Flock Growth Kinetics for Flocculation in an Agitated Tank

R. Šulc, O. Svačina

Abstract

Flock growth kinetics was investigated in baffled tank agitated by a Rushton turbine at mixing intensity 40 W/m^3 and kaolin concentration 0.44 g/l. The tests were carried out with a model wastewater (a suspension of tap water and kaolin). The model wastewater was flocculated with organic Sokoflok 56 A flocculant (solution 0.1 % wt.). The flock size and flock shape were investigated by image analysis. A simple semiempirical generalized correlation for flock growth kinetics was proposed, and was used for data treatment. The flock shape was characterized by fractal dimension D_{f2}. Using the statistical hypothesis test, the fractal dimension was found to be independent of flocculation time, and the value $D_{f2} = 1.442 \pm 0.125$ was determined as the average value for the given conditions.

Keywords: flocculation, flock growth, flock size, mixing, Rushton turbine, kaolin slurry.

1 Introduction

Flocculation is one of the most important operations in solid – liquid separation processes in water supply and wastewater treatment. The purpose of flocculation is to transform fine particles into coarse aggregates – flocks that will eventually settle – in order to achieve efficient separation.

The properties of separated particles have a major effect on the separation process and on 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.

The flocks that are generated are often porous and are irregular in shape, which complicates the flocculation process design. Flock properties such as size, density and porosity affect the separation process and its efficiency.

The aim of this work is to propose a simple semiempirical generalized correlation for flock growth kinetics and to verify the proposed model experimentally.

2 Generalized correlation for flock growth kinetics

Turbidity measurement has been used and recommended for flocculation performance assessment in routine control in industry. Flocculation efficiency has frequently been expressed as the rate of turbidity removal:

$$Z_e^*(t_F) = \frac{Z_e(t_F)}{Z_0} = \frac{Z_0 - Z_r(t_F)}{Z_0} = 1 - Z_r^*(t_F) \quad (1)$$

where Z_e^* is turbidity removal degree, Z_r^* is residual turbidity degree, Z_0 is turbidity of a suspension before

the beginning of flocculation, Z_e is eliminated turbidity due to flocculation, Z_r is residual turbidity of clarified water after flock separation, and t_F is flocculation time.

Šulc [1] found that flocculation kinetics expressed as the dependence of the residual turbidity rate on the flocculation time can be expressed by a simple formula, taking into account flock breaking:

$$Z_r^* = A_{Zr^*} \cdot \log^2(t_F^*) + B_{Zr^*} \cdot \log(t_F^*) + C_{Zr^*} \quad (2)$$

where Z_r^* is residual turbidity degree, t_F^* is dimensionless flocculation time, and A_{Zr^*}, B_{Zr^*}, C_{Zr^*} are the model parameters. For flocculation taking place in an agitated tank, Šulc and Ditl [2] recommend dimensionless flocculation time t_F^* given by:

$$t_F^* = n \cdot t_F \quad (3)$$

where t_F is flocculation time, and n is impeller rotational speed. The proposed definition of dimensionless flocculation takes this into account due to the characteristic time choice. The chosen characteristic time, $t_{char} \propto 1/n$, is proportional to the number of liquid passages through an impeller.

Based on Eq. (2) Šulc [1], Šulc and Ditl [3] have proposed a generalized correlation for flocculation kinetics in an agitated tank that takes into account flock breaking, as follows:

$$\Delta Z_r^* = A_{Zr^*}^* \cdot \left(\Delta[nt_F]_{\log}^*\right)^2 , \quad (4)$$

where

$$\Delta Z_r^* = \frac{Z_r^* - Z_{r_{\min}}^*}{Z_{r_{\min}}^*} \quad (5)$$

$$\Delta[nt_F]_{\log}^* = \frac{\log(nt_F) - \log([nt_F]_{\min})}{\log([nt_F]_{\min})} , \quad (6)$$

where $Z_{r\ min}^*$ is minimal residual turbidity degree achieved at time $[nt_F]_{min}$, $[nt_F]_{min}$ is the dimensionless flocculation time in which $Z_{r\ min}^*$ can be achieved,

A^* is the residual turbidity shift coefficient, t_F is the flocculation time, and n is impeller rotational speed.

