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A CHARACTERIZATION METHOD TO SET THE FLOCCULATION PROCESS OF FINES USING COFACTOR-POLYETHYLENE OXIDE AT THE REQUIRED FLOC SIZE

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ABSTRACT

For the importance of floc size (A) in industrial applications, and that needed in retention of fines in papermaking, the objective of this work has been concentrated on the process factors and their role to affect the flocculation process parameters. In this work, in fines flocculation using the cofactor (CF) polyethylene oxide (PEO) retention aid, we have analyzed the effects of the following process factors on the floc size: the cofactor concentration (C), the fines consistency (C_{fn}), and the floc shearing after production (G_t). To maintain the fines floc at the required size and less cost, we have proposed a methodology by using the values of the effective process factors that lead to the required floc size. In cases that larger flocs are resulted, the methodology proposed to shear the resulted flocs that reduce the larger sizes into the required one. This methodology can be used in flocculation processes in industry as in fines flocculation.

KEYWORDS: Polyethylene oxide, Cofactor, Flocculation, Floc Size, Concentration, Consistency, Shear rate.

INTRODUCTION

Specific properties of the paper sheet such as strength, permeability, opacity and apparent density are needed in papermaking. (Brecht, W. and Klemm, K. 1953; Norman 2008). These properties can be fulfilled by maintaining the required properties of flocs and particles that will retain in paper sheet. Large particles and flocs are rejected since they cause deviation from the required properties. The size of the large particles and flocs can be reduced by dispersion, deflocculation and some other methods, while the small particles and flocs can be enhanced to the required size by flocculation and retention (Abdallah Qasaimeh, M.R. 2011; Hubbe, M. A. et al 2002; Hubbe, M. A. 2007). These particle selections are also required in numerous applications rather than the papermaking, such as food (J. K. Kapoor, Shagufta Jabin and H. S. Bhatia), biomedical (Nicklaus Carter et. al., 2021), oil (Zhaoyang You, et. al., 2018; Carlos E. Santo, et. al., 2-012), water treatment (Tridib Tripathy and Bhudeb Ranjan De, 2006) and more. Each application gives a benefit to improve the quality of the product and reduce the cost. In papermaking flocculation and retention of the fines, the 3 - 76 µm negative fiber fragments (Abdallah, Mohammad. R. 2002; Casey, J.P. 1979), into paper sheet are recommended since the fines comprise more than 50% of the feed pulps (Abdallah, Mohammad. R. 2002). Retention of the fines leads to minimize the effluent, reduce the recycling problems (van de Ven, T.G.M. 1993) and improve the sheet properties. For more, since deflocculation is reported (Abdallah, Mohammad. R. 2002; Abdallah Qasaimeh, M.R., 2011; Mohammad Raji Abdallah Qasaimeh, 2022; Dr. Mohammad Raji Abdallah Qasaimeh, 10 5 2024) occurring after flocculation, then the control of the parameters and balances between flocculation and deflocculation are needed to maintain the required goals. For this, we in previous work (Dr. Mohammad Raji Abdallah Qasaimeh, 10 5 2024; (Mohammad Raji Abdallah Qasaimeh, 2022) studied flocculation and deflocculation, their affective factors and deduced the changes in the parameters. Retention aids are used to bridge fines with fibers in heteroflocculation process and fines with fines in homoflocculation to form flocs, which will retain in the holes of the paper sheet. These processes are always performed at high shear rates in order to produce the required floc size A and maintain the homogeneous sheet. In previous work (Abdallah, Mohammad. R. 2002) in fines flocculation with the cofactor CF and polyethylene oxide PEO at high effective shear rate (G_{eff}), small portions of the fines deposited on the fibers, while majority of the fines flocculated as fines flocs. After equilibrium, the flocs deflocculated and the deposited fines left the fibers after retention (Abdallah, Mohammad. R. 2002; Carignan,A. et al. 1998; van de Ven, T.G.M. 1994). Taking deflocculation and flocculation both as one process, we performed number of works to study the process factors, investigate the causes of deflocculation, and analyze the parameters linking deflocculation with flocculation. This analytical study will help to know the parameters and their behaviors that affect the characteristics of resulting floc, thus we can control the resulting floc to satisfy the process goals. In one work we investigated the cause of deflocculation (Abdallah Qasaimeh, M.R., 2011) and ascribed to factors in PEO. In the second, we studied the CF to PEO ratio and the process shear rate (G_p) as affective factors in flocculation process (Abdallah/ Qasaimeh, M. R. et. al., 2014). In the third, the effect of cofactor concentration C was studied and analyzed (Mohammad Raji Abdallah Qasaimeh, 2022). In the fourth, the effect The consistency the of fines C_{fn} was the main parameter in Smoluchowski isotherm (Smoluchowski, M., 1917) and was used in flocculation modeling (Abdallah, Mohammad. R. 2002), but more investigation is needed (Dr. Mohammad Raji Abdallah Qasaimeh., 10 11 2024, to appear). In the fifth, the effect of the shear rate G_t subjected on the resulted flocs after flocculation process was investigated (Dr. Mohammad Raji Abdallah Qasaimeh., 10 10 2024). Usually these resulted flocs are affected by shear rates during storage and within transportation. Determination of the flocculation, deflocculation and reverse equilibrium rate constants in a large scale of cofactor concentration were performed in previous work (Dr. Mohammad Raji Abdallah Qasaimeh, 10 5 2024). These studies explored us to know the properties of the flocs, the affecting process factors, and the parameters that govern the flocculation process factors. Nevertheless, knowledge guide us to good flocculation, but most often insufficient and need a methodology to guide the resulting floc or particles into the required properties. This methodology is known as characterization, which is essential in the industry. Most of the processes in the industry are influenced by the particle size distribution that affect the material performance in the product line and the product quality. The products get better quality by improving their properties such as appearance, taste, texture and shelf-life, but this improvement requires particle size characterization. Many instruments for particle size characterization are commercially present, but they differ based on the difference of their specific techniques and principles. Selection of the right size characterization technique for process particles becomes essential (Rupinder Kaur Dhamoon, et. al., 2018). In numerous industries, the importance of particle size characterization is to show the importance of the measurements of the particle size and shape (April Jane Argamosa, et. al., 2023). In industry such as ceramics, food, cosmetics, medicines, and metallurgy, number of techniques are discussed and with time modern techniques have been developed.

