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Ultrafine quartz flocculation: Part II. Main variables

http://dx.doi.org/10.1590/0370-44672022760014

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Abstract

Flocculation is a complex process, usually dependant on various conditions to perform adequately. Nevertheless, the relevance and interaction of the process variables are not easily found in literature. In this study, nine variables of ultrafine quartz flocculation systems, defined in Part 1 of this study, were analysed. The effects of each variable and their interactions in the system were investigated based on flocculation and sedimentation tests. The results were statistically evaluated using the supernatant turbidity as experimental response. The evaluations resulting from the statistical approach indicated that suspension pH (5.49%) was the most significant variable, followed by flocculant concentration (4.07%) with the second greatest effect on the turbidity of the supernatant. Solid concentration, agitation intensity, and surfactant conditioning time (4.05%, 3.51% and 3.51%, respectively), also have significant effects on ultrafine quartz flocculation. The flocculant concentration and the surfactant conditioning time were the variables with the most significant interactions with the main variables. The Camp Number values showed a negative exponential relationship with the turbidity results, proving to be an important tool to evaluate flocculation. Backscattered electron scanning microscopy images of flocs formed in the presence of 30 g/t PAM showed compact flocs in the size range between 150 and 365 µm, with noticeable sphericity.

keywords: flocculation, ultrafine quartz, turbidity, Camp Number, fractioned factorial method.

1. Introduction

Strong agitation of the system can result in breakage of the flocculant molecule and reduction in its bridging capacity. A clear relationship has been observed between the shearing effect, the surface chemistry, the structure of flocculant-particle interactions and surface dehydration (McFarlane *et al*., 2005). The intensity of agitation has a strong influence on the sedimentation rate and, at the lower level, on the sediment consolidation (Ofori *et al*., 2011). Also, the agitation of the system was indicated as the third variable among those most important in flocculation, affecting the dispersion and adsorption of the flocculant molecule and the floc formation, growing, and breakage (Bulatovic, 2007). Owen *et al*. (2002) indicated the agitation intensity as a critical factor, with effective action on the mixing and adsorption of the flocculant, promoting adequate conditions for the formation of bridges between the polymer and particles, while excessive agitation breaks the flocs (Heath *et al*., 2006).

Small flocs have been observed to be formed at low flocculant concentrations, while increasing the adsorption density results in the addition of more particles to the floc and an increase in its size and sedimentation rate (Cengiz *et al*., 2009). Slow adsorption of a non-ionic polyacrylamide on a flat glass surface, at concentrations of 50 and 100 g/t, was observed by (Al-Hashmi & Luckham, 2010).

The increase in flocculant adsorption after previous surfactant adsorption was reported by Broseta & Medjahed (1995). The highest aggregation level was observed at a surfactant concentration of 3.86 x 10^{-4} mol.L^{-1}, between pH 3.0 and 8.5, indicating good correlation between hydrophobicity and the quartz flocculation (Lu & Song, 1991). According to Baltar & Oliveira (1998), the co-adsorption of polyacrylamide and surfactant occurs via chain-to-chain interaction between the hydrophobic portions of the flocculant and the exposed portions of the previously adsorbed surfactant.

Regarding the surfactant conditioning time for maximum flocculation, different values have been reported: 15 minutes for quartz < 5 µm (Raju *et al*.,1991), 10 minutes for 1% w/w for fine quartz (Lu & Song, 1991), and 5 minutes for colloidal anatase (Campêlo *et al*., 2017). McFarlane *et al*. (2005) used 5 seconds of flocculant addition and conditioning to a smectite and kaolinite suspension. Cengiz *et al*. (2009) used 15 seconds addition and 120 seconds conditioning times in the flocculation of aqueous ceramic tailings, values similar to those used by Ofori *et al*. (2011) for charcoal tailings. Addition and conditioning times of 180 seconds were selected by Campêlo *et al*. (2017) in flocculation studies of colloidal anatase suspension. The suspension pH, flocculation time, and concentration were checked and compared in literature. However, it is worth mentioning that, from all cases analysed, information on the relevance of each variable alone and their interactions in the flocculation process was scarce.

The motivation for this study was to verify which variables are relevant in the flocculation of ultrafine quartz, indicating the most statistically significant variables and their interactions.

