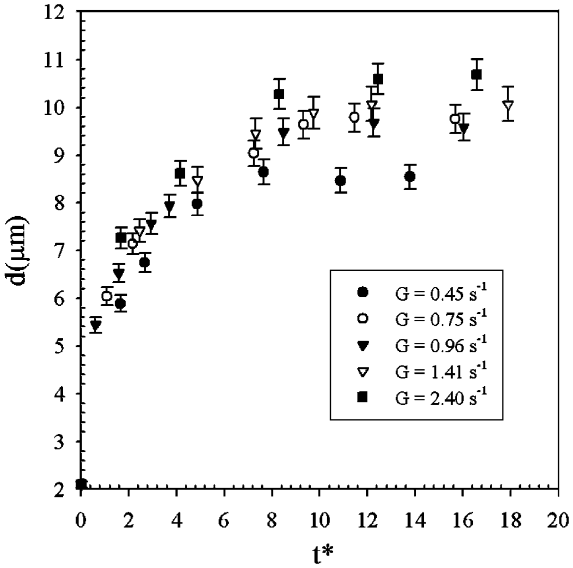

Fig. 1. Evolution of the median aggregate size d with nondimensional time t^* for different shear rates (shaking table experiments) for an initial volume fraction $\phi = 2 \times 10^{-5}$. The diameter of the initial distribution of particles (d_o) is 2.1 μm for all cases.

Usually, the behavior for $\overline{G} > 20\,\mathrm{s}^{-1}$ has been investigated by other authors such as Parker et al. (1972), Galil et al. (1991), Oles (1991), Serra et al. (1997), Liem et al. (2000), Biggs and Lant (2000) and Selomulya et al. (2001). On the contrary, the range $\overline{G} < 20\,\mathrm{s}^{-1}$ has been little studied. Selomulya et al. (2001) report a similar steady-state size for a suspension of 0.38 μm-latex particles using a flow impeller at average shear rates of 16 and 32 s^{-1}. The initial concentration was $\phi = 3.8 \times 10^{-5}$ and in their experiments d/d_o was in the order of 10^3 which can be related to the small particles used for aggregation, as in the case of Spicer et al. (1996). These results concur with the observations of Serra et al. (1997) and Selomulya et al. (2004), that larger primary particles (d_o) brings about a decrease in d/d_o. Parker et al. (1972) and Biggs and Lant (2000) based on experiments of flocculation of activated sludge in baffled batch vessels with blade impellers pointed out that for shear rates larger than 19 s^{-1} a relationship between the steady-state aggregate size and the shear could be represented by the power law

$$d = C\overline{G}^{-a}, \tag{8}$$

where C is a floc strength component and a is the stable floc size component. They found a large variability of a for activated sludge flocs between 0.15 and 0.71, depending on the strength of fracture for aggregates. However, the power law relationship produces very large aggregate diameters at decreasing shear rates with the paradoxical result of huge aggregates at no shear. Our results demonstrate that shear provided a means to keep the particle number count high for collisions to occur but it is small enough that the aggregation balance is dominated by aggregation (by the time the balance between aggregation and breakup prevails). Such results imply that there should be a range of intermediate shear rates where particle aggregation produces large aggregates, which until now has been poorly studied.

The relationship between the steady-state values of d at different $\overline{G}$ for the four particle volume concentrations used in the experiments with the oscillating grid and the orbital shaker are shown in Figs. 2 and 3, respectively. For all volume concentrations the final nondimensional diameter of the aggregate increases as shear enhances the aggregation process. As volume fraction increases, the aggregation of particles is also enhanced and the final diameter of the aggregate increases. Our results also show that for a given $\overline{G}$, mixing in the spherical flasks produces larger aggregates, which implies a better efficiency, probably as a result of different mixing qualities.

The steady-state aggregate size in the oscillating grid experiments is found to increase when the initial particle volume concentration increases (Fig. 4). Although the

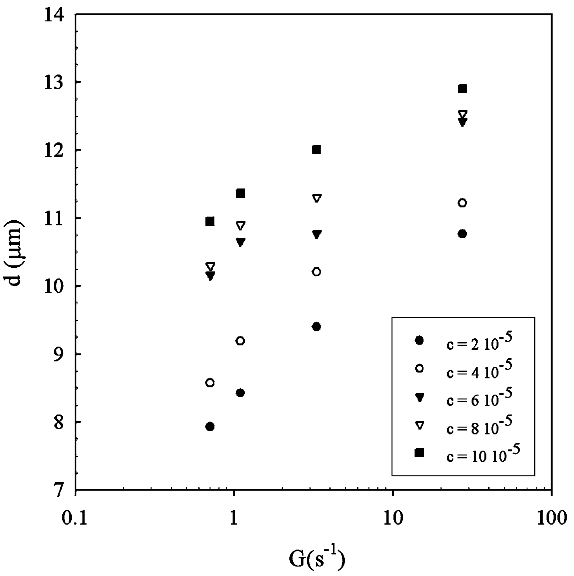

Fig. 2. Representation of the steady-state diameter of aggregate, for the range of $\overline{G}$ studied in the tank with the grid oscillating through the whole volume, at different volume fractions.