In literature, and for the importance of the knowledge on particle size and shape, number of works was performed on particle size characterization. One worked the convolutional neural network (CNN) to obtain the projection image of an irregular particle (Hongxia Zhang. et. al.2021). The second examined the particle shape, size and its impact on the characteristics of the material. Such work gave an overview on the particle size characterization techniques and the criteria for selecting the best technique for a given sample (April Jane Argamosa, et. al., 2023). The third is a company working on the modeling and invention of number of modern instruments to characterize the particle size that apply the different particle types, sizes and shapes (Malvern Instruments, 2012). The forth studied the techniques, the factors, and the quality-by-design (QBD) approach for particle size characterization (Rupinder Kaur Dhamoon., et. al., 2018).

Based on the necessity of the particle size characterization in industry, our previous work results, and the claimed results by the others, the objectives of this work is to analyze the effects of flocculation factors on the floc size A in fines flocculation using the CF-PEO retention aid, and propose a methodology to select the required floc size A from the values of the flocculation parameters.

Experimentation

Materials

The material used to be flocculated was the fines abstracted from number of pulps taken from Masson Maclaren Mill (Canada), which were disintegrated and washed to remove the unwanted fibrils and colloids. The dual retention aid used was the Flocc 999 (the neutral PEO of a 7 million molecular weight) accompanied with the cofactor (Interac 1323), the negative phenol material, which were both supplied by I.Q.U.I.P Inc, (Canada). These materials used in previous works (Abdallah, Mohammad. R. 2002; Abdallah Qasaimeh, M.R. 2011; Abdallah/Qasaimeh, M.R. et al. 2011; van de Ven, T.G.M. et al. 2004), (Mohammad Raji Qasaimeh, 2022), (Dr. Mohammad Raji Qasaimeh, 10 5 2024) were at different conditions based on the objective of the work.

Experimental Setup

The fines were added to the beaker, the flocculation vessel, (Fig.1) at a definite consistency C_{fn}, and were initially mixed at a definite stirring rate (the process shear rate G_p) to maintain a homogeneous suspension as steady state 1. The CF was added first to the fines suspension to maintain a concentration C, followed with PEO addition, after which the flocculation started with an increase in the floc size A. A stream of fines suspension from the beaker was pumped by a peristaltic pump at the selected tube shear rate G_t, passed the photometric dispersion analyzer (PDA) cell to measure the floc size, and then returned to the beaker. Note that the shear rate G_t is selected equal to G_p for the study of the other parameters, but made variable when it is the objective of the study. The floc size A continued increasing during flocculation and reached maximum (A_m) at equilibrium (steady state 2).

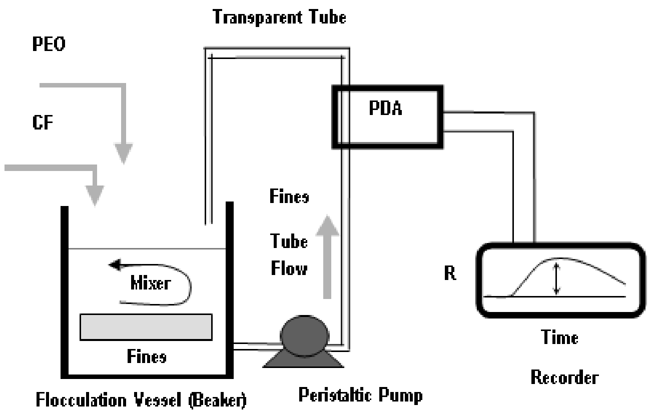

Fig. 1: Experimental Set up of Fines flocculation.

