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THE EFFECT OF KAOLIN CONCENTRATION ON FLOCK GROWTH KINETICS IN AN AGITATED TANK

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Abstract. This paper reports on an investigation of the effect of initial solid particle concentration on flock growth and flock shape characterized by the fractal dimension D_{f2}. The experiments were carried out in a baffled tank agitated by a Rushton turbine at mixing intensity 64 W/m^3 and constant dimensionless flocculant dosage $D_F/c_{k0} = 4.545$ mg/g. The model wastewater (a suspension of tap water and kaolin) was flocculated with Sokoflok 56A organic flocculant (solution 0.1 % wt.). The size and shape of the flocks were investigated by image analysis. The flock growth kinetics was fitted according to a semi-empirical generalized correlation proposed by the authors. The dependences $A_f^* = 100.35c_{k0}^{1.532}$, $d_{f,eq,max} = 1.0474c_{k0}^{-0.311}$ and $(Nt_F)_{max} = 1622c_{k0}^{-0.393}$ were found. The fractal dimension D_{f2} was found to be independent of flocculation time and initial kaolin concentration, and its value $D_{f2} = 1.470 \pm 0.023$ was determined as an average for the given conditions.

Keywords: flocculation, flock growth, flock size, mixing, Rushton turbine, kaolin slurry, fractal geometry, image analysis.

1. Introduction

Flocculation is one of the most important operations in solid–liquid separation processes in water supply and wastewater treatment (e.g. [1–3]). The purpose of flocculation is to transform fine particles into coarse aggregates, flocks, which will eventually settle, in order to achieve efficient separation.

The properties of the separated particles have a major effect on the separation process and on the separation efficiency in a solid–liquid system. The solid particles in a common solid–liquid system are compact and are regular in shape, and their size does not change during the process. The size of these particles is usually sufficiently described by the diameter, or by some equivalent diameter (e.g. [4]). However, the flocks that are generated during flocculation are often porous and are irregular in shape, and this complicates the design of the flocculation process. Flock properties such as size, density and porosity affect the separation process and its efficiency. The flock growth kinetics, and the effects of flocculant dosage and mixing intensity on flock size during flocculation in an agitated vessel were investigated in our previous research [5–7].

The aim of this work is to find the effect of initial solid particle concentration on flock growth kinetics and flock shape characterized by fractal dimension. The experiments were carried out in a baffled tank agitated by a Rushton turbine at mixing intensity 64 W/m^3 and constant dimensionless flocculant dosage $D_F/c_{k0} = 4.545$ mg/g. The model wastewater (a suspension of tap water and kaolin) was flocculated with Sokoflok 56A organic flocculant (solution 0.1 % wt.). The size and shape of the flocks were investigated by image analysis.

2. Theoretical background

The dependence of flock size on flocculation time can be expressed by a simple empirical formula, taking into account flock breaking [5]:

$$\frac{1}{d_f} = A_f \log^2(Nt_F) + B_f \log(Nt_F) + C_f, \qquad (1)$$

where d_f is flock size, Nt_F is dimensionless flocculation time, t_F is flocculation time, N is impeller rotational speed and A_f, B_f, C_f are model parameters.

Based on Eq. (1), a generalized correlation for flock growth kinetics in an agitated tank that takes into account flock breaking can be rewritten as follows [5]:

$$\Delta\left(\frac{1}{d_f}\right)^* = A_f^*\left(\Delta(Nt_F)^*_{\log}\right)^2 \qquad (2)$$

or

$$\frac{d_{f,max}}{d_f} = 1 + A_f^*\left(\Delta(Nt_F)^*_{\log}\right)^2, \qquad (3)$$

where

$$\Delta\left(\frac{1}{d_f}\right)^* = \frac{1/d_f - 1/d_{f,max}}{1/d_{f,max}}, \qquad (4)$$

$$\Delta(Nt_F)^*_{\log} = \frac{\log(Nt_F) - \log(Nt_F)_{max}}{\log(Nt_F)_{max}}, \qquad (5)$$

