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Anionic polyacrylamide efficiency in goethite removal from aqueous solutions: goethite suspension destabilization by PAM

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Abstract

The main aim of the study was to investigate the anionic polyacrylamide (PAM) efficiency as a flocculant relative to goethite particles. This mineral is a toxic waste produced during hydrometallurgical processes and, as a result, poses a threat to the environment. In the experiments, the following analytical methods were applied: potentiometric titration, zeta potential and adsorption amount measurements, nitrogen adsorption/desorption isotherms, Fourier transform infrared spectroscopy, differential centrifugal sedimentation, UV/Vis spectrophotometry. The result analysis showed that polyacrylamide adsorbs on the goethite surface; however, its adsorption amount depends on the pH value. Due to the adsorbent–adsorbate electrostatic repulsion, the smallest number of macromolecules adsorbs at pH 9. The PAM adsorption affects the goethite surface charge and zeta potential values. It makes these parameters more negative. What is more regardless of pH value, the polymer addition contributes to clear aggregation of solid particles (suspension destabilization). Thus, PAM can be considered as a potential flocculant improving the goethite removal from aqueous wastes.

Keywords Goethite · PAM · Wastewaters · FTIR · Zeta potential · Aggregate size

Introduction

Goethite is an oxide mineral occurring in many regions, i.e. in the USA, Great Britain, Germany, the Czech Republic, Slovakia, Algeria, Morocco, Canada, Australia, Cuba, Russia and Poland (Hochleitner 2010). It is a primary mineral in main iron ores as well as important source for yellow–brown pigment (ochre). Goethite is composed of iron oxide (80–90%) and water (about 10%). This is a ferric hydroxide oxide of the following formula: α-FeOOH. In the goethite crystals, oxygen and hydroxyl anions are packed hexagonally in arrays, whereas all iron atoms are coordinated octahedrally (Britannica 1998).

Goethite is produced as a by-product in hydrometallurgical processes, mainly during zinc production. In this case, it is a toxic waste because of the heavy metal presence (Pb, Cd, As, etc.). Due to its large amount, goethite waste creates a big environmental problem (Pelino et al. 1996; Di Maria and Van Acker 2018). Usually, they are landfilled, which is highly expensive and dangerous for the environment. Many scientists conduct research on the management or avoiding of the goethite waste. For example, Di Maria and Van Acker (2018) presented the goethite valorization strategy including plasma fuming and inorganic polymerization of the fumed slag. In turn, Pelino et al. (1996) prepared a glass by mixing wastes with sand, tuff, feldspar, etc. Goethite landfill is not the only environmental problem. As a yellow–brown dye, this substance is widely used in various industries (e.g. paint and pharmacy). Thus, its presence in sewage and wastewaters is inevitable. As a colourful substance, goethite gives aqueous solutions undesirable colour and turbidity which hinder photosynthesis and other life processes.

The goethite removal may be performed by many methods. One of them is flocculation, which occurs in destabilized colloidal systems after the flocculating agent addition (usually a polymeric compound—polyelectrolyte). The flocculant adsorbs on the solid surface mainly through the hydrogen bridges or electrostatic interactions. It forms

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'bridges' between the particles which accelerates solid aggregation (Adamski 2000).

One of the flocculants is polyacrylamide (PAM), used in agriculture and wastewater treatment. This substance improves the soil structure and water–air conditions (Chibowski 2011; Bronick and Lal 2005; Kim et al. 2013). Moreover, it makes the solid removal from wastewaters easier (Adamski 2000). Some researchers examined the PAM use possibilities and its impact on the environment. Buczek et al. (2017) examined the toxicity of the PAM flocculants for early life stages of freshwater mussels. He et al. (2017) studied the sedimentation behaviour of the soil slurry after the flocculant addition. Guezennec et al. (2015) studied the polyacrylamide transfer and degradation in hydrosystems. Mclaughlin and Bartholomew (2007) presented the soil factors influencing the flocculation process accelerated by polyacrylamide. Zhao et al. (2018) described the most favourable conditions for soluble cadmium removal using the anionic PAM-PAC composite. Several scientists are involved in the removal process modelling focusing on selected pollutants (Zhou et al. 2017).

Polyacrylamide use in the wastewater treatment is very advantageous. Compared to other substance (e.g. alum and ferric chloride) use, it requires lower dosage and reduces the amount of sludge produced. Moreover, the polymer is able to connect smaller solid particles and the formed flocs are more resistant to shearing (Ebeling et al. 2005).

