

Impact of anionic polyacrylamide on stability and surface properties of the Al_2O_3 –polymer solution system at different temperatures

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Abstract The stability mechanism and thermal properties of the system alumina–anionic polyacrylamide (PAM) was studied. The polymer's adsorption properties in dependence on the following parameters solution pH (in the range 3–9), temperature (in the range 15–35 °C), and carboxyl groups' content in the PAM chains (in the range 5–30 %) were examined. The turbidimetry method was applied for determination of the suspension stability of alumina in the presence of PAM. The obtained results indicate that the polymer addition improves significantly Al_2O_3 suspension stability at pH 6 and 9 (in the whole examined temperature range). PAM containing a larger number of carboxyl groups stabilizes solid particles more effectively (due to greater contribution of electrosteric interactions). Moreover, the polymer adsorption on the alumina surface causes changes in the thermal stability of the examined systems. In dependence on temperature, the higher the content of carboxyl groups in the PAM molecules, the greater the total mass loss. This is due to increased adsorption of polyacrylamide whose chains contain numerous –COOH groups.

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Introduction

The adsorption process of different substances (simple ions, small molecules, surfactants, macromolecular compounds) takes place in many industrial and ecological operations. It can occur at various types of interfaces: gas–liquid, gas–solid, liquid–solid, or liquid–liquid. Adsorption in a solid–liquid system differs substantially from this process in a gas–solid system. This results from the fact that in the former case, the solution is adsorbate (i.e., in the simplest system—binary mixture of solvent and solute). The competition between the two components of the solution for active sites on the surface occurs. The adsorption process at gas–solid and liquid–solid interfaces finds widespread applications in the environmental protection. Different kinds of adsorbents are used in order to reduce atmospheric emissions of harmful gases and vapors [1–4], as well as for removal of poisonous chemical compounds dissolved or dispersed in wastewaters [5–12].

The ability of polymers to modify surface properties of solids promoted their use in stabilization or destabilization of colloidal particles. For this reason, they are widely used in many fields of industry, technology, medicine, and ecology [13–17]. Nevertheless, adsorption of the polymer on the solid surface is a very complex process which differs significantly from the adsorption of small molecules [18]. Macromolecules binding with the surface of the adsorbent particles are associated with the decrease in free energy of components in the system. This involves multipoint connections of the polymer functional groups to the solid surface. However, not all polymer segments are directly bound to the solid surface. The

polymer adsorption process is possible when the system exceeds the critical adsorption energy value. In such a case, entropy effects counteract attraction forces between the polymer and the surface.

The polymer adsorption kinetics is determined in the following stages of this process: (a) polymeric chains transport toward the solid surface (depending on the convection and diffusion processes), (b) rate of polymer binding to the adsorbent surface (depending on the height of activation energy barrier), (c) reformation of adsorbed macromolecules which minimizes their free energy, and (d) desorption of shorter polymer chains by longer ones (for polydisperse polymers) [19]. Each of these stages is specific to a given polymer–solid system. Accordingly, the polymer adsorption depends, among others, on the molecular weight of the polymer and its distribution (polydispersity), initial polymer concentration, pH and ionic strength of the solution, temperature, solvent type, as well as mixing degree of the individual components of the system.

The addition of the polymer to a colloidal suspension may result in its stabilization or destabilization [20]. The total coverage of the particle surface by polymer film (usually at a high polymer concentration) results in the steric stabilization. It involves the presence of two effects. The first one—entropic—refers to the reduction of the macromolecule conformational entropy by decrease of the surface area available for polymer segments. The second one—mixing—is associated with the increase of polymeric segment concentration in the area of the mutual penetration of polymer adsorption layers and changes in osmotic pressure. The particles whose surfaces are covered by the adsorbed polymer repel due to the Gibbs energy increase. When the stabilizing polymer is a polyelectrolyte, the electrostatic repulsion between polymeric layers (possessing the same charge) can also occur.

When a small amount of polymer (that does not guarantee complete coverage of the surface) is added to the dispersion, one polymer chain can adsorb onto two or more colloidal particles forming polymer bridges between them. These bridges are formed when the length of loops and tails of the adsorbed macromolecules is greater than the range of electrostatic repulsions between the colloidal particles. As a result, flocks undergo sedimentation and the system destabilization occurs.