2. Materials and methods

A natural quartz sample was collected in Minas Gerais state, Brazil, and, after comminution and classification stages, the particles in the size range between 38 and 10 µm were reserved for the experiments. Hydrochloric acid and sodium hydroxide, manufactured by Synth, were used to adjust the suspension pH at 3.0 and 11.0, respectively, controlled using a DM-22 Digimed pHmeter. Etheramine Flotigam EDA, Clariant, was utilized as the surfactant at concentrations of 3.2 x10^{-5} and 1.6 x 10^{-3} mol.L^{-1}. The flocculant was a non-ionic polyacrylamide (PAM), Clariant, with 2.0 x 10^{-6} g.mol^{-1} molecular weight, added at the concentrations of 30 and 300 g/t.

Quartz aliquots of 2.5 and 10.0 g, used to obtain solid concentrations of 5 and 20 g.L^{-1}, respectively, were weighed in an AY220 precision balance (Shimadzu), and then conditioned for 2 minutes in a SoniClean 2PS ultrasonic washer (Sanders), for better particle dispersion. The water used was purified by reverse osmosis in a Q385 filter (QUIMIS), for better control of the chemical composition of the suspension.

A RW20 mechanical stirrer (IKA) was used for controlled agitation of the suspension at 290 and 550 rpm and the supernatant turbidity was measured using a TL2350 turbidimeter (HACH).

The experimental sequence was: (i) weighing and ultrasonic cleaning of the sample, (ii) suspension pH adjustment, (iii) addition of the suspension into the mechanical stirrer, (iv) addition of the surfactant, (v) addition of the flocculant, (vi) addition of the suspension into the beaker and settling for 15 min, (vii) collecting supernatant samples at every 2 min for 12 min, (viii) registering the solid liquid interface at each 2 min, (ix) registering the final compact sediment height after 15 min.

Samples of the compacted quartz sediment from the flocculation and sedimentation tests were dried at room temperature and analysed in a Quanta 200 scanning electron microscope (SEM).

Thirty-two experiments were carried out according to a statistical approach using the fractional factorial design method for nine independent variables at two experimental levels (high and low) to evaluate the statistical significance of the investigated variables and their influence on the experimental responses. More detailed information about the fractional factorial design method can be obtained in the following references: Cox (1958), Duckworth (1960), Box *et al*. (1988), Antony (2014), Montgomery (2017), and MA *et al*. (2017). The nine variables and their respective experimental levels are shown in Table 1. The statistical software Minitab20® was used in the statistical planning of the experiments and statistical evaluation of the results. The replicated central points were used to calculate the pure error, as suggested by Bradley (2007).

Table 1 - Variables and their adopted experimental levels.

Variables	Symbols	Units	Experimental levels	
			(-) low	(+) high
Flocculant concentration	A	g.t^{-1}	30	300
Surfactant concentration	B	mol.L^{-1}	0.000032	0.0016
Surfactant conditioning time	C	min	5	15
Flocculant addition time	D	s	5	180
Flocculant conditioning time	E	s	10	300
Agitation during flocculation	F	rpm	290	550
Suspension pH	G	-	3	11
Flocculation time	H	min	2	10
Solids concentration	J	g.L^{-1}	5	20

3. Results and discussion

The turbidity results at three conditions, one without reagents (blank) and two with reagents, at pH 3.0, are shown in Figure 1. Decrease of the turbidity values was observed in the presence of the flocculant and surfactant at both PAM concentrations and the final turbidity level was reached at a lower settling time (4 min) for the high flocculant concentration. These results agree with the characterization of the system conducted in Part I of the study, since quartz presented an IEP close to pH 2. The mineral's low surface charges found at acidic conditions favors aggregation, which is obtained through flocculation.