In this paper, the anionic polyacrylamide efficiency in goethite removal from aqueous solutions was examined. In other words, the probable stability mechanism of goethite aqueous suspension was described. The experiments were started from potentiometric titration, Fourier transform infrared spectroscopy (FTIR) and nitrogen adsorption/desorption isotherm method, which allowed the goethite characterization. Then the particle electrokinetic potential, in the absence and presence of the polymer, was measured. In this way, the electrical double-layer (EDL) structure formed around the goethite particles, with and without polyacrylamide, was proposed. The adsorption amount measurements performed were helpful in description of the PAM adsorption mechanism on the goethite surface, which is the first step during the flocculation process. The polyacrylamide flocculating ability was checked based on the measurements of suspension stability and aggregate size formed in the system. All above experiments were performed as a function of the pH value. Owing to it, the conditions, under which PAM is the most effective flocculant, were established.
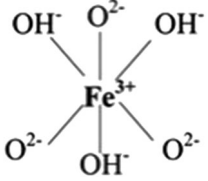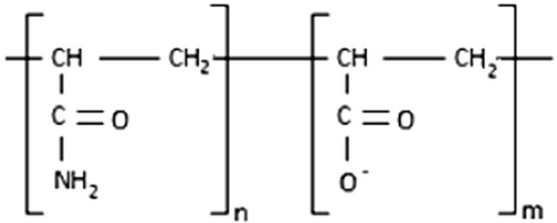
Materials and methods

Materials

Goethite (*Aldrich Germany*, CAS 20344-49-4) was chosen for the research. Its exact composition was determined using the X-ray fluorescence (*Axios mAX*, *PANalytical*, Holland). The selected mineral contained the following elements: Fe (62.2%), S (0.308%), Si (0.0476%), Al (0.0427%), Zn (0.0369%), Na (0.0227%), Cr (0.0192%), Mn (0.0122%), P (0.00785%), Ca (0.00524%) and As (0.00461%).

Anionic polyacrylamide (PAM, *Korona*) was characterized by average molecular weight (M_w) equal to 13,000 kDa and 40% share of ionizable groups. Its pK_a parameter was equal to 5.3, whereas the dissociation degree (α) was as follows: at pH 5—0.33, pH 5.3—0.5, pH 7—0.98, pH 9—0.99 (Szewczuk-Karpisz et al. 2018). The solid and polymer characteristics are presented in Table 1.

Methods

Nitrogen adsorption/desorption isotherms method was used to determine the porosity (total pore volume—V_p, pore diameter—D_p) and specific surface area (S_{BET}) of

Table 1 Adsorbent and adsorbate formulae

Name	Formula	Structure
Goethite	α-FeOOH	
Anionic polyacrylamide	$(C_3H_5NO)_n(C_3H_3O_2)_m$	

goethite. The measurements were taken using an analyser *3Flex* (*Micromeritics*, USA). Specific surface area was calculated based on the BET equation as well as capacity of the monolayer formed on the solid surface. In turn, the goethite porosity parameters were established using nitrogen desorption isotherms. Before the measurement starts, all probes were dried and out-gassed at 105 °C for 12 h.

Goethite characterization also included Fourier transform infrared spectroscopy (FTIR), which identifies surface functional groups. The apparatus used was a spectrometer *Tensor27* (*Bruker*, Germany). The pellets were prepared by mixing 1 mg of goethite with 100 mg of KBr. They were pressed by *Specac*. Both goethite and KBr were dried at 105 °C before the measurement. The FTIR spectra were obtained by 256 scans with the resolution 2 cm^{-1}.

Polyacrylamide concentration, before and after the adsorption process, was measured spectrophotometrically (*Jasco V-530*) using hyamine 1622 (Kang et al. 2013). The obtained difference in PAM concentration indicated the polymer adsorption level on the goethite surface. The probes were prepared by adding 0.04 g of goethite to the solution (10 ml) containing the supporting electrolyte (0.0001 M NaCl) and polymer (10–200 ppm). The process was carried out for 24 h using an orbital shaker *Sky Line*, at various pH values (5, 7 or 9). After its completion, the samples were centrifuged (10,000 rpm).

The goethite surface charge density (σ_0) was determined by a potentiometric titration method (Janusz et al. 1997). A titrator *Titrino 702 SM* (*Metrohm*) was used. The probes were titrated by 0.1 M NaOH, from pH 3.5 to 10. They were prepared by adding 0.4 g of goethite to the solution (10 ml) containing only supporting electrolyte (0.0001 M NaCl) or simultaneously supporting electrolyte and polymer (100 ppm).

Electrophoretic mobility of goethite particles was measured by zetameter *Nano ZS* (*Malvern*). Based on the obtained results and Henry's equation (Oshima 1994), the particle zeta potential (ζ) was calculated. In the sample preparation, 0.01 g of goethite was added to 100 ml of the supporting electrolyte solution (0.0001 M NaCl). Then the suspension was sonicated (3 min) and divided into eight identical parts. Each of the obtained probes has different pH value (3–9).

Stability measurements were taken using a UV/Vis spectrophotometer (*Jasco V-530*). A single experiment lasted 2.5 h, during which the absorbance was measured every 10 s. The study was carried out at the wavelength 630 nm, at which a clear proportionality between the light absorbance and amount of suspended particles occurs. The probes were prepared by adding 0.04 g of goethite to the supporting electrolyte solution (0.0001 M NaCl) containing or non-containing polyacrylamide (0–100 ppm). Then the system pH value (3–9) was adjusted and the measurement was started.

