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Ultrafine quartz flocculation: Part I. System characterization and variables selection

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Abstract

A characterization study was performed to verify the more relevant physicochemical properties for quartz flocculation with polyacrylamide, as well as to define which variables should be further investigated regarding this flocculation system. Polyacrylamide was evaluated through molecular weight, radius of gyration, and infrared spectrometry. A natural quartz sample was investigated regarding its particle size distribution, specific surface area, mineralogical and chemical composition. The zeta potential of quartz with flocculant and surfactant was also analyzed. The results indicated that the quartz had high purity and particle sizes between 38 and 10 µm, which was within the intended particle size range; the flocculant presented a suitable molecular weight for the proposed flocculation system. Based on literature, nine variables were chosen to be investigated in Part II of this study: flocculant and surfactant concentration, flocculant and surfactant conditioning time, flocculation addition time, agitation intensity, pH of the suspension, flocculation time, and solid concentration.

keywords: quartz, flocculation, system characterization, variables.

1. Introduction

The adsorption of flocculants and surfactants depends on the microelectrophoretic behavior of minerals which can be affected by even small variations in the type and content of impurities in the mineral lattice (Al Mahrouqi *et al*., 2017).

Contaminating crystalline species in the lattice are detected by X-ray diffraction (XRD). X-ray fluorescence (XRF) is a commonly used technique to check the purity of samples analysed by XRD, since it detects elements present at concentration below the detection limit of XRD (Carvalho *et al*., 2020).

The degree of polymerization, measured by the molecular weight, determines the action of a polymer as flocculant, dispersant or depressant. Molecular weights above 1.000.000 Da are considered adequate for flocculating action (Attia, 1992). Peres *et al*. (1994) reported the use of the PAM's SPA 15 and SPA 20, with molecular weights of 1200 Da and 2500 Da, respectively, in the successful dispersion of an oxidized zinc ore.

Different classes of flocculants are commercially available, but the most commonly used are the polyacrylamides, PAM. Based on the presence of ionic groups, PAMs are classified as non-ionic, anionic, or cationic. Most

PAMs referred as non-ionic are slightly anionic containing 1-3% of anionic groups arising from the hydrolysis of amide groups (Shatat *et al.*, 2018). The ionic class is important regarding electrostatic interactions of PAMs.

Etheramines are surfactants which are fully dissociated in the acidic pH range. The dissociation degree decreases with the pH increase. The pH at which the concentrations of the ionized and molecular species are equal is designated as pKa. The pKa depends on the chain length and is not disclosed by the manufacturers. Fernandes (2017) reported pKa values of 9.02 for medium chain ethermonoamine, 8.87 for medium chain etherdiamine, and 8.17 for long chain etherdiamine. Rocha *et al.* (2021) determined the pKa of Flotigam 7100 ethermonoamine at 9.4.

Oberlerchner *et al.* (2015) related that for a given polymer-solvent pair, the intrinsic viscosity is a unique function of the molecular mass. The polyacrylamide viscosity is dependent on the shear rate, indicating that their solution is a non-Newtonian fluid. The viscosity decreases with increasing shear rate up to approximately 1200 rpm, after which, it becomes nearly constant (Jung *et al.*, 2016). Another important measure of the effective size of a polymer molecule is the root-mean-square distance of the elements of the chain from its center of gravity, designated by $\sqrt{s^2}$, referred to as the radius of gyration of the molecule (Flory, 1953).

The flocculant chain length plays a key role on the adsorption process. Its contribution on collision efficiency, bridging bond and flocs strength is significant, as well as the particle sizes, which also affect the floc formation mechanism (Otsubo, 1992). There is a relevant equivalence between the flocculant molecular weight and the particle size. The agitation of the system was also indicated among the most important in flocculation, affecting the dispersion and adsorption of the flocculant molecule and the floc formation, growing, and breakage (Bulatovic, 2007). The chemical environment of the solution strongly affects the flocculant efficiency. Hulston *et al.* (2004) reported that the variables: pH, ionic strength, dosage, mixing condition, among others, affect the floc structure.

In general, a high molecular weight polymer adsorbs simultaneously on the surface of several mineral particles, yielding aggregates designated as flocs. The higher sedimentation velocity of these flocs enhances the solid liquid separation. Nevertheless, flocs of ultrafine or colloidal particles present low settling velocities, rendering necessary the use of a surfactant to cause a previous aggregation stage (Campêlo *et al.*, 2017).