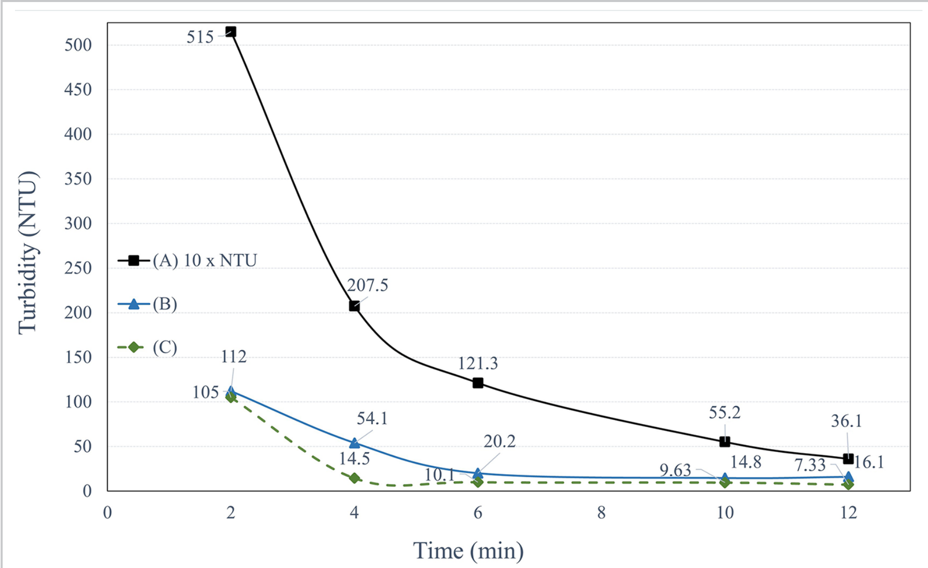

Figure 1 - Turbidity curves of quartz -38+10 μm, pH 3.0, 0.2% solids, $3.2x10^{-5}$ mol.L^{-1} EDA, 550 rpm. (A) blank (B), 300 g/t PAM, (C) 30 g/t PAM.

Figure 2 shows the results of turbidity, at pH 11.0, for three conditions, one without reagents (blank) and two in the presence of reagents. The use of reagents (flocculant and surfactant) resulted in the decrease of turbidity at pH 11. However, when compared to the acidic conditions shown in Figure 1, the turbidity levels are much higher, indicating the influence of the more electronegative surface of quartz on the flocculation conditions. This behaviour is consistent with the results of Part I of the study, which indicates that the mineral surface charges favor stability and not aggregation at alkaline pH values.

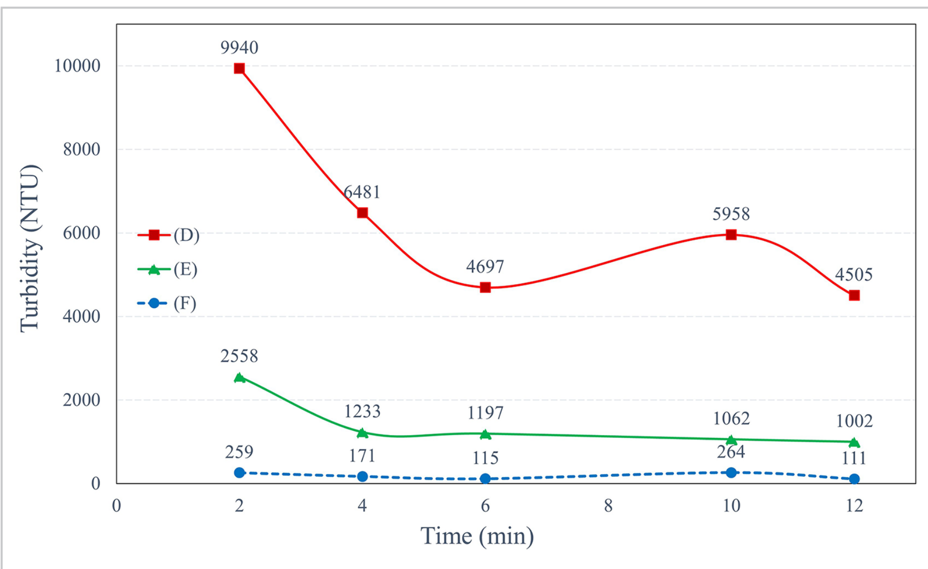

Figure 2 - Turbidity curves of quartz -38+10μm, pH 11.0, 0.2% solids, 550 rpm. (D) blank, (E) 300 g/t PAM + $1.6x10^{-3}$ mol.L^{-1} EDA, (F) 30 g/t PAM + $3.2x10^{-5}$ mol.L^{-1} EDA.

The sediment images at the final settling time of the six conditions addressed in Figures 1 and 2 are presented in Figure 3. The clarification of the suspension was fast for conditions B and C, with well-defined separation between the solid and liquid phases in less than two minutes, with the formation of compact sediment with minimal water retention. Regarding conditions E and F, obtained for flocculation at pH 11.0, it was not possible to achieve a clear visible separation between the solid and liquid phases.