The size of goethite particles and aggregates was determined using a differential centrifugal sedimentation method (*CPS analyser*, *CPS Instruments*). Based on the light absorption changes, the particle weight and concentration were calculated. The suspensions were prepared by addition of 0.04 g goethite to 10 ml of the supporting electrolyte (0.0001 M NaCl) in the absence and presence of polyacrylamide (1–100 ppm). The disc rate was 2200 rpm. 8% and 24% sucrose were used in the gradient formation.

Results and discussion

Goethite characteristics

Fourier transform infrared spectroscopy (FTIR) showed the functional groups on the goethite surface. The obtained spectrum is presented in Fig. 1a, and there were observed the following bands:

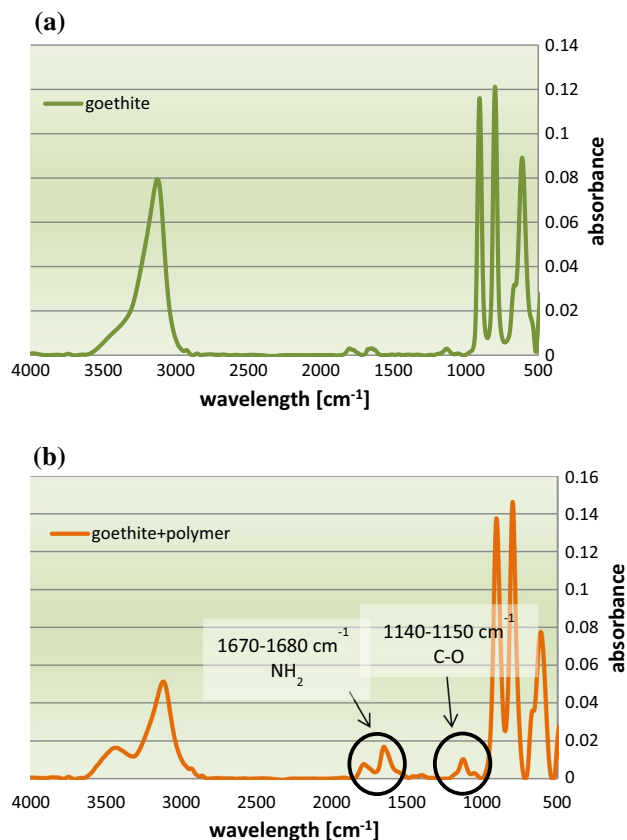- 3145 cm^{-1} corresponding with stretching of OH groups forming hydrogen bonds,

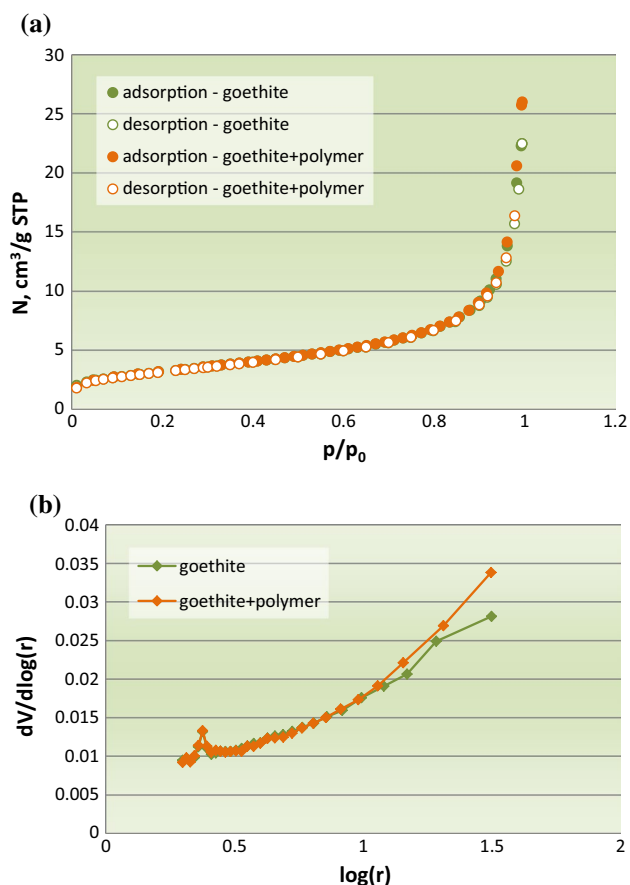**Fig. 1** FTIR spectrum for: goethite (**a**) and goethite with polymer adsorbed (**b**)

(a)

(b)

Fig. 2 Nitrogen adsorption/desorption isotherms at 105 °C (**a**) and BJH pore size distribution (**b**) of goethite with and without polyacrylamide

Table 2 Parameters characterizing the porosity of goethite and goethite with polymer obtained from the nitrogen adsorption/desorption isotherms at 105 °C: the specific surface area—S_{BET}, the total pore volume—V_p, the pore diameter—D_{PSD} at the maximum of PSD obtained from the N_2 desorption branch and $D_{4V/A\ BET}$

Sample	S_{BET} (m²/g)	V_p (cm³/g)	D_{PSD} (nm)	$D_{4V/A}$ (nm)
Goethite	11.10	0.034	4.7	12.2
Goethite + polymer	11.08	0.038	4.7	13.7

- 1690 cm^{-1}—both bending of OH groups and stretching of H_2O molecules on the surface,
- 908 and 802 cm^{-1}—the FeOOH bending,
- 619 cm^{-1}—the FeO stretching.