$$A_{\mathrm{f}}^{*} = \frac{B_{\mathrm{f}}^{2}}{4A_{\mathrm{f}}C_{\mathrm{f}} - B_{\mathrm{f}}^{2}}, \tag{6}$$

where $d_{\mathrm{f,max}}$ is the maximum flock size reached at dimensionless time $(Nt_{\mathrm{F}})_{\mathrm{max}}$, A_{f}^{*} is the flock size shift coefficient, $\Delta(1/d_{\mathrm{f}})$ and $\Delta(Nt_{\mathrm{F}})_{\mathrm{log}}^{*}$ are the variables defined by Eqs. (4) and (5), and $A_{\mathrm{f}}, B_{\mathrm{f}}, C_{\mathrm{f}}$ are the parameters of Eq. (1). The generalized correlation parameters $d_{\mathrm{f,max}}, (Nt_{\mathrm{F}})_{\mathrm{max}}$ and A_{f}^{*} generally depend on the flocculation process conditions, such as mixing intensity, flocculation dosage and initial solid particle concentration. The flock growth kinetics model was successfully tested on published experimental data [8] and was also successfully adopted for quantifying the effect of flocculant sonication on the flock growth kinetics occurring in an agitated vessel [9]. In the experiments, a model suspension of commercial chalk dust in distilled water and an industrial suspension of ultrafine coal particles were used.

3. Experimental

3.1. Experimental apparatus

The flocculation experiments were conducted in a fully baffled cylindrical vessel of diameter $D = 150$ mm with a flat bottom and 4 baffles per 90°, filled to height $H = D$ with a model wastewater (tap water + kaolin particles) (Fig. 1). The model wastewater was agitated by a Rushton turbine (RT) $d = 60$ mm in diameter placed at an off-bottom clearance of $H_{\mathrm{t}}/d = 0.85$. The baffle width B/D was 0.1. The impeller motor and the Cole Parmer Servodyne model 50000-25 speed control unit were used in our experiments. The impeller speed was set up and the impeller power input value was calculated using the impeller power characteristics.

The flock size was determined using a non-intrusive optical method. The method is based on an analysis of the images obtained by a digital CMOS camera in a plane illuminated by a laser light. The method consists of the following steps:

(1.) illuminate a plane in the tank with a laser light sheet (also called a laser knife) in order to visualize the flocks;

(2.) record images of the flocks using a camera;

(3.) process the images captured by the image analysis software.

The illuminated plane is usually vertical, and a camera is placed horizontally (e.g. [10, 11]). Kysela and Ditl [11] used this technique for observing flocculation kinetics. They found that the application of this method is limited by the optical properties of the system. The required transparency limits the maximum particle concentration in the system. We determine the flock size not during flocculation but during sedimentation. Thus this limitation should be overcome. Therefore, the laser-illuminated plane is horizontal and perpendicular to the impeller axis. The scheme of the experimental apparatus for image analysis is shown in Fig. 1. The agitated vessel was placed in an optical box (a water-filled rectangular box). The optical box reduces laser beam dispersion, and thus it improves the optical properties of the measuring system. The camera with the objective and the laser diode are placed on the laboratory support stand. The hardware & software installation and setup are described in detail in [12]. The technical parameters are presented in Tab. 1.