Flocculation and Deflocculation Intensity Readings

The floc size was measured by the PDA photocell as a ratio (R) reading, which is the ratio of the alternating voltage to the direct voltage, (Gregory, J. 1984; Rank Brothers Ltd.). As flocculation proceeded with time (t), the floc size (Fig. 2) increased in the form of the R reading increase, which was drawn by the recorder as a curve. At the equilibrium, the R reading reached maximum and was taken as the vertical distance A_m in arbitrary unit (A.U.), the recorder pen moved within the equilibrium time (τ_e). The slope $r_f \ (= A_m / \tau)$ of the curve at initial flocculation was the initial flocculation rate, where (τ) is the characteristic time of flocculation, the time needed to reach A_m at initial rate r_f, indicating flocculation speed (Abdallah, Mohammad. R. 2002). The

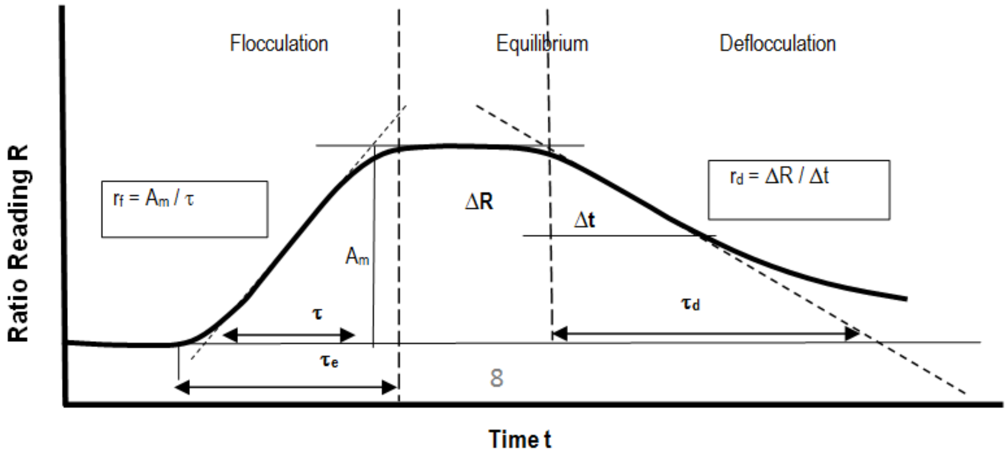

Fig. 2: Flocculation and Deflocculation Intensity Readings.

deflocculation was recorded after equilibrium, where the reading R started to decrease. The initial rate of deflocculation r_d $(= A_m / \tau_d)$ was taken as the slope at initial deflocculation, and the characteristic time of deflocculation (τ_d) is the time needed to drop the floc size from the maximum A_m to the initial size of the fines (Abdallah Qasaimeh, M.R. 2011).

RESULTS AND DISCUSSION

In this work, since the main objective is to get the required floc size, while floc strength can be satisfied from the choices of the types of the particle and retention aids. In this work the particles were the fines and the retention aids were the cofactor CF and polyethylene oxide PEO. Now the choice of floc size can come from the effects of the affective process factors on the resulted floc size in fines flocculation. These factors are mainly, the consistency of the fines (Dr.Mohammad Raji Abdallah Qasaimeh 10 11 2024 to appear), the cofactor concentration (Dr. Mohammad Raji Abdallah Qasaimeh, 10 5 2024; Mohammad Raji Abdallah Qasaimeh, 2022), and the shearing of the flocs (Dr. Mohammad Raji Abdallah Qasaimeh, 10 10 2024), which were investigated in our previous work. Now to produce the required floc size, we have proposed a new methodology in which we fix the required floc size on the horizontal coordinate, and then find out the value of the corresponding process factors. Now the operators in the mill will run the flocculation process at the found process factors that definitely produce the required floc size.