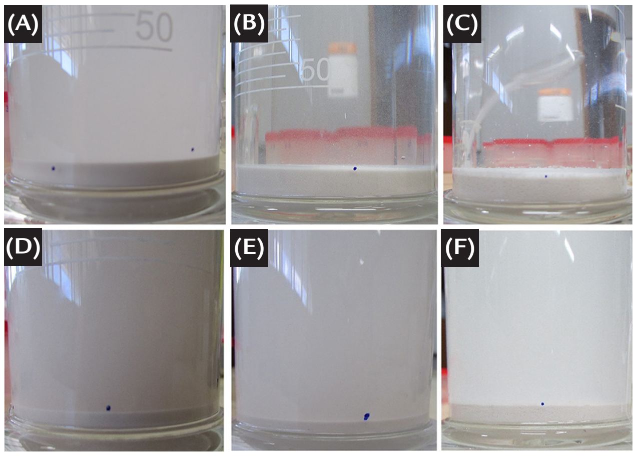

Figure 3 - Final quartz -38+10μm sediment, at 0.2% solids, 550 rpm and at pH 3.0: (A) blank, (B) 300 g/t PAM, (C) 30 g/t PAM, $3.2x10^{-5}$ mol. L^{-1} EDA, and at pH 11.0: (D) blank, (E) 300 g/t PAM + EDA $1.6x10^{-3}$ mol. L^{-1} EDA, (F) 30 g/t PAM + $3.2x10^{-5}$ mol. L^{-1} EDA.

The turbidity results and the sediments images, shown in Figure 3, indicate that effective flocculation was obtained only in the acidic condition. Although Figure 2 shows a great decrease in turbidity at pH 11.0, Figures 3E and 3F display no effective flocculation under this condition.

Table 2 presents the results of compact sediment height and settling velocity at the six conditions addressed in Figures 1, 2, and 3.

Table 2 - Compact sediment height and sedimentation velocity for conditions A, B, C, D, E and F.

Symbol	Condition	Compacted sediment height (mm)	Sedimentation velocity (cm/min)
A	without reagents (pH 3.0)	3.0	0.018
B	pH 3.0	3.0	4.0
C	pH 3.0	3.0	5.5
D	without reagents (pH 11.0)	2.0	0.0018
E	pH 11.0	2.0	0.02
F	pH 11.0	4.0	0.06

Significant flocculation and settling results in the presence of PAM and EDA were obtained at pH 3.0, but not at pH 11.0, also showing the effect of pH on the process. Considering that the negative surface charge of oxides increases at more alkaline pH values (Griot & Kitchener, 1965; Gebhardt & Fuerstenau, 1982) and that polyacrylamide hydrolyses at pH 10 (Hollander, 1981 apud Lee & Somasundaran, 1989), the decrease or absence of PAM adsorption at pH 11, due to electrostatic repulsion, explains the results displayed in Table 2.

Figures 4 and 5 shows the results of Camp Number for the experiments on quartz flocculation, as a function of the system's turbidity. The Camp

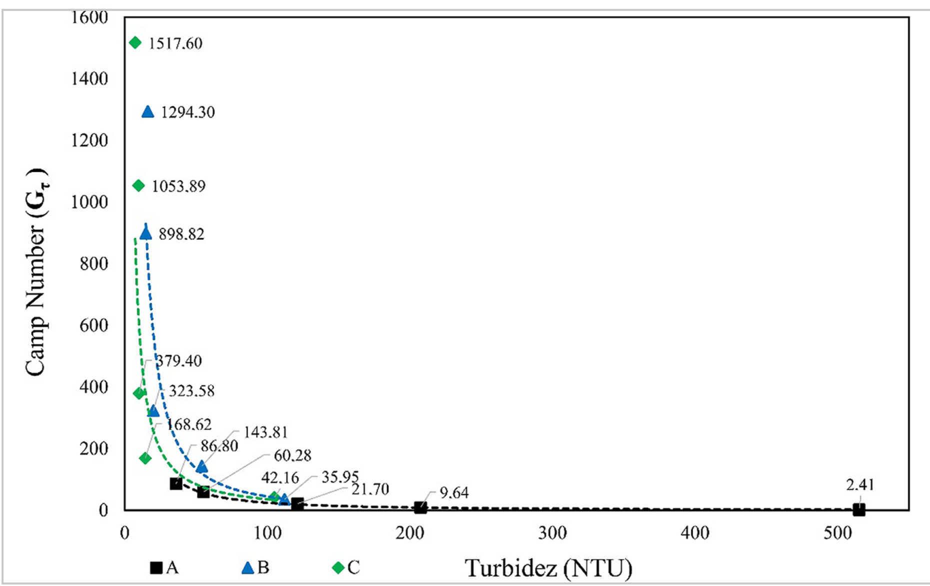

Figure 4 - Camp Number (Gτ) for the flocculation of quartz sediment -38+10μm, at pH 3.0, 0.2% solids, $3.2x10^{-5}$ mol.L^{-1} EDA, 550 rpm. (A) blank, (B) 300 g/t PAM, (C) 30 g/t PAM.