According to Lambert's law, the turbidity depends on the cross-sectional area of the flock σ and flock concentration N_p, as follows:

$$Z_r \propto N_p \cdot \sigma \qquad (7)$$

Assuming that the cross-sectional area of flock σ is proportional to flock size d_f

$$\sigma \propto d_f^2 \qquad (8)$$

and particle/flock mass conservation must be fulfilled at any time, flock concentration N_p can be expressed as follows:

$$N_p \cdot d_f^3 \propto \text{const.} \qquad (9)$$

Using Eqs. (7), (8) and (9), the dependence of flock size on flocculation time can be given by a simple formula taking into account flock breaking:

$$(1/d_f) = A_f \cdot \log^2(nt_F) + B_f \cdot \log(nt_F) + C_f \qquad (10)$$

where $(1/d_f)$ is reciprocal flock size, nt_F is dimensionless flocculation time, and A_f, B_f, C_f are the model parameters. Assuming that the minimum residual turbidity degree corresponds to the maximum flock size, the dimensionless model variable ΔZ_r^* can be rewritten as follows:

$$\Delta Z_r^* = \frac{Z_r^* - Z_{r_{\min}}^*}{Z_{r_{\min}}^*} = \frac{Z_r - Z_{r_{\min}}}{Z_{r_{\min}}} \propto$$
$$\frac{N_f}{N_{f_{\max}}} \cdot d_f^2 - d_{f_{\max}}^2 = \frac{(1/d_f) - (1/d_{f_{\max}})}{(1/d_{f_{\max}})} \qquad (11)$$

where d_f is flock size and d_{fmax} is maximal flock size.

Then the generalized correlation for flock growth kinetics that takes into account flock breaking can be derived as follows:

$$\Delta(1/d_f)^* = A_f^* \cdot (\Delta[nt_F]_{\log}^*)^2 \qquad (12)$$

rewritten

$$\frac{d_{f_{\max}}}{d_f} = 1 + A_f^* \cdot (\Delta[nt_F]_{\log}^*)^2 \qquad (13)$$

where

$$\Delta(1/d_f)^* = \frac{(1/d_f) - (1/d_{f_{\max}})}{(1/d_{f_{\max}})}, \qquad (14)$$

$$A_f^* = \frac{B_f^2}{4 \cdot A_f \cdot C_f - B_f^2}, \qquad (15)$$

where $d_{f_{\max}}$ is the maximum flock size reached at time $[nt_F]_{\max}$, $[nt_F]_{\max}$ is the dimensionless flocculation time in which $d_{f_{\max}}$ can be achieved, $\Delta[nt_F]_{\log}^*$ is the variable defined by Eq. (6), and A_f, B_f, C_f are parameters of Eq. (10).

The generalized correlation parameters $d_{f_{\max}}$, $[nt_F]_{\max}$ and A_f^* depend generally on the flocculation process conditions, e.g. mixing intensity and flocculation dosage.

3 Model evaluation

The proposed generalized correlation was tested on the published experimental data by Kilander et al. [4]. Kilander et al. [4] investigated the local flock size distributions in square tanks of different sizes (5, 7.3, 28 and 560 l). The areas investigated in the 7.3 and 28 l tanks were the upper left corner (noted UC), the lower left corner (noted LC), directly over the impeller (noted OI) and directly under the impeller (noted UI). In the 5 and 560 l tanks, the areas LC, OI and UI were investigated. A suspension of buffered water and kaolinite clay was used as the model flocculation system. The tanks were agitated by a Lightnin A 310 hydro foil impeller. The 5, 7.3 and 28 l tanks were operated at two specific energy inputs, 1.72 W/m^3 and 2.55 W/m^3, corresponding to average gradient velocity 41.2 and 50.4 s^{-1}, respectively. The 560 l tank was operated only at specific energy input 2.55 W/m^3. No model of flock growth kinetics was applied for data interpretation.