The last two bands are typical of goethite (Pigna et al. 2005).

Textural characterization of goethite, with and without the polymer adsorbed, was analysed by the N_2 sorption experiment (data present in Fig. 2 and Table 2). The

obtained nitrogen adsorption/desorption isotherms represent type IV with the H3 hysteresis loop (Fig. 2a), which describes a multilayer adsorption with capillary condensation on mesoporous materials. The H3-type hysteresis loop is present in aggregate, parallel corrugate pellets with slit-like pores typical of clay minerals. Pore size distributions (Fig. 2b) calculated from the desorption branch of nitrogen isotherms using the BJH procedure prove the mesoporous character of both samples.

Small values of the porosity parameters collected in Table 2 are typical of that type of clay minerals (Ismadji et al. 2015). What is surprising the polymer adsorption on the goethite particles does not change its adsorption parameters significantly. It can be suggested that the PAM adsorption amount is too small to decrease the solid specific surface area and pore volumes. However, as it will be shown below, it is sufficient to change other physicochemical properties (e.g. electrokinetic ones) of the goethite particles. This is a positive aspect because the usage of the investigated polymer, as a flocculating agent acting at low concentration, is non-destructive for the mineral adsorptive properties.

The PAM adsorption amount on the goethite surface

The anionic polyacrylamide adsorption amounts on the goethite surface are presented in Fig. 3. This polymer adsorbs on the mineral surface in the whole examined pH range, but the higher the pH value is, the fewer the PAM macromolecules are bound to the solid. The above dependence is associated with the changes in the polymer dissociation degree and the character of mutual interactions between the system compounds.

The polymer dissociation degree (α) is strongly dependent on the pH value. This parameter increases with the solution pH growth. As it was mentioned above, at pH 5 $\alpha = 0.33$, which is equivalent to the dissociation of 33% carboxylic groups in the PAM macromolecules. This amount is not large, so within the polymer chains between the segments
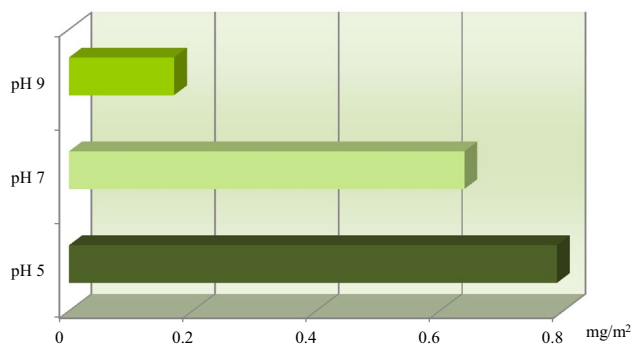

Fig. 3 PAM adsorption amount on the goethite surface as a function of pH value

there is no strong electrostatic repulsion. As a result, the macromolecules assume a coiled conformation. During the adsorption on the goethite surface, a single chain occupies a small fragment and owning to it, a relatively large PAM amount can bind to the surface.

The pH increase is connected with the higher and higher PAM dissociation degree. At pH 7, it is equal to 0.98, whereas at pH 9 it is 0.99. Such high values indicate that almost all carboxylic groups are dissociated ($–COO^-$) and almost all polymer segments are negatively charged. Thus, the PAM fragments repel each other, and as a result, the macromolecules have a much expanded structure. On the goethite surface, they occupy large parts blocking many active sites. As a result, the adsorption amount decreases significantly.

The evident difference in the adsorption amount between pH 7 and 9, despite similar polymer conformations, is related to the nature of electrostatic interactions between the adsorbent and adsorbate. At pH 7, the solid surface is positively charged—the charge density is about $+1.8$ $\mu C/cm^2$. Therefore, there is an electrostatic attraction between the system compounds (i.e. positive solid and negative polymer) that can promote PAM adsorption. On the other hand, at pH 9 the goethite surface is negative—the charge density is about -1.7 $\mu C/cm^2$. This indicates that the electrostatic repulsion occurs between the solid and polymer which makes their contact difficult.

It is also worth mentioning that at pH 5 the goethite surface charge density is the same as at pH 7. Therefore, the strength of electrostatic forces acting between the system compounds is identical. Thus, it can be stated that a higher adsorption amount observed at pH 5 is mainly due to the coiled conformation of the polymer macromolecules.