Baltar & Oliveira (1998) reported adsorption of PAM on colloidal silica at pH 3 and no adsorption at pH5.5. Guévellou *et al.* (1995) indicated that PAM adsorption on the sand surface decreased from 387 μg/m^2 at pH 9.5 to 3 μg/m^2 at pH 11.2.

McFarlane *et al.* (2005) measured the stirring speed in the range between 60 and 500 rpm. Better sediment consolidation was achieved in the speed range below 200 rpm and enhanced collision and aggregate formation conditions were observed between 200 and 300 rpm. It was also observed, through parabolic curves of sedimentation rate that, in this condition, the collisions were sufficient for reaching maximum flocculant adsorption and particle aggregation, keeping floc breaking at a minimum level. Owen *et al.* (2002) determined the formation of large and voluminous aggregates, as well as a high sedimentation rate, at 100 rpm stirring speed.

Regarding flocculant concentration, Owen *et al.* (2002) and Al-Hashmi *et al.* 2012) investigated the range between 30 and 100 g/t. Surfactant concentrations of 1×10^{-5} and 5×10^{-3} were used, respectively, by Baltar & Oliveira (1998) and (Campêlo *et al.* 2017). Flocculant conditioning times of 15, 120, and 180 seconds were used, respectively, by Addai-Mensah *et al.* (2007), Ofori *et al.* (2011), Campêlo *et al.* (2017). Surfactant conditioning times of 15, 10 and 5 minutes were selected, respectively, by Raju *et al.* (1991), Lu & Song, (1991), Campêlo *et al.* (2017).

Solid concentrations between 10 and 45 mg/L were reported by Baltar & Oliveira (1998) and Owen *et al.* (2002). Flocculation times between 3 and 10 minutes were mentioned by McFarlane *et al.* (2005) and Campêlo *et al.* (2017). However, none of the references consulted addressed the effects of the variables and their interactions on the flocculation process. In this sense, the knowledge of the variable interactions in a flocculation system is of high relevance concerning the process efficiency. Therefore, the motivation of this investigation was to know the characteristics of the non-ionic polyacrylamide PAM and of the natural quartz, as well as to evaluate and define the variables that affect the destabilization of an aqueous suspension of ultrafine quartz.

The multiple variables of the mineral slurry flocculation process rendered this Part I of the study essential for the knowledge of the physicochemical properties of the flocculant non ionic polyacrylamide (PAM), the characteristics of the natural quartz sample, the electrokinetic behavior of quartz after adsorption of the flocculant (PAM) and surfactant (amine EDA). These results were used for the definition of the variables and their levels to be used in the flocculation system, Part II of the study.

2. Materials and methods

The quartz sample used in the study was collected at Corinto city in Minas Gerais, Brazil, presenting crystals of approximately 15 cm which weighed a total of 3.5 kg. The sample was subjected to comminution and classification steps, whereupon a fraction in the particle size range between 38 and 10 μm was separated. To evaluate the role of surfactants on flocculation, a sample of etheramine Flotigam EDA (Clariant, Brazil) was obtained. The non-ionic polyacrylamide (PAM) was investigated as the flocculant, derived by polymerization of acrylamides, being also provided by Clariant (Brazil). Hydrochloric acid and sodium hydroxide, manufactured by Synth, were used as pH regulators.

The reagents were methylene blue, bromophenol blue, chloroform and were used in chemical analyses for the determination of charged surfactant groups present in the flocculant chemical structure, according to the methodology described by Jiang *et al.* (2014).

A Mastersize Micro (Malvern Instruments, United Kingdom) was used for particle size analysis and a S2 Ranger energy dispersion X-ray fluorescence spectrometer (Bruker, Germany) was used to evaluate the purity of the sample. The sample was also analyzed using an Empyrean X-ray diffraction in a diffractometer (Panalytical, United Kingdom). The specific surface area was analyzed using the BET multipoint method, in a NOVA-1000 surface area analyzer (Quantachrome Instruments, Germany).