The data in the UC area for the 7.3 l and 28 l tanks at specific power input 1.72 W/m^3 were analyzed. A comparison of the experimental data and a generalized correlation are depicted in Fig. 1. The generalized correlation parameters fitted for the measured data are presented in Table 1.
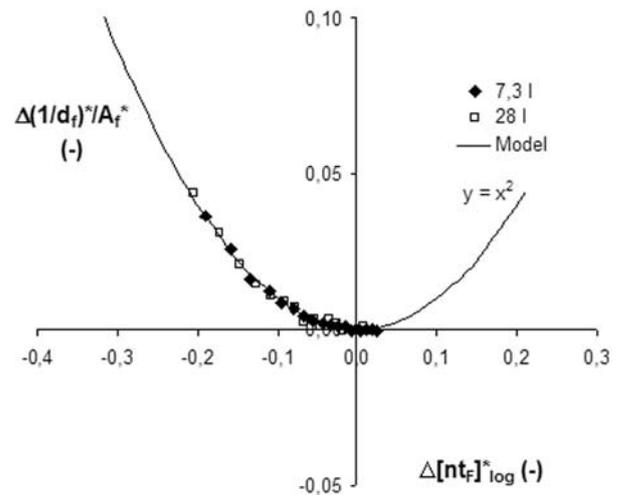
Fig. 1: Generalized correlation $\Delta(1/d_f)^* = f(\Delta[nt_F]_{log}^*)$ – Kilander et al. [4]

Table 1: Generalized correlation parameters fitted – data Kilander et al. [4]

V	ε_V	n	$[nt_F]_{\max}$	$t_{F_{\max}}$	$d_{f_{\max}}$	A_f^*	I_{yx}	$\delta_{r_{\max}}/\delta_{r_{ave}}$ [*1]
(L)	(W/m^3)	(rev/min)	(–)	(min)	(mm)	(–)	(–)	(%)
7.3	1.72	199	6 429	32.3	0.231 4	39.108	0.998 5	1.4/3.2 [*2]
28	1.72	148	5 217	35.3	0.169 9	18.998	0.997 8	1.6/4 [*2]

[*1] Relative error of flock size d_f: maximum/average absolute value.

[*2] Data for $t_F = 2.22$ min and $t_F = 3.89$ min were excluded.

4 Experimental

The flock growth kinetics was investigated in a baffled tank agitated by a Rushton turbine at mixing intensity 40 W/m^3 and kaolin concentration 0.44 g/l. The tests were carried out on the kaolin slurry model wastewater. The model wastewater was flocculated with organic Sokoflok 56A flocculant (solution 0.1 % wt.). The flock size and its shape were investigated by image analysis. The proposed simple semiempirical generalized correlation for flock growth kinetics was used for data treatment. The fractal dimension of the flocks was also determined.

4.1 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 $H = D$ by a kaolin slurry model wastewater (tap water + kaolin particles). The vessel was agitated by a Rushton turbine (RT) of diameter $d = 60$ mm that was placed at an off-bottom clearance of $H_2/d = 0.85$. 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 agitated vessel dimensions are shown in Fig. 2.

Image analysis technique

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 camera in a plane illuminated by a laser light. The method consists of three steps:

1. illumination of a plane in the tank with a laser light sheet (sometimes called a laser knife) in order to visualize the flocks,
2. a record of the images of the flocks, using a camera,
3. processing the images captured by image analysis software.