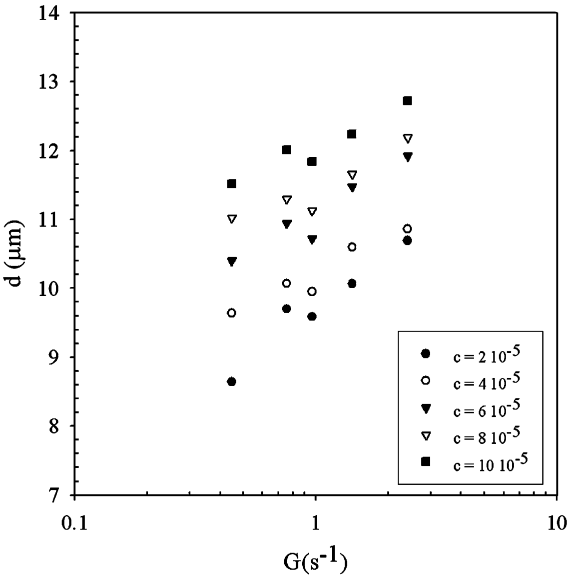

Fig. 3. Representation of the steady-state diameter of aggregate, for the range of $\overline{G}$ studied in the spherical flask on a shaking table, at different volume fractions.

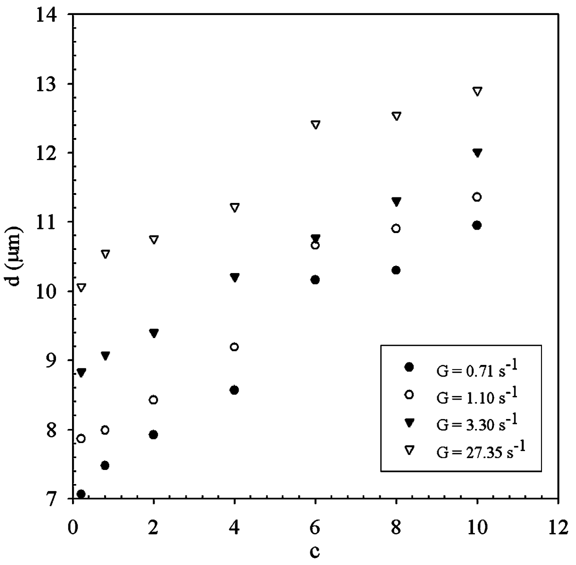

Fig. 4. Evolution of d with ϕ (0.2, 0.8, 2, 4, 6, 8 and 10×10^{-5}) for the $\overline{G}$ used in the oscillating grid experiment.

Coulter Counter technique used in our measurements gives smaller sizes of aggregates than image analysis and light scattering (in the order of $\frac{1}{2}$, Serra and Logan (1999)), it is worthwhile to point out that our results are similar to floc growth of activated sludge concentrations between 3.5 and 140 mg/L reported by Chaignon et al. (2002) working at a $\overline{G} = 135\,\mathrm{s}^{-1}$, to the results of flocculation of polystyrene particles and aluminum sulfate reported by Spicer and Pratsinis (1996b) working at $\overline{G} = 63\,\mathrm{s}^{-1}$ and concentrations of 4.3 and 3 mg L^{-1}, to the aggregation of polystyrene particles in a Couette flow, for a range of shear rates between 25 and 50 s^{-1} (Serra et al. 1997), and to the results of aggregation of polystyrene particles and sodium chloride in a six-bladed Rushton disc turbine impeller reported by Kusters et al. (1997) working at $\overline{G} = 163\,\mathrm{s}^{-1}$ and concentrations of $\phi = 3.1\times10^{-5}$, 6.2×10^{-5} and 2.1×10^{-4}.

It is worth noting that, in aquatic systems, turbulence ranges from 0.01 to 10 s^{-1}. Typical values of $\overline{G}$ are 0.01–1 s^{-1} in open oceans, 0.3–10 s^{-1} in coastal zones (Kiørboe and Saiz, 1995), and 0.1–3 s^{-1} in lakes (Estrada and Berdalet, 1997). In our study, when $\overline{G}$ ranged between 0.45 and 2.40 s^{-1} in the spherical flasks and between 0.70 and 27.36 s^{-1} in the cylindrical tanks, the associated energy dissipation calculated gives $\bar{\varepsilon}$ in the orders of 2×10^{-3}–5×10^{-2} cm^2 s^{-3} in the first set of experiments and of 5×10^{-3}–7.5 cm^2 s^{-3} in the second set which lies in the range of medium to high field turbulence taken from the oceanographic literature. Our results demonstrate the effect of increasing flocculation at increasing levels of turbulence and that particle concentration should be taken into account when studying the dynamics of sediment particles and small phytoplankton populations suspended in a fluid under the action of turbulence mixing. General food web models that take into account particle aggregation processes (Jackson, 2001) should take advantage of knowing the median aggregate size that is achieved under a certain shear stress condition.

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