The PAM was analyzed using Alpha II FTIR spectrometer (Bruker, Germany) ATR mode, 32 scans, at 4 cm^{-1} resolution. The PAM molecular weight was determined by its inherent viscosity, with the use of a DV-I Prime viscosimeter (Brookfield, USA). The radius of gyration of the flocculant molecule was then calculated from the values of inherent viscosity and molecular weight.

The zetameter ZD3-D-G 3.0+ (Zeta Meter, USA) was used for zeta potential determinations of quartz prior to and after PAM and surfactant adsorption. Mineral aqueous suspensions were prepared in the presence of: (i) supporting electrolyte (10^{-3}M of NaCl), PAM (300g/t) and surfactant (10^{-4}M).

3. Results and discussion

The result of the particle size analysis shown in Figure 1, indicates that 100% of the material is < 38 µm and 28% is < 10 µm.

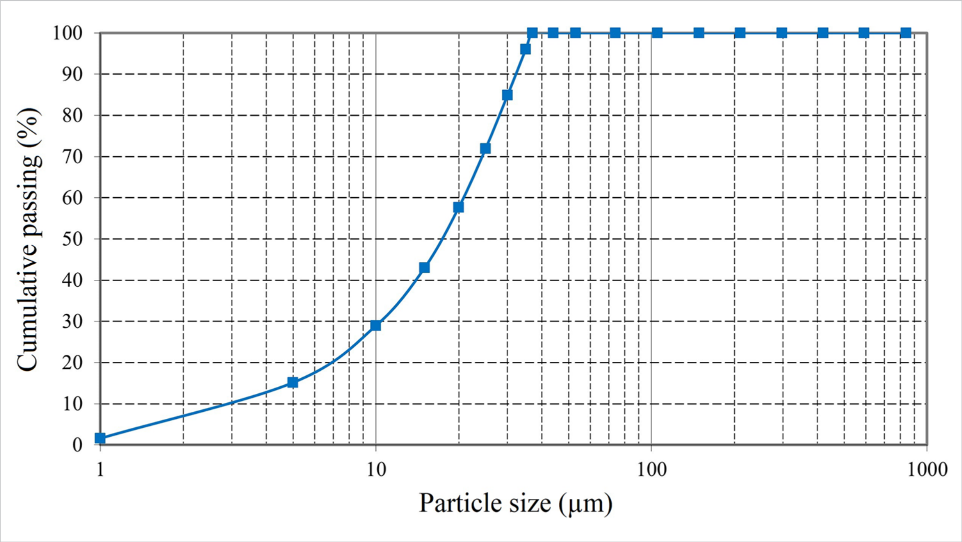


Figure 1 - Particle size analysis of the quartz sample.

The results of the chemical analysis and the X-ray diffractogram of the quartz sample are shown, respectively, in Table 1 and Figure 2. It is observed that Si is the major element in the sample, being directly related to the mineral composition, which only identified quartz. No other mineral phases, which could be associate to other elements, were observed.

Table 1 - Chemical analysis of the quartz sample.

LOI	Al_2O_3	CaO	Cr_2O_3	Fe_2O_3	K_2O	MgO	MnO	Na_2O	P_2O_5	SiO_2	TiO_2
0.13	<0.1	<0.01	<0.01	0.01	<0.01	<0.1	<0.01	<0.1	<0.01	99.72	0.01