The illuminated plane is usually vertical and a camera is placed horizontally (e.g. Kilander et al. [4], Kysela and Ditl [5, 6]). Kysela and Ditl [5, 6] used this technique for flocculation kinetics observation. 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. Therefore we do not observe the flock size during flocculation but we observe it during sedimentation, thus the 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. 2.
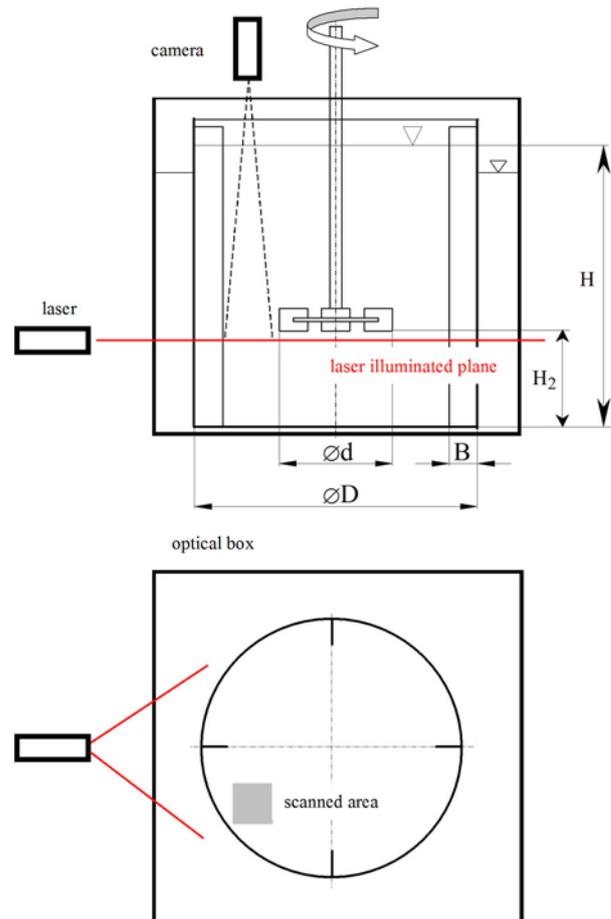
Fig. 2: Schema of the experimental apparatus for image analysis

Table 2: Technical parameters

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

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 laser diode are placed on the laboratory support stand. The technical parameters are presented in Table 2.

4.2 Experimental procedure

The maximum flock sizes formed during flocculation were measured for various flocculation times at mixing intensity $\varepsilon = 40$ W/m^3, constant flocculant dosage $D_F = 2$ ml/l and initial kaolin concentration 440 mg/l. The flock sizes were measured during sedimentation. The experimental parameters are summarized in Table 3.

Table 3: Experimental conditions

		Kaolin concentration
Parameter		$c_{C0} = 0.44$ g/l
ε_V	(W/m^3)	40
n	(rev/min)	180
t_F	(min)	4; 6.66; 9.33; 13.33; 20
nt_F	(–)	720, 1 200, 1 680, 2 400, 3 600
D_F	(ml/l)	2
No. of date		5

The experimental procedure was as follows:

1. *Calibration and experimental apparatus setting*

   Before each flocculation experiment the calibration grid was placed in an illuminated plane and the camera was focused manually onto this calibration grid. Then the image of the calibration grid was recorded. For camera resolution 800×800 pixels, the scale 1 pixel $\propto 45\mu$m was found for our images. This corresponds to a scanned area of 35 mm $\times$ 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 (18 672 Kaolin powder finest Riedel-de Haen)) was used as a model system. The solid fraction of kaolin was 440 mg/l.

3. *Flocculation*

   The model wastewater was flocculated by the organic Sokoflok 56A 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 Table 3. 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 and having 10-bit depth were captured 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 1 200 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 removal of objects touching on an image border, and filter No. 11 for filling an empty space in identified objects caused by capture error) and our macros (Svačina [7] in detail).

Table 4: Generalized correlation $\Delta(1/d_{feq})^* = f(\Delta[nt_F]^*_{log})$: parameters fitted

ε_V	n	$[nt_F]_{max}$	$t_{F_{max}}$	$d_{feq_{max}}$	A_f^*	I_{yx}	$\delta_{r_{ave}}/\delta_{r_{max}}$ *1
(W/m^3)	(ot/min)	(−)	(min)	(mm)	(−)	(−)	(%)
40	180	1 440	8	1.406 2	16.373	0.895 2	2.5/4.4 *2

Note: *1 Relative error of equivalent flock size d_{feq}: average/maximum absolute value.