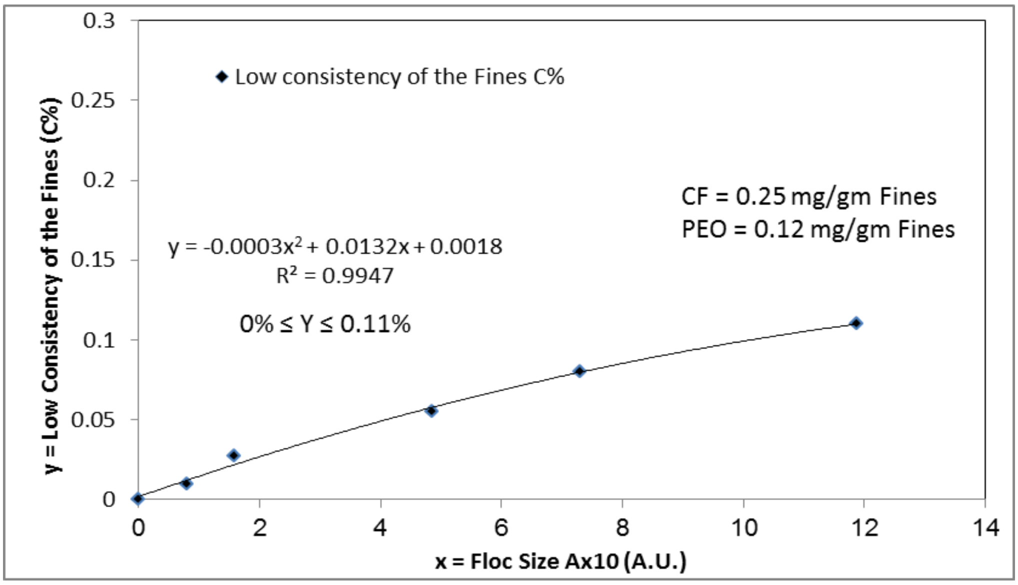

Fig. 3: Selection of the low consistency of fines from the required floc size of fines.

Selection of the consistency of the fines

One of the affective factors in fines flocculation using the CF-PEO retention aid is the consistency of the fines (Dr. Mohammad Raji Abdallah Qasaimeh 10 11 2024 (to appear). In studying the consistency effect on floc size, all the process factors were kept constant, except the consistency was variable. Results have been taken from the previous work (Dr. Mohammad Raji Abdallah Qasaimeh 10 11 2024 (to appear). In investigation of the consistency effect on the size of the fines floc, results have shown two curves; the floc size resulted at low consistency values (Fig. 3), and the floc size resulted at high consistency values (Fig. 4). In analysis of the consistency effects, it has been found that the sizes of the fines floc at low consistency values $(C_{fn} \leq 0.11\%)$ increased with the increase in the consistency, reached maximum, and then started to decrease giving small floc sizes at high consistency values $(0.11\% \leq C_{fn} \leq 0.33\%)$. In this work, the fines consistency at low values (Fig. 3) and at high values (Fig. 4) have been plotted in the vertical coordinate versus the floc size taken in the horizontal coordinate. Each curve has given the best fit correlation. To set flocculation process to get the required floc size, we use the new methodology just we fix the required floc size on the horizontal coordinate and then find out the corresponding consistency on the vertical coordinate. Or we use the best fit correlations shown in the figures 3 and 4. For example, let the required floc size is $A(=7.3)$ A.U.; this floc can be obtained either using the $C_{fn} = 0.08\%$ at low consistency (Fig. 4), or the $C_{fn} = 0.288\%$ at high consistency(Fig. 5); the economical one the operator will choose. An alternative to using figures is the use of the best fit correlations (shown in Fig. 3 and Fig. 4) at low and high consistencies respectively.