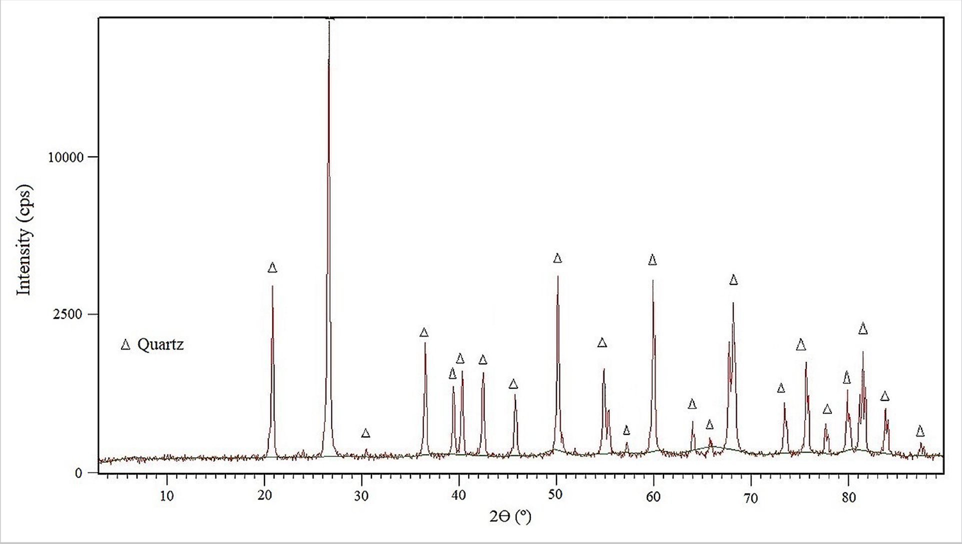


Figure 2 - X-ray diffractogram of quartz sample (λKα Cu=1,54060Å).

Figure 3 displays the result of the specific surface area of the quartz sample, obtained via BET multipoint analysis. The results show that the specific surface area is 0.496 m^2/g.

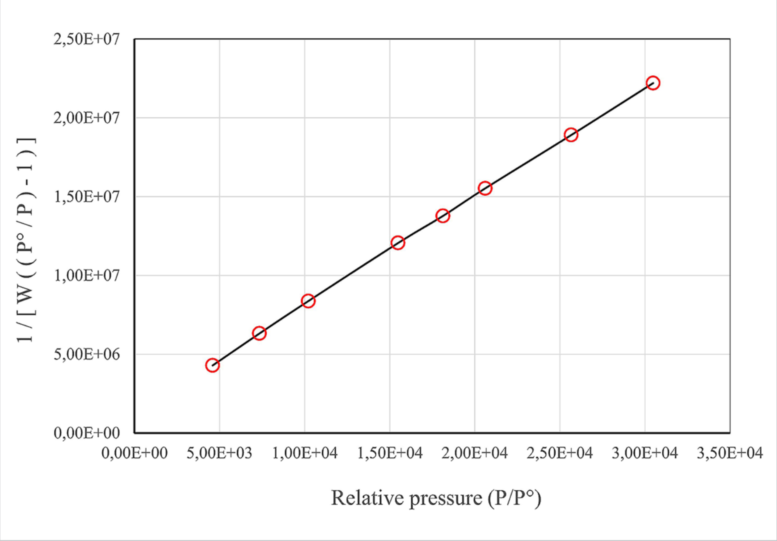


Figure 3 - BET multipoint curve of the quartz sample.

Figure 4 shows the result of the PAM inherent viscosity. From extrapolation of the curve, the viscosity value found was 5.35 dl/g. Using Equation 1, given by Mark-Houwink-Sakurada (Oberlerchner *et al.*, 2015):

$$[\eta] = KM^{\alpha} \qquad (1)$$

Where: M is molar mass and K and α are the polymer and system constants (Barnes, 2000). Considering the values of 3.7×10^{-4} and 0.66 for *K* and α, respectively, the molecular weight value of the flocculant was obtained:

$$M = (5.35 / 3.7 \times 10^{-4})^{1/0.66} = 2.0 \times 10^{-6}\ g.mol^{-1}.$$

The calculated PAM molecular weight, 2.0×10^{-6} $g.mol^{-1}$, is within the range considered ideal for flocculation (Baltar & Oliveira, 1998).

The radius of gyration (R_g) of the molecule was calculated using the inherent viscosity value and the molecular weight, using Equation 2 (Otsubo & Watanabe, 1990).

$$[\eta_{iner}]\, M_w = 6^{3/2}\, \Phi\, (R_g^{\,2})^{3/2} \qquad (2)$$

Where $(R_g^{\,2})3/2$ is the root mean square of gyration and Φ is the Flory-Fox parameter whose adopted value of 2.2 x 1021 $dl.mol^{-1}.cm^{-3}$ was suggested by Newman et al. (1954) and Otsubo & Watanabe (1990) and used by Baltar & Oliveira (1998). Therefore, the calculated value of the radius of gyration is 65.2 nm. The radius of gyration of the molecule is much smaller than the particles size. On flat surfaces, the thickness of the adsorbed polymer layer is roughly of the same order of magnitude as the radius of gyration of an isolated polymer chain in solution (Otsubo, 1992).