Number (Gτ), defined as the product of shear rate and residence time in a stirred tank, is considered the total amount of shear applied to a slurry, being used for evaluating the extent of aggregation (Khanna *et al.*, 2019). According to Camp (1955), aggregates can be formed at high values of Camp Number, which are obtained either by high shear during short periods or low shear during long periods. The results in Figures 4 and 5 indicate a negative exponential relationship between the Camp Number and the turbidity of the system. For each condition shown in Figures 1, 2 and 3, as longer times improved sedimentation of flocs and reduced turbidity, larger Gτ values were observed. The best flocculation conditions, according to turbidity results, also showed the largest Camp Numbers, which indicates greater aggregation. This was observed for conditions C and F.

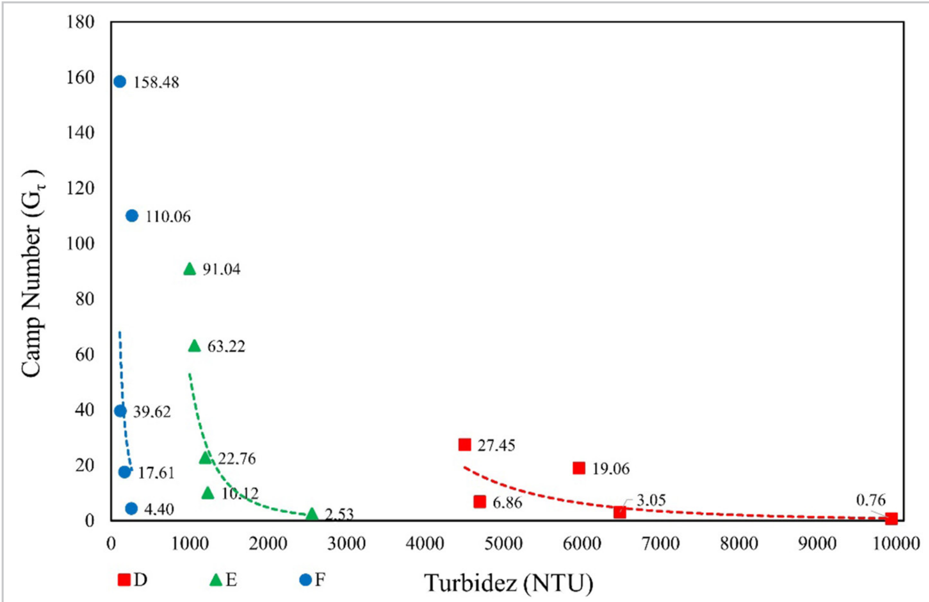

Figure 5 - Camp Number (Gτ) for the flocculation of quartz sediment -38+10μm, at pH 11.0, 0.2% solids, 550 rpm. (D) blank, (E) 300 g/t PAM + 1.6x10^{-3} mol.L^{-1} EDA, (F) 30 g/t PAM + 3.2x10^{-5} mol.L^{-1} EDA.

Back scattered scanning electron microscopy (BSE SEM) images of flocs formed under condition C, presented and discussed in Figures 1,3 and 4 and Table 2, are shown in Figure 6. It is possible to observe compact floc in the size range between 150 and 400 μm, with noticeable sphericity, in agreement with Baltar & Oliveira (1998), who stated that the previous adsorption of surfactant yields large and compact flocs at high sedimentation velocity. It is important to mention that the characteristics of the aggregates observed under SEM are dependant on sample preparation, which can lead to errors during the measurement. This stage was carefully conducted to avoid this problem.

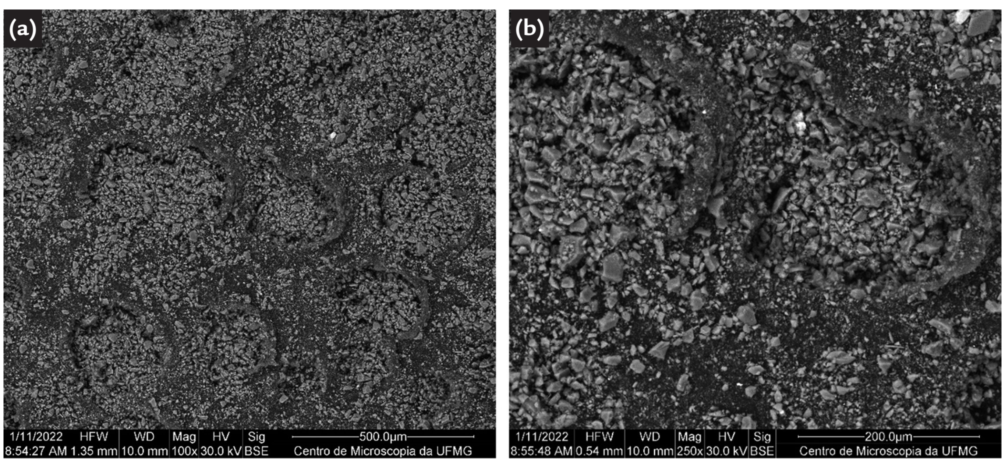

Figure 6 - Backscattered scanning electron microscopy images of flocs formed in condition C. (a) 100x magnification e (b) 250x magnification.