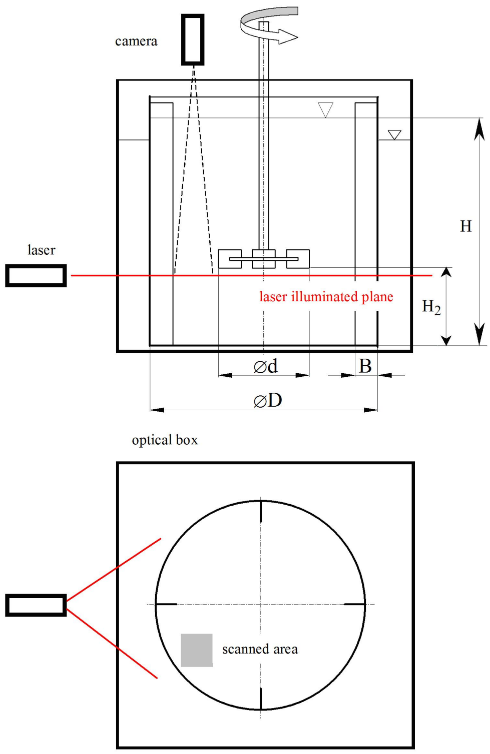

Figure 1. Scheme of the experimental apparatus for image analysis.

3.2. Experimental procedure

The maximum flock sizes formed during flocculation were measured during sedimentation. The experimental parameters are summarized in Tab. 2.

The experimental procedure was as follows:

(1.) **Calibration and experimental apparatus setting.** The calibration grid was placed in an illuminated plane and the camera was focused manually onto this calibration grid before each flocculation experiment. Then the image of the calibration grid was recorded. For camera resolution 800 × 800 pixels, the scale 1 px ∝ 45 µm was found for our images. This corresponds to a scanned area of 35 mm × 35 mm (approx. 6 % of the cross-section area of the tank). The scanned area was located in the middle of one quarter of the vessel, between the vessel wall and the impeller.

(2.) **Model wastewater preparation.** Kaolin slurry (a suspension of water and kaolin particles [18672 Kaolin powder finest Riedel-de Haen]) was

Item	Specification
Laser diode	NT 57113, 30 mW, wave length 635 nm (red light), Edmund Optics, Germany
Diode optics	optical projection head NT54-186, Projection Head Line, Edmund Optics, Germany
Camera	colour CMOS camera SILICON VIDEO®SI-SV9T001C, EPIX Inc., USA
Camera optics	objective 12VM1040ASIR 10–40 mm, TAMRON Inc., Japan
Image processing card (so-called grabbing card)	PIXCI SI PCI Image Capture Board, EPIX Inc., USA
Camera control software	XCAP®, EPIX Inc., USA
Operation software	Linux CentOS version 5.2, Linux kernel 2.6
Software for image analysis	SigmaScan Pro 5.0

Table 1. Technical parameters.

Parameter		Experiment
ϵ_V	[W/m^3]	64
N	[rpm]	210
t_F	[min]	3.43, 5.71, 8, 11.4, 17.1
Nt_F	[–]	720, 1200, 1680, 2400, 3600
c_{k0}	[mg/l]	440, 560, 640
D_F/c_{k0}	[mg/g]	4.545
D_F	[ml/l]	2, 2.55, 2.91
No. of exp. points		15

Table 2. Experimental conditions

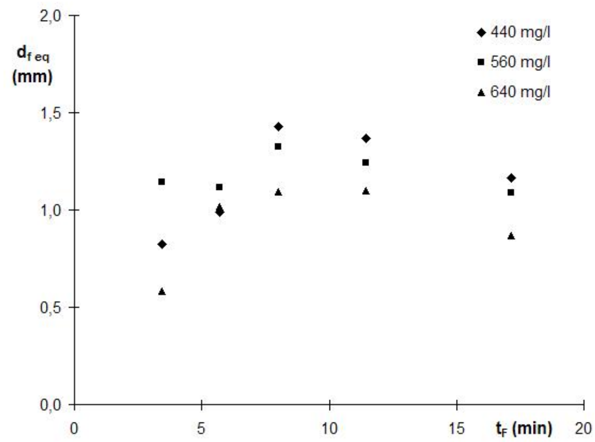

Figure 2. Experimental data — Maximum flock size vs. flocculation time — $d_{f,eq} = f(t_F)$.

used as a model system. The solid concentration of kaolin was 440, 560 and 640 mg/l.