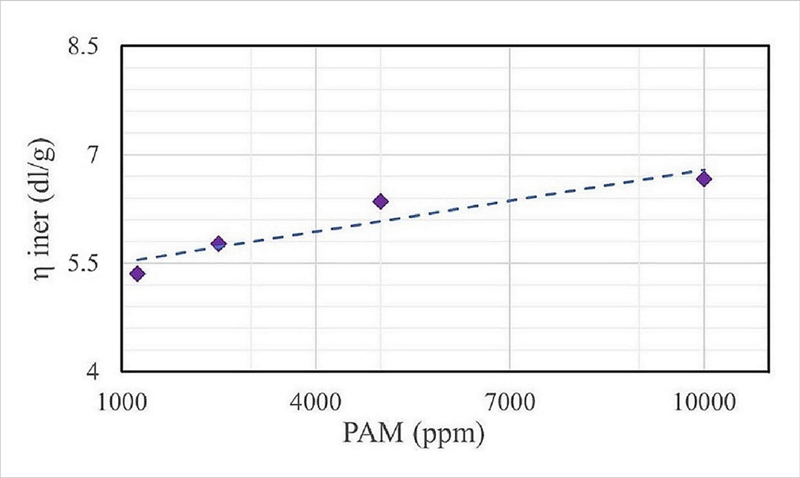


Figure 4 - Viscosity variation with the concentration of the PAM in the presence of NaCl (1M). Viscosity determined at 25°C at a shear rate of 100 rpm.

Figure 5 presents the results of the investigation on the presence of cationic, anionic and non-ionic groups in the PAM structure. After reaction with bromophenol blue, the PAM solution did not present the blue color characteristic of cationic groups (image A). After the reaction with methylene blue and chloroform, the color of the solution changed to dark blue as the only color (image C), indicating the presence of non-ionic groups. If anionic groups were present, the solution would present different shades of blue in different layers.

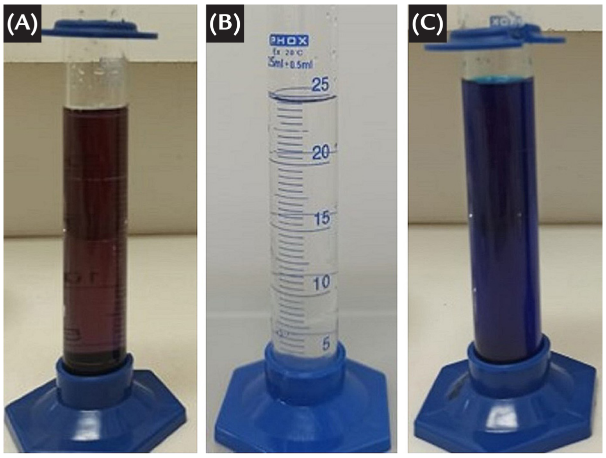


Figure 5 - (A) PAM + bromophenol blue, (B) PAM blank (C) PAM + methylene blue + chloroform.

The result of the FTIR analysis of non-ionic PAM is shown in Figure 6.