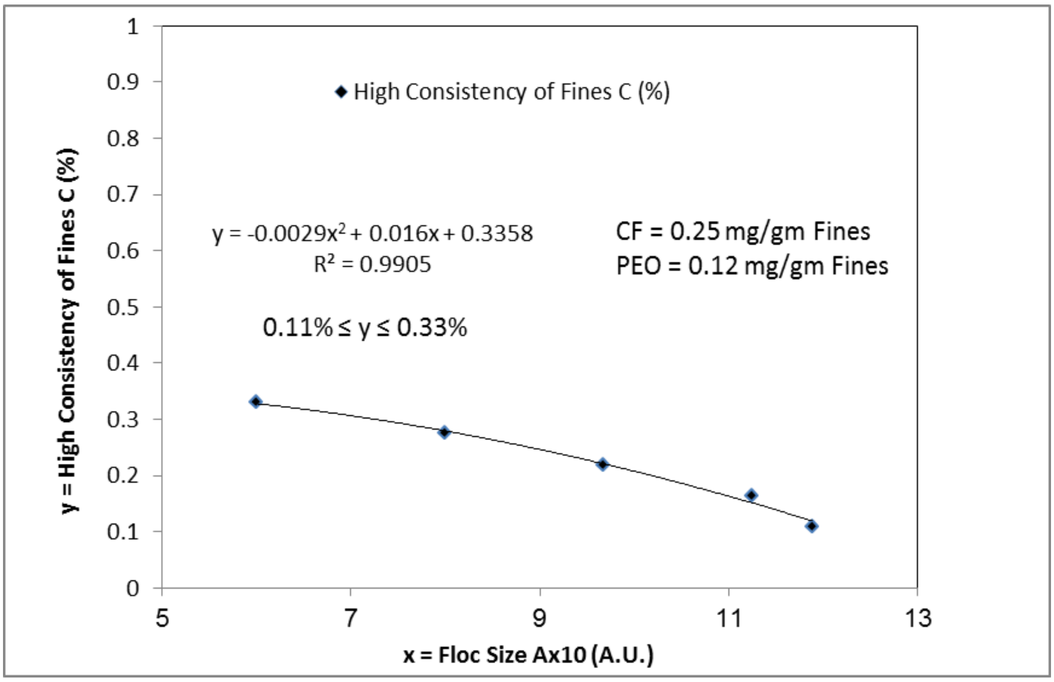

Fig. 4: Selection of high consistency of the fines from the required floc size of fines.

Selection of the cofactor concentration

In fines flocculation, where a cofactor is added to induce the CF-PEO bridging to the fines (van de Ven, T.G.M. and Alince, B. 1996) and form floc, the CF concentration C was found an affective process factor having a significant effect on the floc size. In studying the effect of CF concentration C on floc size, all the other process factors were kept constants, while the CF concentration C was variable. Floc sizes were found at low and high values of C. At low values of C, small flocs of the fines have been produced, and the size of the resulted flocs started to increase with the increase in C, reaching maximum in the optimum zone, and then the resulted floc sizes started to decrease at high values of C (Mohammad Raji Abdallah Qasaimeh, 2022). Now we have three zones of floc sizes: the low CF concentration zone; the optimum CF concentration zone; and the high CF concentration zone. Every value of concentration C works to fix a definite floc size, and to get the required floc size A in flocculation process, it requires to use the value of C that produces the floc size A. The small floc sizes were found to result at low values of C, increased with the increase in the C,

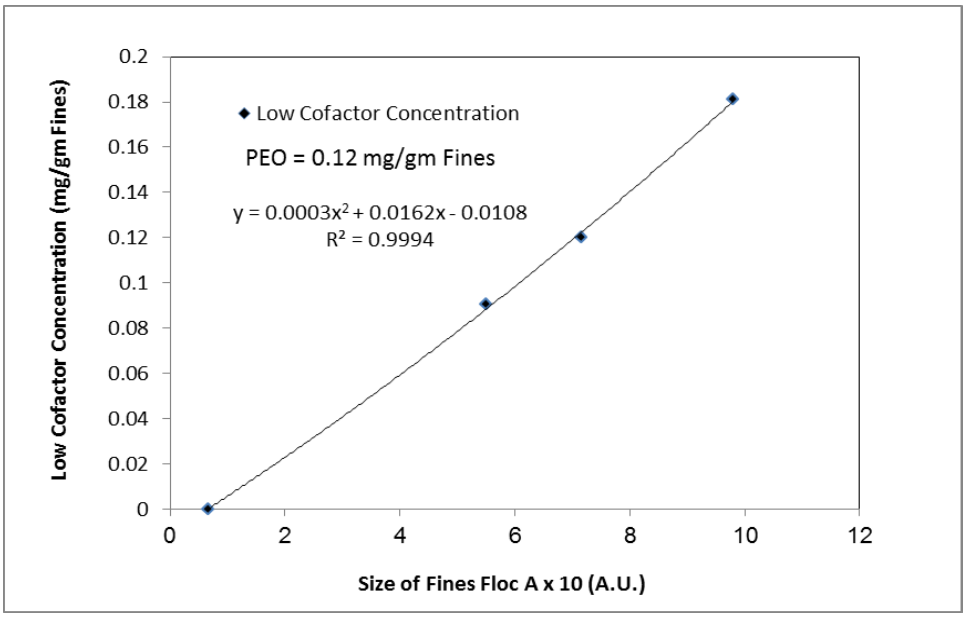

Fig. 5: Selection of low cofactor concentration from the required floc size of fines.

reaching maximum at optimum values, and then started to decrease at high values of C, resulting in small sizes of flocs (Mohammad Raji Abdallah Qasaimeh, 2022). Plots of the CF concentrations versus the floc sizes A are made in this work. These plots are shown at low values of C (Fig. 5) in the range $(C \le 0.181)$, at optimum values of C (Fig. 6) in the range $(0.181 \le C \le 0.543)$, and at high values of C (Fig. 7) in the range $(0.543 \le C \le 1.276)$. Similarly to the choice of the required size from