Note: *2 Flock size for $nt_F = 3\,600$ was excluded from the evaluation.
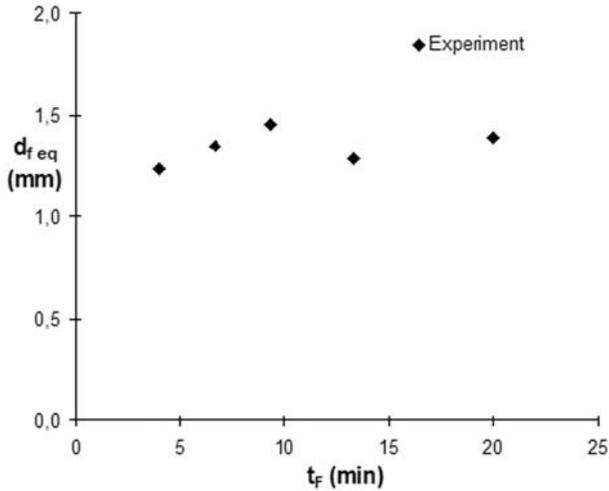
Fig. 3: Experimental data – Maximum flock size vs. flocculation time – $d_{feq} = f(t_F)$

Fig. 4: Generalized correlation $\Delta(1/d_{feq})^* = f(\Delta[nt_F]^*_{log})$

4.3 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 flocculant dosage and mixing intensity is shown in Fig. 3. When flocculation time increases, the flock size increases up to the maximum value, due to primary aggregation, and then decreases due to flock breaking.

Generalized correlation
$\Delta(1/d_{feq})^* = f(\Delta[nt_F]^*_{log})$

The dependence of calculated equivalent diameter d_{feq} on flocculation time was fitted according to the generalized correlation (12). The generalized correlation parameters are presented in Table 4. A comparison of the experimental data and the generalized correlation is depicted in Fig. 4.

Fractal dimension

The flocks generated are often porous and have an irregular shape, which complicates the design of the floc-

culation process. Fractal geometry is a method that can be used for describing the properties of irregular objects. Some flock properties such as shape, density, 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 in our case we measured the projected area of the flocks, the fractal dimension of the 2nd order D_{f2} was used for flock shape characterization. The relation among projected area A, characteristic length scale L_{char} and fractal dimension D_{f2} is given by:
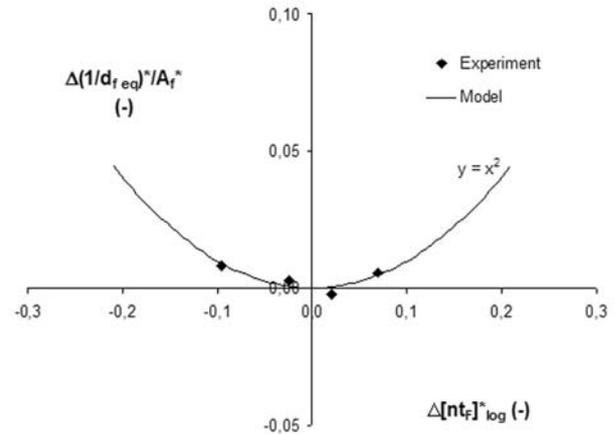
$$A = C \cdot L_{char}^{D_{f2}}. \tag{16}$$