Table 3 shows the confounding structure of interaction for the flocculation variables, up to the third order. This structure describes the confounding pattern which occur in the analysed experiment. The confounding terms are designated as aliases.

Table 3 - Structure of confounding interaction for the flocculation variables.

Aliases	Continuation of aliases
I	I
A	AC + BDF + BEG + DEJ + FGJ
B + CHJ + DGJ + EFJ	AG + BCE + BFH + CFJ + EHJ
C + BHJ + DGH + EFH	AJ + CDE + CFG + DFH + EGH
D + BGJ + CGH + EFG	BD + GJ + ACF + AEH
E + BFJ + CFH + DFG	BE + FJ + ACG + ADH
F + BEJ + CEH + DEG	BH + CJ + ADE + AFG
G + BDJ + CDH + DEF	CF + EH + ABD + AGJ
H + BCJ + CDG + CEF	CG + DH + ABE + AFJ
J + BCH + BDG + BEF	DE + FG + ABH + ACJ

Figure 7 shows the results of normal probability of the standardized effects and the individual contributions of each variable and their interactions on the supernatant turbidity for each flocculant experiment indicated in Table 1. More significant contributions were observed from flocculant concentration, surfactant conditioning time, agitation intensity, suspension pH, and solids concentration.

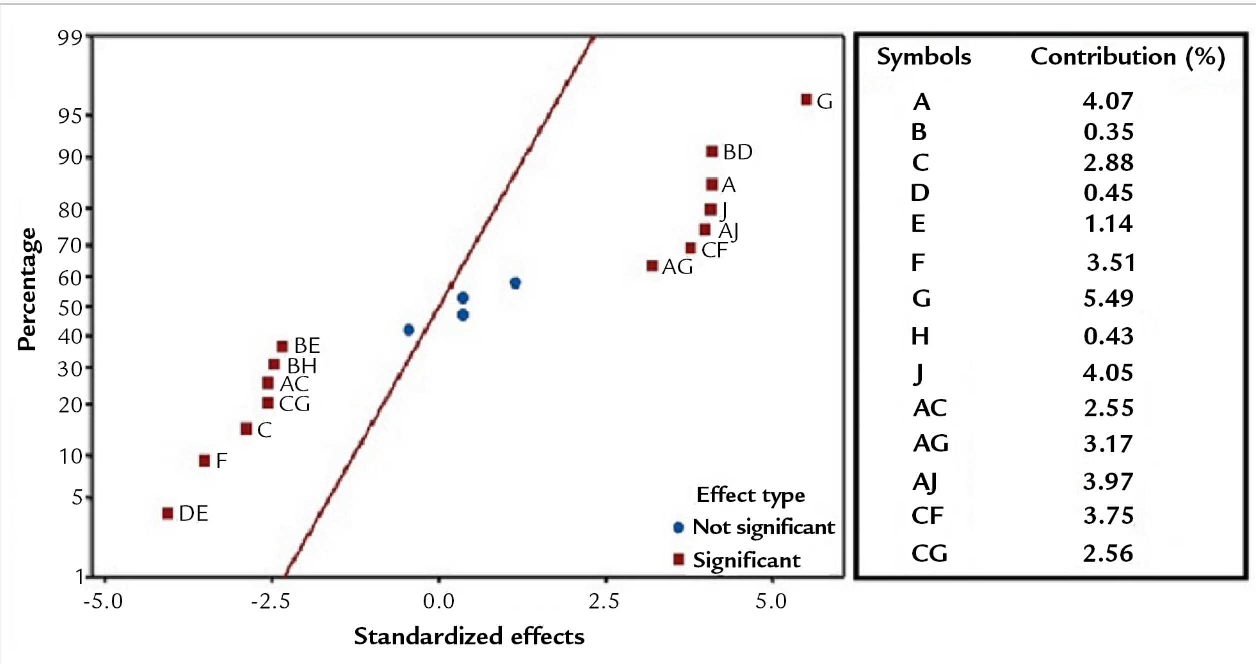

Figure 7 - Normal probability of the supernatant turbidity effects for the flocculation analyses.