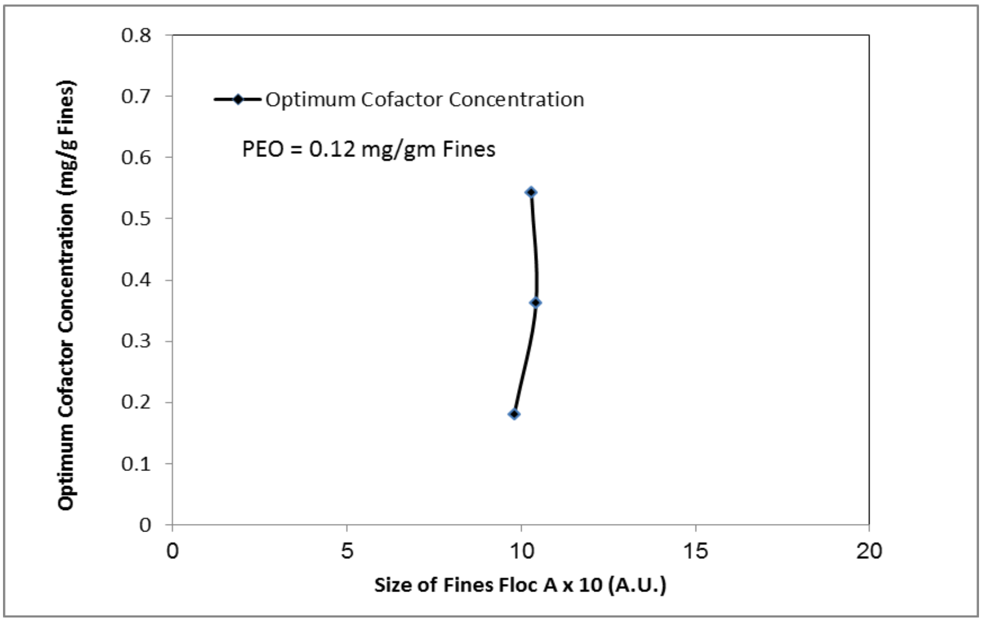

Fig. 6: Selection of optimum cofactor concentration from the required floc size of fines.

consistency, we have used the same methodology in case of CF concentration to select the required floc size. Using the figures 5, 6 and 7, we first fit the required floc size on the horizontal coordinate and then find out the corresponding needed CF concentration C on the vertical coordinate. Let the required floc size is the maximum, then we use figure 6 and the value of C will be one in the range $(0.181 \le C \le 0.543)$. Let the required floc size is a value less than the maximum, then we use either figure 5 or figure 7, the economical one the operator will use, and C value will be the corresponding one on the vertical coordinate. For example, let the required floc size is

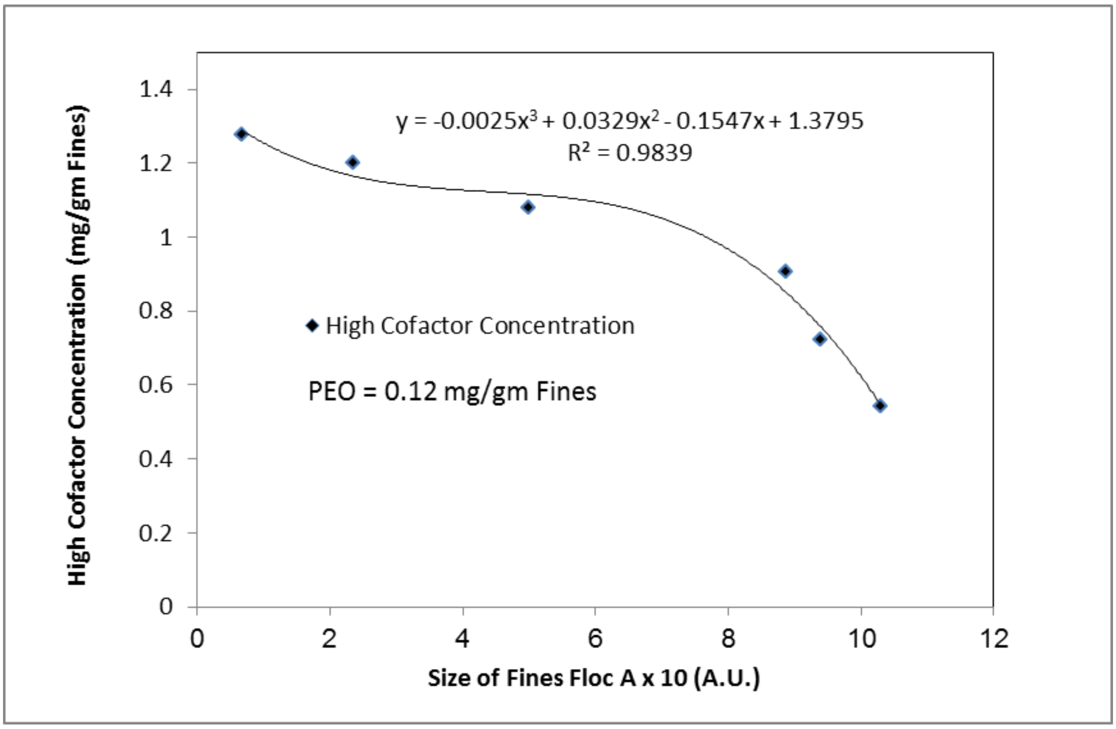

Fig. 7: Selection of high cofactor concentration from the required floc size of fines.