(3.) **Flocculation.** The model wastewater was flocculated using Sokoflok 56A organic polymer flocculant (medium anionity, 0.1 % wt. aqueous solution; flocculant weight per flocculant solution volume $m_F/V_F = 1$ mg/ml; Sokoflok Ltd., Czech Republic). The experimental conditions are specified in Tab. 2. The flocculation was initiated by adding flocculant into the agitated vessel, and the flocculation time measurement was started.

(4.) **Image acquisition.** After impeller shutdown the flocks began to settle. During sedimentation, the images of flocks passing through the illuminated plane were captured. The camera was set to 10-bit depth at frame rate 10 s^{-1}, exposure 5 ms and gain 35 dB. Image capturing started 20 s after impeller shutdown and took 120 s. Finally 1200 images were obtained for the flocculation experiment and some were stored in a hard disk in 24-bit jpg format.

(5.) **Image analysis.** The images were analyzed using SigmaScan software and its pre-defined filters (filter No. 8 for removing one-pixel points, filter No. 10 for removing objects touching on an image border, and filter No. 11 for filling an empty space caused by capture error in identified objects and our macros (Svačina [13] in detail).

3.3. Experimental data evaluation

From the images that were captured, the largest flock was identified and its projected area was determined for the given flocculation time. The equivalent diameter calculated according to the flock area plotted in dependence on the flocculation time for a given dimensionless flocculant dosage and various initial kaolin concentrations is shown in Fig. 2. When the flocculation time increases, the flock size increases up to the maximum value, due to primary aggregation, and then decreases due to flock breaking.

The experiments were conducted at dimensionless flocculant dosage D_F/c_{k0} =const. We expected that

c_{k0} [mg/l]	N [rpm]	$(Nt_F)_{max}$ [–]	$t_{F,max}$ [min]	$d_{f,eq,max}$ [mm]	A_f^* [–]	I_{yx} (a) [–]	$\delta_{r,ave}/\delta_{r,max}$ (b) [%]
440	210	2252	10.7	1.334	30.379	0.986	3.6/7.3
560	210	2006	9.6	1.304	34.631	0.999	0.2/0.3 (c)
640	210	1952	9.3	1.174	56.723	0.986	2.1/10.4

(a) Correlation index.
(b) Relative error of equivalent flock size $d_{f,eq}$: average/maximum absolute value.
(c) Outlier flock size for $Nt_F = 720$ was excluded from the evaluation.

Table 3. Generalized correlation $\Delta(1/d_{f,eq})^* = f(\Delta(Nt_F)^*_{log})$: parameters fitted.

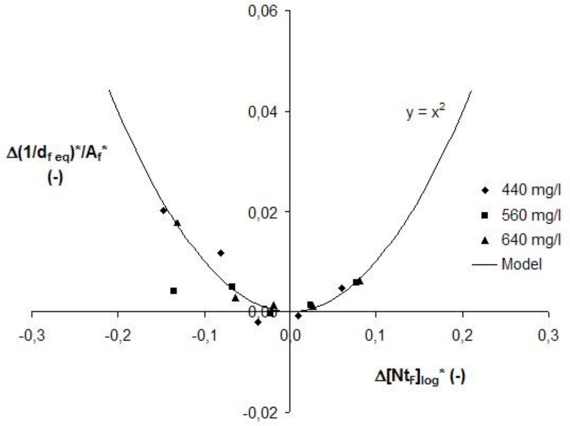

Figure 3. Generalized correlation $\Delta(1/d_{f,eq})^* = f(\Delta(Nt_F)^*_{log})$.