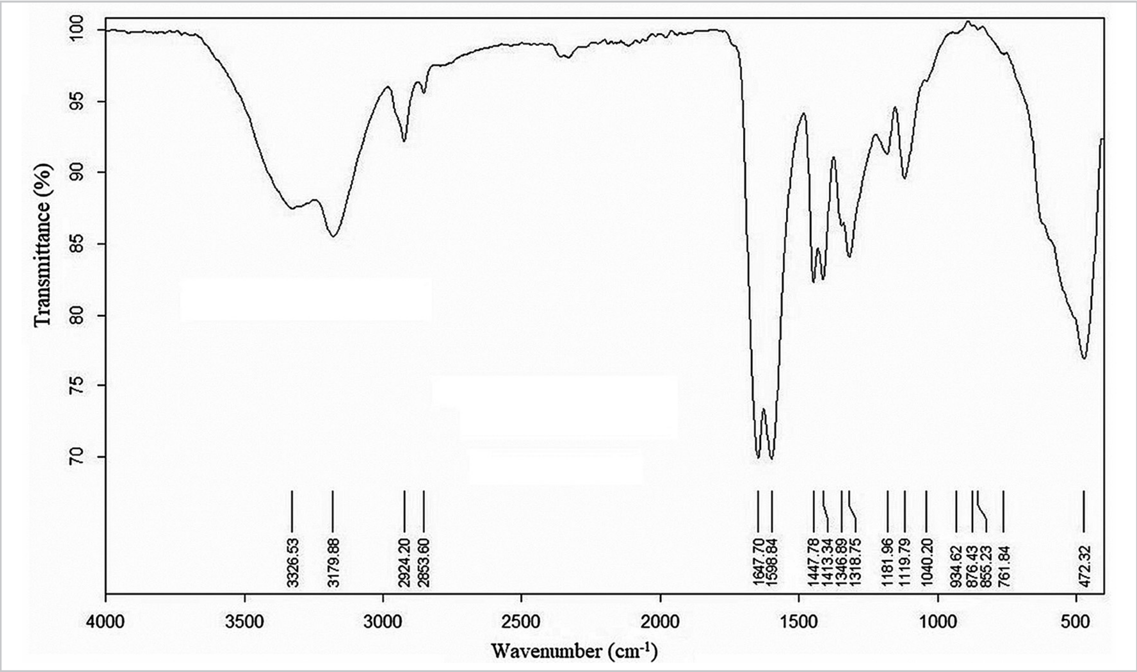


Figure 6 - FTIR spectra of the PAM at wavelength between 500cm^{-1} and 4000 cm^{-1}.

Bands between 472 and 1040 cm^{-1} are indicated as shake and twist movements of the NH_2 group. There are two important stretching vibrations C – C in 1612 and 1650 cm^{-1}, such vibrations were noted by Murugan *et al.* (1998) in 1125 and 1176 cm^{-1}. These vibrations can be observed, in the spectrum of figure 6, at the wavelengths of 1119 and 1181 cm^{-1}.

The amide group has three characteristic peaks: 1672 cm^{-1}, attributed to stretching vibration – C = O; 1610 cm^{-1} referring to flexion vibration – NH_2 and the 1425 cm^{-1} band, indicated to stretching vibration – CN by Chiem *et al.* (2006), who also observed a characteristic peak in 1458 cm^{-1} attributed to scissor vibration – CH_2.

The two broad peaks observed at 1598 and 1647 cm^{-1} are attributed to NH_2 deformation vibration and C – O vibration, respectively. The wavelengths at 2853 and 2924 cm^{-1} are associated with the symmetrical stretching of CH_2. The two high intensity bands at 3326 and 3179 cm^{-1} are attributed to the N – H stretching vibrations. Analysis of the normal coordinates of polyacrylamide predicts the bands at 3338 and 3171 cm^{-1}, noted by Murugan *et al.* (1998) at 3335 and 3198 cm^{-1}, referring to the asymmetric and symmetric stretching vibrations of NH_2, respectively.

Therefore, the spectra of scissor vibration – CH_2 and stretching vibrations of NH_2 and – C = O indicate the presence of polyacrylamide main groups as alkane and carboxamide, respectively.

Table 2 presents the results of the PAM FTIR analysis.

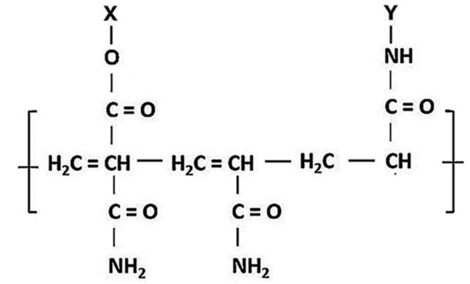
The proposed structure and composition for the PAM is presented in Table 3.

Table 2 - PAM FTIR bands and assigned functional groups.

Wave number (cm^{-1})	Vibration	Functional group	References
472 - 1040	wagging and twisting vibration – NH_2	carboxamide	Jiang *et al.* 2014; Murugan *et al.* 1998
1119 - 1181	stretching vibration C – C	alkyl ethers	Murugan *et al.* 1998; Jiang *et al.* 2014
1318	deformation vibration –CH	secondary aromatic amines	Murugan *et al.* 1998 Jiang *et al.* 2014
1346	wagging vibration – CH_2	secondary aromatic amines	Murugan *et al.* 1998 Jiang *et al.* 2014
1413	vibration – C – N	carboxamide	Murugan *et al.* 1998; Chiem *et al.* 2006; Jiang *et al.* 2014
1447	scissoring vibration – CH_2	alkanes	Chiem *et al.* 2006
1598	bending vibration a – NH_2	carboxamide	Murugan *et al.* 1998; Chiem *et al.* 2006; Jiang *et al.* 2014
1647	stretching vibration C = O	carboxamide	Murugan *et al.* 1998; Chiem *et al.* 2006; Jiang *et al.* 2014
2853 e 2924	asymmetric and symmetric stretching – CH_2	alkanes	Murugan *et al.* 1998; Chiem *et al.* 2006
3179 e 3326	stretching vibration – NH_2	carboxamide	Murugan *et al.* 1998; Chiem *et al.* 2006

Table 3 - Proposed structure and composition for the PAM.