$A(=5)$ A.U.; the concentration of CF will be either the $C = 0.08$ mg/gm fines using figure 5 at low CF concentration values, or it will be $C = 1.08$ mg/gm fines using the figure 7 at high CF concentration values. An alternative to use the figures is using the best fit correlations shown in the figures at low and high CF concentrations.

Usage of shearing to get the required size

For any flocculation process, either at a definite value of fines consistency or at a definite value of cofactor concentration, but the size of the resulted floc is larger than the required size, in this case we can use shearing of the resulted flocs to maintain the required size. This shearing can be maintained by a pump at the exit of flocculation vessel, by increasing the exit shear rate G_t that transports flocs into the delivery line. In this work, we have sheared the large resulted flocs (Fig. 8), which have been reduced into smaller ones. When the floc sizes are at the required size by setting the values of the consistency and the cofactor concentration, we just set the shear rate G_t into the delivery line at a value not changing the floc size. In case the floc size is

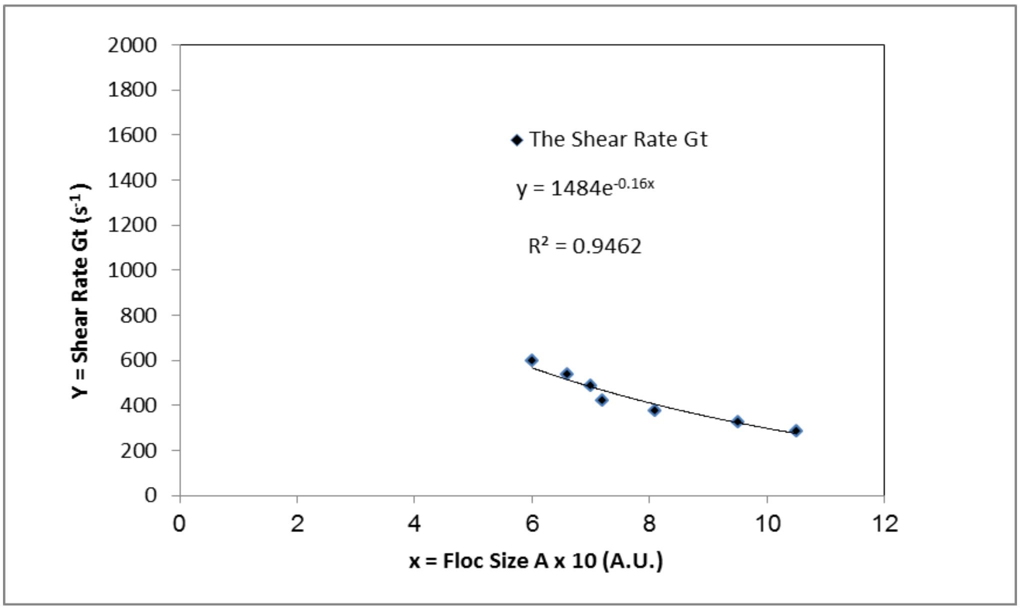

Fig. 8: Selection of the shear rate to reduce the floc size to the required size.

larger than the required size, then we need to subject the flocs to shearing at the exit by selecting the exit shear value using figure 8; now the large floc sizes will be reduced into the required size. Let the required floc size is $A \approx 7.2$ (A.U.), then by using figure 8, we fix this size on the horizontal-coordinate and then find out the corresponding shear rate on the vertical coordinate that fit $G_t\,(=420\,s^{-1})$. This shear rate can also be found using the best fit equation (Fig. 8), and this equation can be used to extrapolate to very high shear rates that reduce the floc into small sizes (dispersion). Figure 8 shows that by extrapolation, to disperse the flocs into small particles, the required shear rate can be at values higher than $G_t\,(=1000\ s^{-1})$. Instead of performing high shearing to get small sizes, and since the high shearing is cost full,