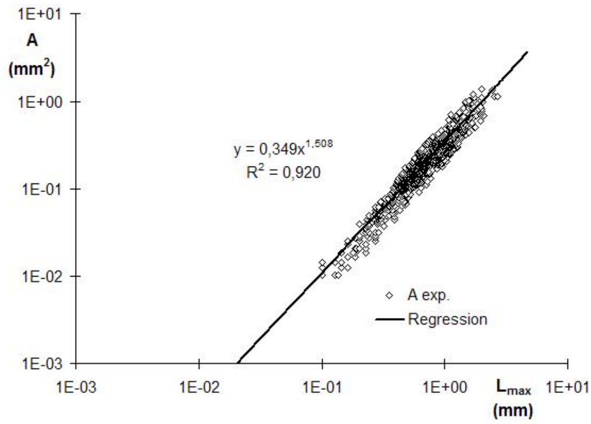

Figure 4. Fractal dimension determination — an example for $Nt_F = 1680$ and $c_{k0} = 560$ mg/l.

the effect of initial solid particle concentration could be eliminated by satisfying this condition, and thus the same flock growth curves would be observed. This assumption was not clearly confirmed by the experiment.

3.3.1. Generalized correlation $\Delta(1/d_{f,eq})^* = f(\Delta(NtF)^*_{log})$

The dependence of calculated equivalent diameter $d_{f,eq}$ on flocculation time was fitted according to the generalized correlation (2). The generalized correlation parameters are presented in Tab. 3. A comparison of the experimental data and the generalized correlation is depicted in Fig. 3.

3.3.2. Fractal dimension

The flocks generated are often porous and have an irregular shape [14, 15], which complicates the design of the flocculation process. Fractal geometry [16] is a method that can be used for describing the properties of irregular objects. Some flock properties, e.g. shape, density and sedimentation velocity can be described on the basis of fractal geometry. A fractal dimension of the 3rd order D_{f3} evaluated from the flock mass was usually determined. Since we were measuring the projected area of the flocks, the fractal dimension of the 2nd order D_{f2} was used for characterizing the flock shape. The dependence of the projected area A on characteristic length scale L_{char} and on the fractal dimension D_{f2} is given by:

$$A = CL_{char}^{D_{f2}}. \quad (7)$$

The largest flocks determined in the images were used for estimating the fractal dimension. The maximum flock size was used as a characteristic length scale. The fractal dimension D_{f2} was determined for each flocculation time and initial kaolin concentration. For illustration, the dependence of the projected area on the maximum flock size is shown in Fig. 4 for dimensionless flocculation time $Nt_F = 1680$. The fractal dimension D_{f2} plotted in dependence on flocculation time for a given flocculant dosage and mixing intensity is shown in Fig. 5.

The effect of flocculation time on fractal dimension D_{f2} was tested by hypothesis testing. The statistical method of hypothesis testing can estimate whether the differences between the parameter values that are predicted (e.g. by some proposed theory) and the parameter values that are evaluated from the measured data are negligible. In this case, we assumed the dependence of fractal dimension D_{f2} on dimensionless flocculation time, described by the simple power law $D_{f2} = B(Nt_F)^{\beta}$, and the difference between predicted exponent β_{pred} and evaluated exponent β_{calc} was tested. The hypothesis test characteristics are

c_{k0} [mg/l]	m [–]	t-distribution[a] $t_{m-2,\alpha=0.05}$	Relation $D_{f2} = B.(NtF)^{\beta}$ β_{calc}	t-characteristics $\lvert t\rvert$ Hypothesis $D_{f2} = B(Nt_F)^0$ $\beta_{pred} = 0$	Average value D_{f2} [–]
440	5	3.1825	0.016	1.1 (acceptable)	1.470 ± 0.041
560	5	3.1825	0.015	0.7 (acceptable)	1.476 ± 0.057
640	5	3.1825	0.002	0.1 (acceptable)	1.463 ± 0.073

[a] The t-distribution for $m-2$ degrees of freedom and significance level $\alpha = 0.05$.

Table 4. Fractal dimension — effect of flocculation time.