The largest flocks determined in the images were used for fractal dimension estimation. The maximum flock size was used as a characteristic length scale. The fractal dimension D_{f2} was determined for each flocculation time. For illustration, the dependence of the projected area on the maximum flock size is shown in Fig. 5 for dimensionless flocculation time $nt_F = 1\,680$. The fractal dimension D_{f2} plotted in dependence on flocculation time for a given flocculant dosage and mixing intensity is shown in Fig. 6.
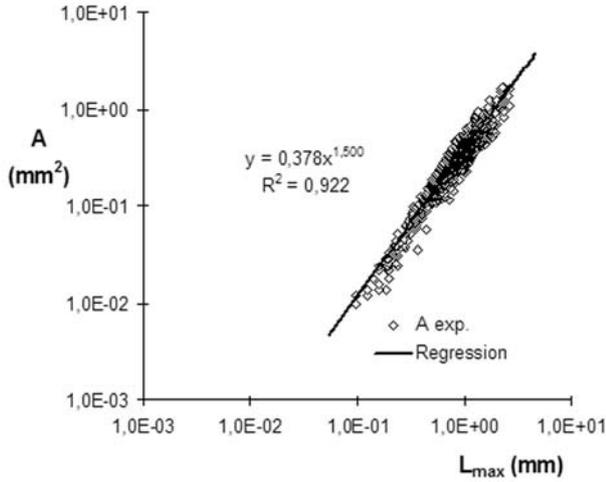
Fig. 5: Fractal dimension determination – example for $nt_F = 1\,680$
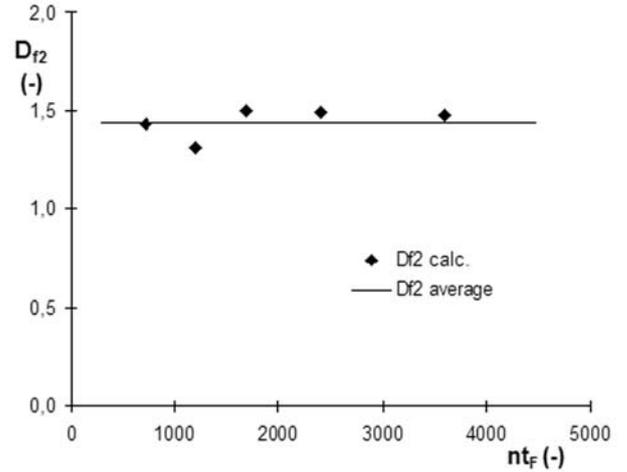
Fig. 6: Fractal dimension vs. dimensionless flocculation time – $D_{f2} = f(nt_F)$

Table 5: Fractal dimension – hypothesis testing

ε_V (W/m^3)	m $(-)$	t-distribution $t_{(m-2),\alpha=0.05}$	Relation $D_{f2} = B \cdot (nt_F)^\beta$ β_{calc}	t-characteristics $|t|$ Hypothesis $D_{f2} = B \cdot (nt_F)^0$ $\beta_{pred} = 0$
40	5	3.182 5	0.045	0.97 (Yes)

Note: The absolute value of parameter t is presented in brackets.

Note: The t-distribution for $(m-2)$ degrees of freedom and significance level $\alpha = 0.05$.

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 predicted (e.g. by some proposed theory) and the parameter values 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 $D_{f2} = B \cdot (nt_F)^\beta$, and the difference between predicted exponent β_{pred} and evaluated exponent β_{calc} was tested. The hypothesis test characteristics are given as $t = (\beta_{calc} - \beta_{pred})/s_\beta$ where s_β is 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 is presented in Table 5. The fractal dimension was found to be independent of the flocculation time, and the value $D_{f2} = 1.442 \pm 0.12$ was determined as an average value.

5 Conclusions

The following results have been obtained:

- A simple semiempirical generalized correlation for flock growth kinetics has been proposed.

- A generalized correlation has been successfully tested and verified, using published data from Kilander et al. (2007). The generalized correlation parameters are presented in Table 1. A comparison of the experimental data and the generalized correlation is depicted in Fig. 1.

- The flock growth kinetics was investigated in a baffled tank agitated by a Rushton turbine at mixing intensity 40 W/m^3 and kaolin concentration 0.44 g/l. The flock size and flock shape were investigated by image analysis.

- The tests were carried out on the kaolin slurry model wastewater. The model wastewater was flocculated with organic Sokoflok 56A flocculant (solution 0.1 % wt.).

- The largest flock was identified from the 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 flocculant dosage and mixing intensity is shown in Fig. 3.

- 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 proposed simple semiempirical generalized correlation for flock growth kinetics was used for data treatment. The model parameters are presented in Table 4.