The polyacrylamide adsorption on the goethite surface was confirmed by FTIR spectroscopy. The spectrum obtained for the sample after the adsorption process is presented in Fig. 1b. In this diagram, except the bands typical for goethite, the bands characteristic of the polymer were noticed. There were:

- 1670–1680 cm^{-1} corresponding with N–H deformation,
- 1140–1150 cm^{-1}—the C–O stretching.

This means that polyacrylamide was present on the solid surface. The spectrum obtained for polyacrylamide has been already published (Szewczuk-Karpisz et al. 2018).
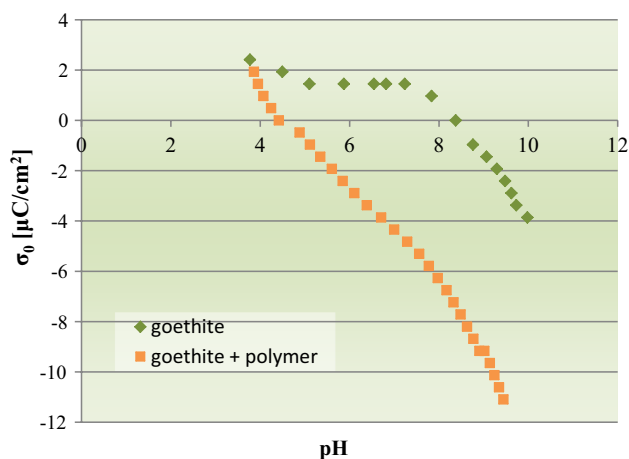

Fig. 4 Goethite surface charge density in the absence and presence of polyacrylamide
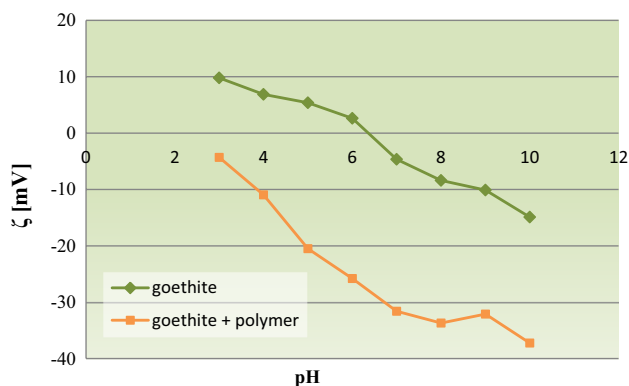

Fig. 5 Goethite electrokinetic potential with and without polyacrylamide

The electrical double-layer structure at the goethite/supporting electrolyte interface in the absence and presence of PAM

The zeta potential measurements and potentiometric titration provided valuable data about electrokinetic properties of the goethite/supporting electrolyte system in the absence and presence of anionic polyacrylamide.

The solid surface charge density with and without polyacrylamide is presented in Fig. 4. The goethite pH_{pzc} parameter (point of zero charge), i.e. the pH value, at which the concentrations of positive and negative groups on the solid

surface are identical and the goethite total charge is equal to 0, is about 8.2. This value is consistent with the published data (Kosmulski 2011). According to Pauling's rules, goethite surface oxygens are singly, doubly and triply coordinated, whereas all iron atoms are octahedrally coordinated. Thus, at pH 8.2 neutral groups, i.e. $\equiv Fe_2OH$, predominate on the goethite surface. At pH < 8.2, the solid is positively charged because the $\equiv FeOH_2^{1/2+}$ and $\equiv Fe_3OH^{1/2+}$ groups dominate. Then the Stern layer is composed of chloride ions (Cl^-) coming from the supporting electrolyte (NaCl). On the other hand, at pH > 8.2 the goethite particles are negative. The $\equiv FeO^{1/2-}$ and $\equiv Fe_3O^{1/2-}$ groups are dominant on the surface, and the Stern layer is formed by sodium ions (Na^+) of the supporting electrolyte (Tadanier and Eick 2002).

The anionic polyacrylamide addition makes the σ_0 values more negative. The pH_{pzc} point in the PAM presence is about 4.3. This effect is probably induced by the –COO^- groups present in the non-adsorbed polymer segments, but located close to the goethite surface. The polymer macromolecules formed 'tails' and 'loops' structures perpendicular to the solid. Within them, there are the above-mentioned dissociated carboxylic groups responsible for lower values of the solid surface charge.

The zeta potential values for goethite in the absence and presence of polyacrylamide are illustrated in Fig. 5. The goethite pH_{iep} (isoelectric point), i.e. the pH value, at which the quantities of positive and negative moieties in the goethite slipping plane are the same and the slipping plane charge is very close to 0, is about 6.4. This value is consistent with that reported in the literature (Madigan et al. 2009). Thus, at pH 6.4 the charge of the slipping plane is equal to 0. The amounts of positive and negative charges are identical. At pH < pH_{iep}, the slipping plane is positive, whereas at pH > pH_{iep} it is the negative one.