The surfactant concentration (B), flocculant addition time (D), flocculant conditioning time (E), and flocculation time (H) did not present significant effects on the experimental response. The sequence of significance is: pH (G) > flocculant concentration (A) > solid concentration (J) > variable interaction AJ > variable interaction CF > agitation intensity (F) > variable interaction AG > surfactant conditioning time (C) > variable interactions CG > variable interaction AC. Nevertheless, it is relevant to notice that all factors and interactions to the left of the plot central line have a negative effect on the response, for example, a change from low level (-) to high level (+) of the main variables (F and C), indicates reduction of turbidity. The opposite occurs with the variables to the right side of the center line, for which the same change indicates increased turbidity.

The results of the influence of interactions between the variables on the supernatant turbidity are presented in Figure 8. The flocculant concentration (A) presented strong interaction with the surfactant conditioning time (AC), the pH (AG) and the solids concentration (AJ). The surfactant conditioning time (C) also showed strong interaction with the agitation intensity (CF), the suspension pH (CG) and the solid concentration (CJ). These considerations are consistent with the values of contribution on Figure 7 for such interactions. It is important to observe (see Table 3) that these interactions, besides being of the significant variables, are also confounded with third or second order interactions of variables which are not significant to the experimental response.

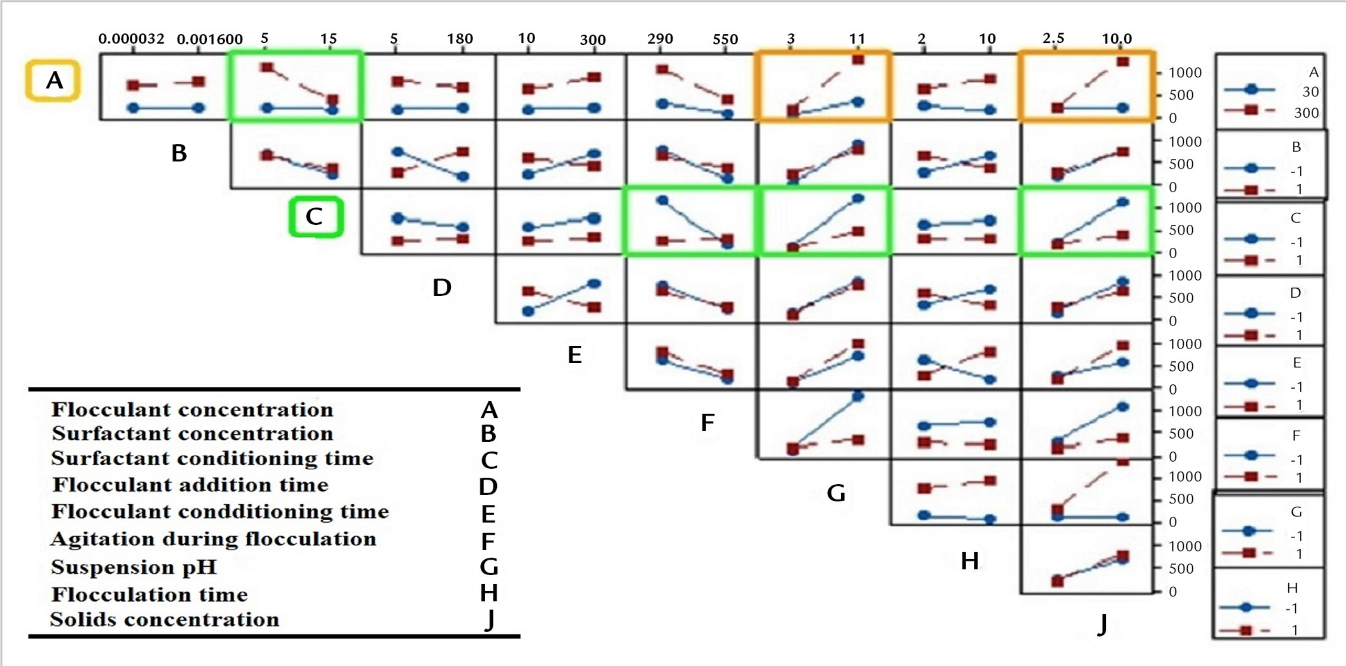

Figure 8 - Interaction of variables and its influence on the supernatant turbidity.

The results of the main effects on the supernatant turbidity are presented in Figure 9. The statistical analyses indicated that the pH was the variable with the major influence on the supernatant turbidity, followed by flocculant concentration (2nd), solids concentration (3rd), agitation intensity (4th), and surfactant conditioning time (5th).