- 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 was found independent of flocculation time, and the value $D_{f2} = 1.442 \pm 0.125$ was determined as the average value.

Acknowledgement

References

[1] Šulc, R.: *Flocculation in a Turbulent Stirred Vessel.* PhD. thesis. Czech Technical University, Faculty of Mechanical Engineering, 2003 (in Czech).

[2] Šulc, R., Ditl, P.: Effect of Mixing on Flocculation Kinetics. In: *Proceedings of 14th International Congress of Chemical and Process Engineering CHISA 2000*, (CD ROM), Prague, 2000.

[3] Šulc, R., Ditl, P.: The Effect of Flocculant Dosage on the Flocculation Kinetics of Kaolin Slurry in a Vessel Agitated by Rushton Turbine at Mixing Intensity 168 W/m³ and Kaolin Concentration 0.58 g/l. *Czasopismo Techniczne – Seria: MECHANIKA*, Vol. **105** (2008), pp. 341–349.

[4] Kilander, J., Blomström, S., Rasmuson, A.: Scale-up Behaviour in Stirred Square Flocculation Tanks. *Chem. Eng. Sci.*, Vol. **62** (2007), pp. 1 606–1 618.

[5] Kysela, B., Ditl, P.: The Measurement of Floc Size and Flocculation Kinetics by the Laser Knife and Digital Camera. In: *Proceedings of 16th International Congress of Chemical and Process Engineering CHISA 2004*, (CD-ROM), Prague, 2004, ISBN 80-86059-40-5.

[6] Kysela, B., Ditl, P.: Floc Size Measurement by the Laser Knife and Digital Camera in an Agitated Vessel. *Inzynieria Chemiczna i Procesowa*, Vol. **26** (2005), pp. 461–468.

[7] Svačina, O.: *Application of Image Analysis for Flocculation Process Monitoring.* Diploma Project. Czech Technical University, Faculty of Mechanical Engineering, 2009 (in Czech).

Ing. Radek Šulc, Ph.D.
Phone: 420 224 352 558, Fax: +420 224 310 292
E-mail: Radek.Sulc@fs.cvut.cz
Department of Process Engineering,
Faculty of Mechanical Engineering,
Czech Technical University in Prague,
Technická 4, 166 07 Prague, Czech Republic

Notation

A	flock area projected	mm²
A_f^*	coefficient; model parameter (12)	–
$A_{Zr^*}^*$	residual turbidity shift; model parameter (4)	–
d_{feq}	equivalent flock diameter according to flock area	mm
d_f	flock size	mm
$d_{f_{\max}}$	maximum flock size; model parameter (12)	mm
D	tank diameter	m
D_F	flocculant dosage	ml/l
D_{f2}	fractal dimension of 2nd order	–
I_{yx}	correlation index	–
L_{char}	characteristic length scale	mm
n	impeller rotational speed	rpm

$[nt_F]_{\min}$	model parameter (4)	–
$[nt_F]_{\max}$	model parameter (12)	–
t	hypothesis test characteristics	–
t_F	flocculation time	minute
$t_{(m-2),\alpha}$	critical value of t-distribution for $(m-2)$ degrees of freedom and significance level α	–
t_{sed}	sedimentation time	minute
Z_0	turbidity before flocculation	FAU
Z_r	residual turbidity after flocculation	FAU
Z_e^*	turbidity removal degree	–
Z_r^*	residual turbidity degree	–
$Z_{r_{\min}}^*$	model parameter (4)	–

Greek letters

δ_r	relative error;	%
	$\delta_r = 100^* \,(\text{experimental value} - \text{regression value})/\text{regression value}$	
$\Delta(1/d_f)^*$	variable	–
$\Delta[nt_F]_{log}^*$	variable	–
ΔZ_r^*	variable	–
ε_V	specific impeller power input (per volume unit)	W/m^3

Indices

$*$	dimensionless
$* \cdot x$	link
ave	average
max	maximum
min	minimum