The PAM adsorption makes the electrokinetic potential negative in the whole examined pH range. There is no isoelectric point of the solid in the polymer presence. The decrease results from several effects. At first, the PAM macromolecules adsorbed on the goethite surface shift the slipping plane. The 'tails' and 'loops', formed by PAM on the solid, interact with this structure moving it towards the solution. As a result, the charge distribution in the electrical double layer changes and the zeta potential is more negative. Secondly, as it was mentioned above the adsorbed macromolecules contain the dissociated carboxylic groups. These moieties additionally decrease the electrokinetic potential value. What is more, the PAM macromolecules block some active adsorption sites on the goethite surface which also contributes to the charge distribution change. The mechanism of the solid zeta potential change by the adsorbed polymer at pH 7 is presented in Fig. 6.
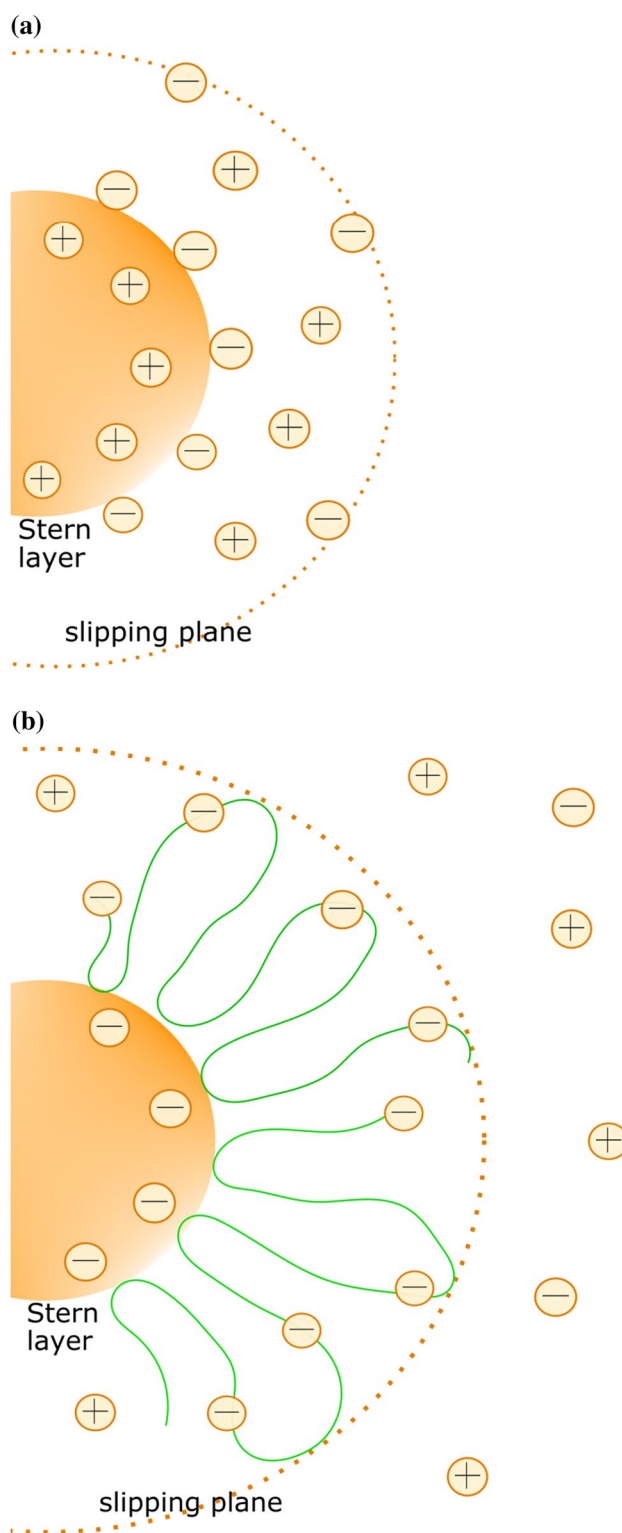

Fig. 6 Electrical double layer of the goethite particles without (**a**) and with (**b**) PAM, at pH 7
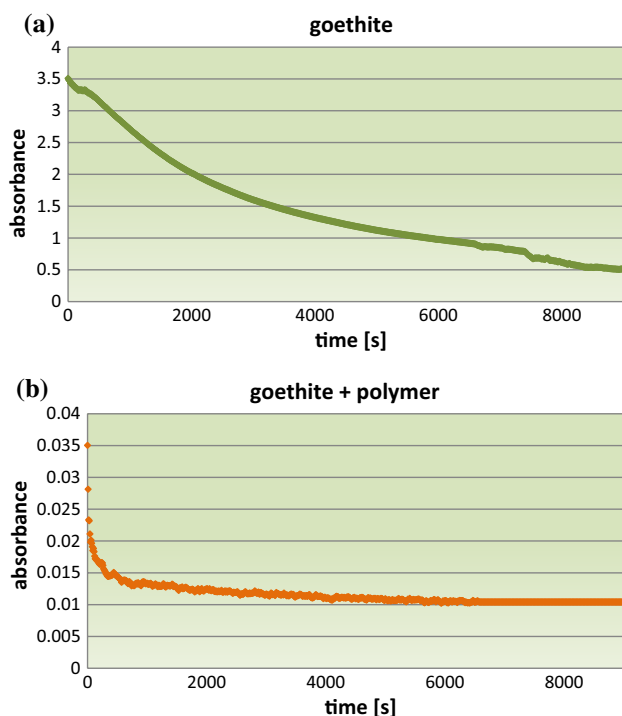
(a)