The variables surfactant concentration (B), flocculant addition time (D), flocculant conditioning time (E), and flocculation time (H) did not present significant effects on the experimental response. The sequence of significance is: pH (G) > flocculant concentration (A) > solids concentration (J) > interaction AJ > interaction CF > agitation intensity (F) > interaction AG > surfactant conditioning time (C) > interactions CG > interaction AC.

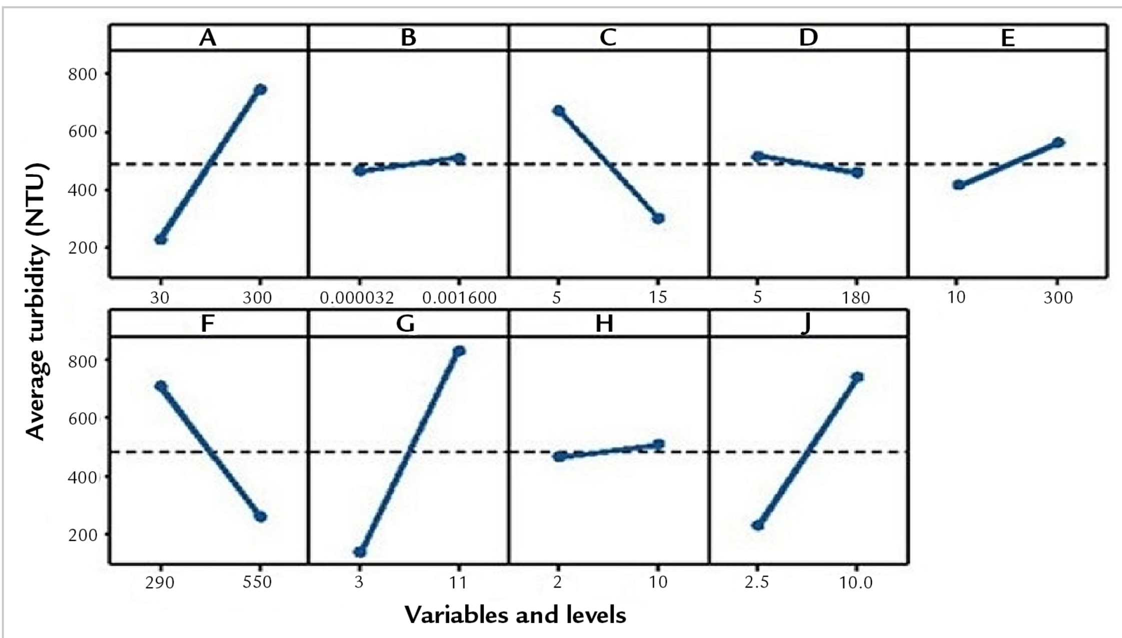

Figure 9 - Effects of the main variables on the supernatant turbidity.

4. Conclusions

The conditions of low surfactant (etheramine EDA) concentration, low flocculant (non-ionic PAM) concentrations, high agitation intensity, and acidic pH yielded significant flocculation and settling, observed by low turbidity, compressed sediment, high settling velocity and large Camp Number.

The Camp Number values showed a negative exponential relationship with the turbidity results, confirming the best flocculation conditions as well as proving to be a valuable tool to evaluate flocculation.

The flocs formed at the best condition presented high sedimentation velocity and minimal water retention, which are important characteristics for the thickening and filtration processes.

Photomicrography of the quartz sediment recorded the formation of large and compact flocs with some sphericity.

The Fractional Factorial Design Method was used for the analysis of 32 flocculation and settling experiments with ultrafine quartz suspensions. Nine variables in two experimental levels, low (-) and high (+), were evaluated using the a statistical method for processing the numeric values.

The evaluations resulting from the statistical approach adopted in this study indicated that suspension pH was the most significant variable, followed by flocculant concentration with the second greatest effect on the turbidity of the supernatant. Solids concentration, agitation intensity and surfactant conditioning time, in descending order, also showed significant effects on ultrafine quartz flocculation.

The flocculant concentration and the surfactant conditioning time showed important interactions with the main variables, in which the effects of these interactions indicate interesting contributions to the experimental response. These interactions, as well as the most significant variables, should be investigated using a second-degree model to better understand their effect on quartz flocculation.

Acknowledgements

This study was financed in part by Coordenação de Aperfeiçoamento de Pessoal de Nível Superior - Brasil (CAPES) - Finance Code 001. The authors also thank the Centro de Microscopia (UFMG), for the technical support in the SEM experiments.

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Received: 23 February 2022 - Accepted: 20 June 2022.