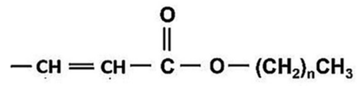
Structure and composition:



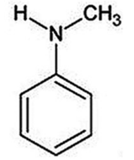
where:

X = alkylethers $(CH2 - CH2 - O)_m - R$

Y = polyesther



Y = aromatic secondary amine



The zeta potential results of the quartz suspension in the presence of supporting electrolyte NaCl (10^{-3}M), PAM (300g/t), and surfactant (10^{-4}M) are shown in Figure 7.

The zeta potential values in the presence amine in the pH range above the PIE, pH 2, are in agreement with the less negative surface charge of the mineral. This is explained by the adsorption of the cationic amine species on the quartz surface by an electrostatic attraction mechanism and immobilization by formation of hemimicelles proposed by Fuerstenau & Palmer (1976) and still presently accepted (Baltar, 2021).

The PAM adsorbs on the hydrophilic surface of quartz with excess negative charge through two mechanisms, first by electrostatic attraction and second by Van der Waals, adsorbing by hydrogen bonds with the PAM NH_2 group Baltar (2021), therefore decreasing the amount of negative sites from the surface and, consequently, rendering the zeta potential value less negative.

When considering H bonding between the polyacrylamide and the adsorbent, Lee & Somasundaran (1989) indicated that, more likely, the electronegative C=O function of the amide acts as an H-bonding base and the oxide surface hydroxyls as an H-bonding acid. Therefore, not only the neutral undissociated SiOH group but also the positive $SiOH^{2+}$ group can act as proton donors.

Although H-bonding is not considered as an electrostatic interaction, the fact that it is a bond between an electronegative and an electropositive group could render it charge dependent, therefore the positive $SiOH^{2+}$ group should be at least as favorable or even more favorable than the neutral SiOH to form an H bond with the C – O (Lee & Somasundaran, 1989).

For both reagents, adsorption was favored near the isoelectric point and decreases with increasing pH, indicating that adsorption decreases as the oxide surface charge increases.

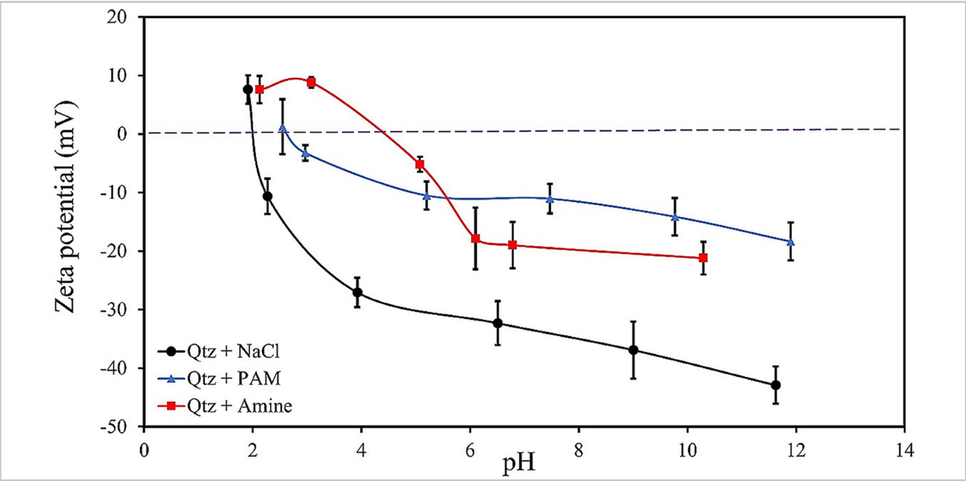


Figure 7 - Quartz zeta potential in the presence of (i) supporting electrolyte NaCl (10^{-3}M), (ii) PAM (300g/t), (iii) surfactant (10^{-4}M).