(b)

Fig. 7 Absorbance changes in time for the goethite suspension in the absence (**a**) and presence (**b**) of polyacrylamide, at pH 5

It is also worth mentioning that a large difference between the pH_{pzc} and pH_{iep} is associated with the goethite porosity. Electrical double layers are formed in pores, but due to small pore sizes the structures overlap each other and, as a result, the pH_{iep} point is shifted. The same phenomenon was observed for mixed oxides (Gun'ko et al. 2004).

The PAM effect on the goethite suspension stability

The goethite suspension stability, with and without polyacrylamide, was measured spectrophotometrically. The absorbance change observed during 2.5 h, at pH 5 is presented in Fig. 7. The absorbance noticed for the systems 30 min after the measurement starts is given in the text below.

The stability of the goethite suspension without polymer strongly depends on the pH value. At pH 5, the system is characterized by high absorbance over time (the absorbance observed was equal to 2.13). This means that the suspension is relatively stable. The solid particles are equally distributed in the system due to electrostatic interactions occurring

between them. Positively charged goethite particles are surrounded by chloride ions (coming from supporting electrolyte) and, as a result, their mutual contact is limited. The described phenomenon is called 'electrostatic stabilization'. At pH 7 and 9, the suspensions are characterized by lower absorbance levels (0.75 and 0.55, respectively). These values are typical for systems of relatively low stability, in which solid particles aggregate and sediment to the vessel bottom. The observed properties are connected with low values of solid zeta potential and surface charge density. At pH 7, the charge of goethite slipping plane is small (-4.6 mV) and, due to this fact, the particles repel each other slightly. In turn at pH 9, the solid surface charge is -1.4 $\mu C/cm^2$, which is also equivalent with very weak repulsive forces between particles. The polyacrylamide addition changes the goethite system stability. The absorbance noticed for the suspensions containing polymer was much lower. It was about 0.01, regardless of the pH value. Thus, it can be stated that anionic polyacrylamide causes the system destabilization.

The PAM adsorption effect on the goethite aggregate size was already visible during the sample preparation. With naked eye, it was noted that the polymer accelerates the mineral aggregation. The particle size measurements confirmed the visual observations. Figure 8 shows the results obtained for the goethite samples with and without polyacrylamide at pH 5. At pH 7 and 9, the results were similar. The result analysis showed that the highest light absorption was noted for the suspension without polymer (Fig. 8a). This system is relatively stable and contains small particles of sizes about 0.62 μm, which do not fall into the vessel bottom. When PAM was added, the absorbance shifted towards larger particles. Moreover, the light absorption level decreased because the amount of small particles had become smaller due to the formation of large, falling aggregates. In the PAM presence (10 ppm), the aggregates of 1.59 μm are formed. What is more, the higher the polymer concentration is, the more and heavier the aggregates are formed (Fig. 8b, c). It is also worth mentioning that the polyacrylamide concentration equal to 100 ppm initiates so strong aggregation that it was not possible to measure the aggregate sizes using a CPS method. The apparatus determines the solid particle and aggregate diameter in the range of 0.005–75 μm. Therefore, the flocs formed in the system must have been larger than the upper limit.

The presented results indicates that anionic polyacrylamide has flocculating ability relative to goethite particles and may be used as a substance improving this solid