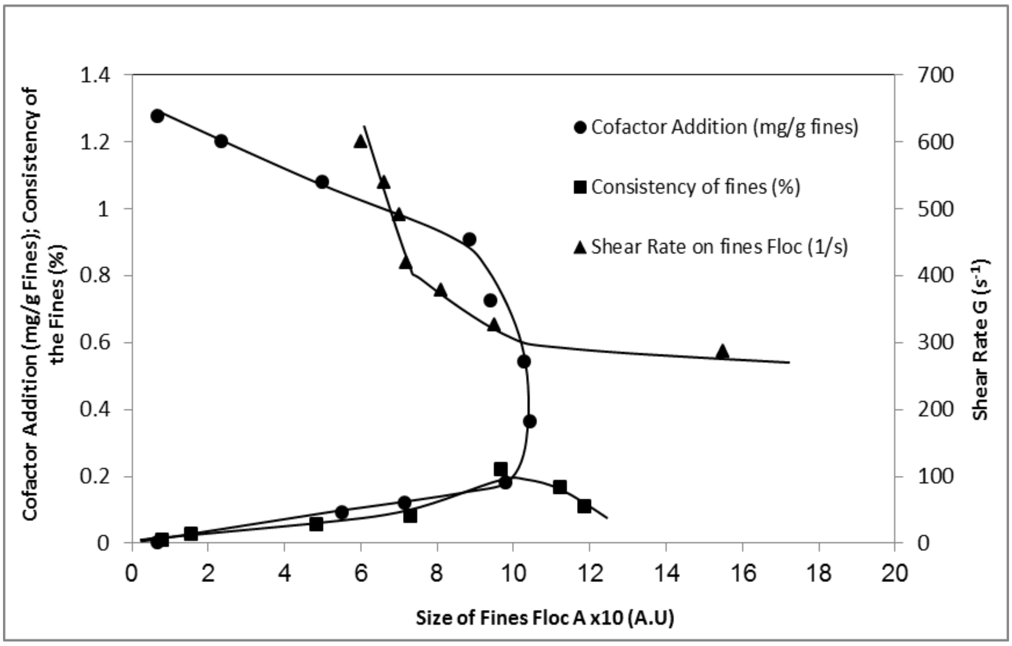

Fig. 9: Floc size of fines (the primary horizontal axis) versus cofactor concentration (the primary vertical axis) and the shear rate (the seconday vertical axis).

then these sizes can be obtained just by setting the values of the consistency and the values of the cofactor concentration at the required ones.

Now the effects of flocculation process factors on floc size A have been studied and investigated in this work. These effects have been plotted versus the floc size from which we can select the value of the process factors that give the required floc size. These plotted curves are collected and shown (Fig. 9) in one figure, which can be taken as a reference. This figure can serve us the following choices. One is to get any required floc size as described before. Second, the maximum floc size $A \approx 10$ (A.U.) can be obtained by the cofactor CF addition at $C(=0.18-0.905)$, and this size can be

Table 1: Summary of the process factor effects on the fines floc size.

Process Factor	**Fines Floc Size** $A(AU.)$			
	$A \approx 10$	$A < 10$	$A > 10$	$A \leq 2$
CF Concentration $C(\frac{mg}{gm\ fines})$	$C = (0.18-0.905)$	$C < 0.18)$ $C > 0.905)$		$C(\leq 0.03)$ $C(\geq 1.2)$
Tube Shear Rate $G_t\ (s^{-1})$	$G_t\ (=300)$	$G_t\ (>300)$	$G_t\ (<300)$	$G_t\ (\approx 1078)$ best fit eqn.
Fines Consistency C_{fn}	$= 0.22\%$	$< 0.22\%$	$< 0.22\%$	$\leq 0.03\%$

maintained at floc shearing $G_t\,(=300\,s^{-1})$. Larger floc sizes can be obtained only by reducing the shear rate $G_t\,(\leq 300\,s^{-1})$ to some lower. Third, to get small flocs say $A \approx 2$ A.U., this can be maintained either at low CF concentration $C\,(=0.03)$ mg/gm.

Fines or at very high CF concentration $C\,(=1.2)$ mg/gm fines, the 40 times larger concentration, and no need to shear the flocs after exiting the flocculation vessel. Smaller flocs (say $A \leq 2$ A.U. and lower) can also be maintained by lowering the fines consistency to $C_{fn} \leq 0.028\%$. It can be maintained by lowering the CF concentration less than $C\,(\leq 0.03)$ mg/gm fines, or increasing the CF concentration above the $C\,(\geq 1.2)$ mg/gm fines. The summary of the process factor effects on floc size are shown in table 1.

CONCLUSION

In this work we have studied the effects of the main process factors on the size of the resulted flocs in fines flocculation using cofactor-polyethylene oxide retention aid system. Here we concentrated on the floc size since the main object from flocculation is the size of the floc that required to industry. The other required properties of the resulted floc is ascribed to the raw materials and the retention aid which already are chosen in work. To get the required size by controlling the operation will reduce the cost of operation. In this work we have proposed a methodology to control the flocculation process to produce the required floc size.

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