Knowledge of the properties of the system ensures better control of flocculation, in addition to assisting in the identification and evaluation of variables in the flocculation system under study.

The main flocculation variables and ranges found in literature are presented in Table 4. As each system is dependant on its unique physicochemical properties, the best flocculation conditions for quartz with PAM may be significantly different from those listed in Table 4, making it important to conduct an evaluation of these conditions and the variables synergistic interactions in the light of the characterization conducted in the present study.

Table 4 - Main variables and conditions found in flocculation systems.

A		B		C	
PAM (g.t^{-1})	**References**	**Amine (mol. L^{-1})**	**References**	**Surfactant conditioning time (min)**	**References**
30 – 86	Owen *et al*., 2002	3.86 x 10^{-4}	Lu & Song, 1991	10	Lu & Song, 1991
250	McFarlane *et al*., 2005	2.26 – 27.07 x 10^{-5}	Raju *et al*., 1991	15	Raju *et al*., 1991
15 – 100	Cengiz *et al*., 2009	1 – 8 x 10^{-5}	Baltar & Oliveira, 1998	5	Baltar & Oliveira, 1998
50 e 100	Al-Hashmi & Luckham, 2010	0,1 – 5 x 10^{-3}	Campêlo *et al*., 2017	5	Campêlo *et al*., 2017
15; 40; 80 e 100	Al-Hashmi & Luckham, 2012				
5 – 25	Campêlo *et al*., 2017				

D		E		F	
Addition time (s)	**References**	**Flocculant conditioning time (s)**	**References**	**Agitation intensity (rpm)**	**References**
180	Attia & Fuerstnau, 1980	180	Attia & Fuerstnau, 1980.	200	Gregory, 1988
60	Scott *et al*., 1996	15	McFarlane *et al*., 2005	250 - 1.500	Scott *et al*., 1996
5	McFarlane *et al*., 2005	15	Addai-Mensah *et al*., 2007	100	Baltar & Oliveira, 1998
15	Cengiz *et al*., 2009	120	Cengiz *et al*., 2009	75 - 400	Owen *et al*., 2002
180	Campêlo *et al*., 2017	120	Ofori *et al*., 2011	60 - 500	McFarlane *et al*., 2005
		180	Campêlo *et al*., 2017	350	Alagha, L. *et al*., 2016
				300	Campêlo *et al*., 2017

G		H		I	
pH	**References**	**Flocculation time (min)**	**References**	**Solids (g/L)**	**References**
3 - 12	Lee e Somasundaran, 1989	4	Attia e Fuerstnau, 1980.	30	Lee & Somasundaran 1989
4 - 8	Lecourtier *et al*., 1990	5	Scott *et al*., 1996	20	Page *et al*., 1993
7 - 11,3	Guévellou *et al*., 1995	10	McFarlane *et al*., 2005	10	Baltar & Oliveira, 1998
3,5 - 5,5	Baltar & Oliveira 1998	2	Cengiz *et al*., 2009	45	Owen *et al*., 2002
2	Bessaies-Bey *et al*., 2018	3	Câmpelo *et al*., 2017	24	Câmpelo *et al*., 2017

4. Conclusions

The present work characterized a quartz flocculation system. The quartz mineral sample with a high degree of purity, in the size range is between 38 and 10 µm and specific surface area of 0.496 m^2/g.

The calculated molecular weight of the non-ionic PAM, 2.0 x 10^{-6} $g.mol^{-1}$, is within the range considered ideal for flocculation and the gyration radius of the molecule was determined to be 65.2 nm.

The FTIR spectrum showed the vibrations of the non-ionic PAM main constituents, carboxamide and alkane.

Electrokinetic determinations of quartz in the presence of the surfactant etheramine EDA showed strong adsorption near isoelectric points. The adsorption of the non-ionic PAM is favored near the isoelectric point and adsorbs by two adsorption mechanisms, electrostatic and Van der Waals binding.

The characterization of the system is relevant for the control of the flocculation process, since the physicochemical properties of each system plays an important role in the definition of its optimal operation conditions. The main variables identified in literature should then be investigated in detail to better understand the influence of these properties in flocculation.

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