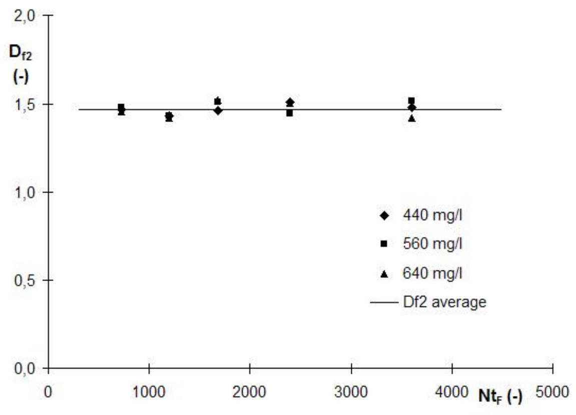

Figure 5. Effect of initial kaolin concentration on the fractal dimension.

given as $t = (\beta_{calc} - \beta_{pred})/s_{\beta}$, where s_{β} is the standard error of parameter β_{calc}. If the calculated $|t|$ value is less than the critical value of the t-distribution for $(m-2)$ degrees of freedom and significance level α, the difference between β_{calc} and β_{calc} is statistically negligible (statisticians state: "the hypothesis cannot be rejected"). The hypothesis test result and parameter β evaluated from the data are presented in Tab. 4. The fractal dimension was found to be independent of flocculation time for all three initial kaolin concentrations.

4. Effect of initial solid particle concentration

We found that the model parameters $A_f^*, d_{f,eq,max}$ and $(Nt_F)_{max}$ depend on the initial kaolin concentration for the given constant dimensionless flocculant dosage D_F/c_{k0}. The relations can be described simply as follows:

$$A_f^* = 100.35 c_{k0}^{1.532} \qquad (I_{yx} = 0.883), \qquad (8)$$

$$d_{f,eq,max} = 1.0474 c_{k0}^{-0.311} \qquad (I_{yx} = 0.869), \qquad (9)$$

$$(Nt_F)_{max} = 1622 c_{k0}^{-0.393} \qquad (I_{yx} = 0.984). \qquad (10)$$

Finally, the effect of initial kaolin concentration on fractal dimension D_{f2} was tested in the same manner. The power exponent -0.009 and t-characteristics $|t| = 0.2$ were evaluated from the data. Based on this, the fractal dimension was found to be independent of the initial kaolin concentration, and the value $D_{f2} = 1.470 \pm 0.023$ was determined as an average value for the given conditions.

5. Conclusions

The following results have been obtained:

- The effect of initial solid particle concentration on flock growth and shape was investigated in a baffled tank agitated by a Rushton turbine at mixing intensity 64 W/m^3 and constant dimensionless flocculant dosage $D_F/c_{k0} = 4.545\,\mathrm{mg_F/g_K}$. Flock size and flock shape were investigated by image analysis.
- The experiments were carried out on a kaolin slurry of initial particle concentration 440, 560 and 640 mg/l as a model wastewater. The model wastewater was flocculated with Sokoflok 56A organic flocculant (solution 0.1 % wt.).
- The largest flock was identified from the obtained images, and its projected area was determined for a given flocculation time. The calculated equivalent diameter plotted in dependence on flocculation time for the given initial kaolin concentration is shown in Fig. 2.
- The flock size increases with increasing flocculation time up to a maximum value due to primary aggregation, and then decreases due to flock breaking. The flock growth curves are similar and shifted for various initial kaolin concentrations. With increasing particle concentration, the flock sizes are smaller for flocculation time > 6 minutes.
- The flock growth kinetics were fitted according to a semi-empirical generalized correlation proposed by the authors. A comparison of the experimental data and the generalized correlation is depicted in Fig. 3. The dependences $A_f^* = 100.35 c_{k0}^{1.532}, d_{f,eq,max} = 1.0474 c_{k0}^{-0.311}$ and $(Nt_F)_{max} = 1622 c_{k0}^{-0.393}$ were found.
- The fractal dimension D_{f2} was determined for each flocculation time on the basis of the experimental data. Using the statistical hypothesis test, the fractal dimension D_{f2} was found to be independent of flocculation time and initial kaolin concentration, and its value $D_{f2} = 1.470 \pm 0.023$ was determined as an average for the given conditions.

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