Fig. 8 Dependence of light absorption (**a**), particle weight (**b**) and number (**c**) on the goethite particle diameter for the suspensions with and without polyacrylamide, at pH 7
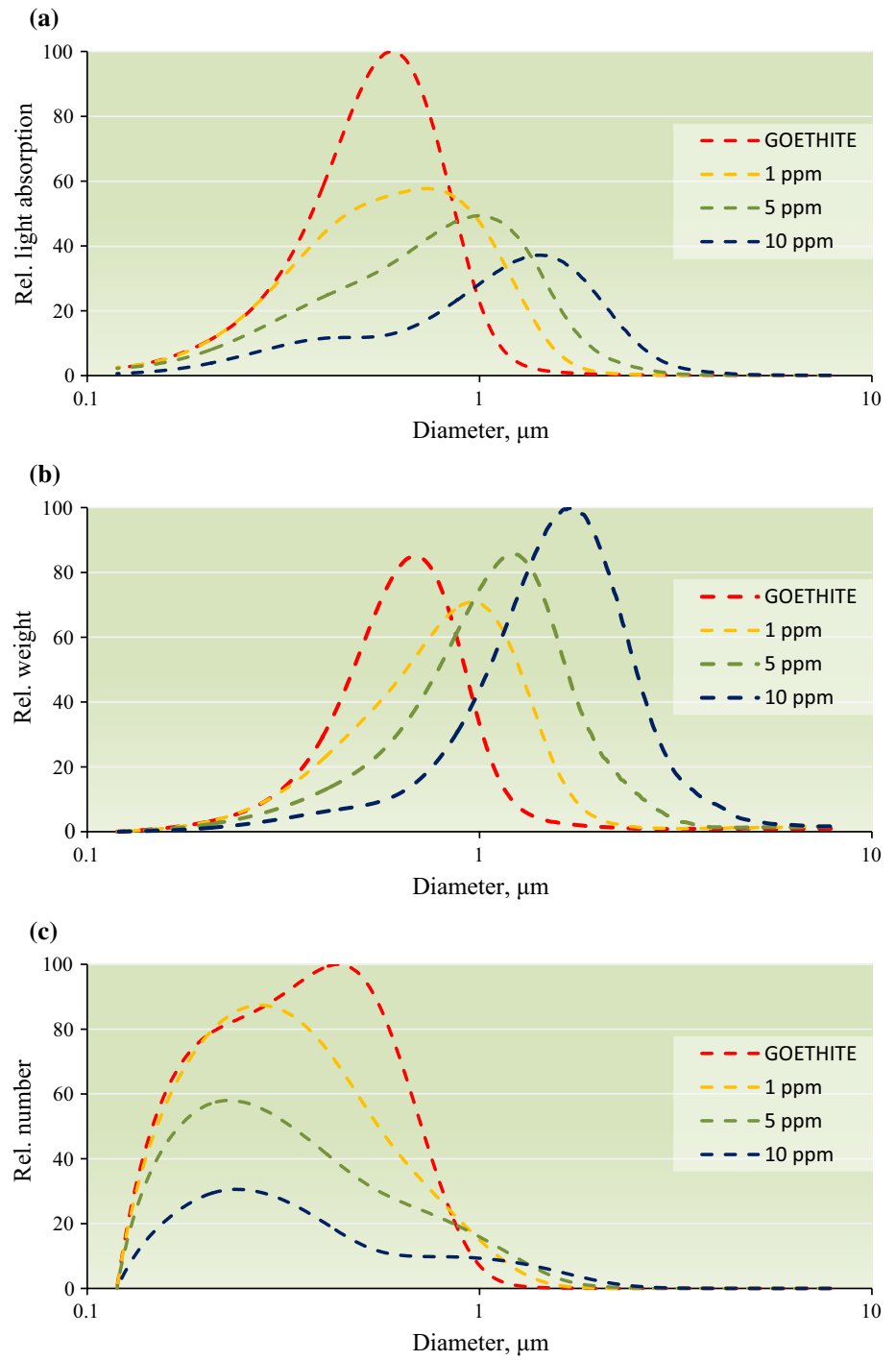
removal from water and wastewaters. The examined suspensions without anionic polyacrylamide are characterized by higher stability at pH 7 and 9 than at pH 5. That may indicate that the pH change towards basic values improves the goethite aggregation and makes its removal easier. However, the destabilization of goethite suspension caused by the polyacrylamide addition is even stronger. At pH 7, the absorbance noted for the system containing the polymer was 75 times smaller compared to the system without PAM, whereas at pH 9 it is 55 times smaller. Thus, the polymer addition is a more effective way to accelerate the goethite removal from aqueous solutions than the change in pH value. Based on the absorbance noted for selected systems, the percentage of goethite removal was calculated. It was very high and equal to: 99.53% at pH 5, 98.67% at pH 7, 98.18% at pH 9. The obtained results confirmed the PAM effectiveness in this process.

The polymer contributes to the goethite suspension destabilization based on two mechanisms. Under the conditions in which the goethite particles are positively charged (i.e. at pH 5), the solid surface charge neutralization by negative polymer macromolecules is highly possible. As a result, the electrostatic forces occurring between the particles are weakened and goethite may aggregate. High adsorption amount observed at pH 5 promotes this mechanism, significantly. On the other hand, at pH 7 and 9, when the goethite surface charge is close to 0, the neutralization by negative PAM macromolecules is not possible. Under these conditions, bridging flocculation phenomenon occurs. A single polyacrylamide chain adsorbs on at least two goethite particles and forms specific bridge between them. This mechanism is highly probable when the particles are not completely covered by the polymer, i.e. when the PAM adsorption amount is low.

Conclusion

PAM adsorbs on the goethite surface in the whole pH range, but the adsorption amount decreases with the pH growth. The PAM adsorption changes the goethite surface charge density. It is more negative due to the impact of –COO$^-$ groups located in the non-adsorbed segments but close to the surface. The goethite zeta potential values are also more negative in the PAM presence which is mainly associated with the slipping plane shift. Regardless of pH value, anionic polyacrylamide accelerates the goethite aggregation which is caused by solid surface charge neutralization (pH 5) or bridging flocculation (pH 7 and 9).

Compliance with ethical standards

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