Polymers which do not adsorb on the solid surface (high affinity of the polymer for the solvent, complete coverage of the colloidal particles by the surfactant molecules) cause depletion stabilization or flocculation.

The main aim of this study was the determination of the stability properties of anionic polyacrylamide (PAM; with a differing content of carboxyl groups) in the aqueous suspension of aluminum(III) oxide in the temperature range 15–35 °C. Thermogravimetric analysis of the examined systems was also performed to obtain the additional information about

PAM adsorption mechanism. The temperature impact on the conformation of adsorbed macromolecules is significant [21–23] due to modification of interactions between the polymeric chains and the solvent molecules. The ability to influence the structure of polymeric adsorption layer by temperature change is closely related to the suspension stability. Nevertheless, this problem is marginally described in the scientific literature [24–26]. Therefore, the presented studies can supplement incomplete knowledge of this topic.

Materials and methods

Aluminum(III) oxide—Al₂O₃ (Merck)—with the specific surface area 155 m²/g was used as an adsorbent. This metal oxide was washed with doubly distilled water to achieve the supernatant conductivity below 2 mS/cm. The mean particle diameter of the solid was 469 nm (Zetasizer 3000, Malvern Instruments). High surface area, minimal solubility, and high mechanical strength promoted to Al₂O₃ use in the experiments.

Anionic PAM (Korona) was applied as an adsorbate. Polymer samples differed in contents of carboxyl groups (5, 20, and 30 %). These anionic groups remained in macromolecules as a result of incomplete hydrolysis of a number of the amide groups during the PAM preparation. Carboxyl groups underwent dissociation with the increasing pH value and are a source of negative charge of the polyacrylamide chains [27, 28]. The characteristics of the polyacrylamide samples are listed in Table 1. pK_a values of PAM were determined using the potentiometric titration method. Knowing the pK_a value, calculation of dissociation degree (α) of the polymer carboxyl groups was possible. Table 1 presents the values of these parameters. At pH 3, 16.6 % of anionic groups are ionized, whereas at the pH values 6 and 9, the dissociation is practically complete.

All measurements were carried under the following conditions: pH range 3–10, temperature range 15–35 °C, and supporting electrolyte—NaCl with the concentration 0.01 mol/dm³.

Stability measurements of the alumina suspensions (without and with PAM) were carried out with Turbiscan Lab^{Expert} with the cooling module TLab Cooler (France). This apparatus registers light (with the initial wavelength 880 nm) passing through both the examined system and scattered by the solid particles dispersed in the liquid medium. The computer programs (T Lab EXPERT 1.13 and Turbiscan Easy Soft) working with a turbidimeter present the obtained data in the form of transmission and backscattering curves (so-called scans). On the *y* axis, the intensity of transmission (or backscattering) is marked, whereas on the *x* axis, the suspension level in the measurement vial is shown. The suspension was added into the glass vial (70 mm long) to about 40 mm of its height.

Table 1 PAM probe characteristics

Molecular weight/Da	Carboxyl group content/%	Symbol	pKa	α /% pH 3
11,000,000	5	11_5%	3.7	16.6
14,000,000	20	14_20%	3.7	16.6
14,000,000	30	14_30%	3.7	16.6

Changes in the suspension stability were monitored for 15 h, and single scans were collected every 15 min (appropriate colors of scans correspond to particular times of the experiment).

The backscattering data are also used for calculation of the stability coefficient Turbiscan stability index (TSI) according to the equation:

$$TSI = \sqrt{\frac{\sum_{i=1}^n (x_i - x_{BS})^2}{n-1}} \quad (1)$$

where x_i is the average backscattering for each minute of measurement, x_{BS} is the average x_1 , and n is the number of scans.

The suspension with 0.02 g of aluminum(III) oxide in 20 cm³ of NaCl solution was sonicated for 1 min (Sonics, LABO PLUS). Then, the required pH of the solution was adjusted (3, 6, or 9 ± 0.1) using pH Φ 360 pH/Temperature/mV Meter (Beckman). The suspension was shaken in a water bath (OLS 200, Grant) for 30 min, and during this time, its pH was checked. The probes of the alumina suspension containing polyacrylamide were prepared in a similar way. An appropriate volume of the stock PAM solution (with the concentration 1100 ppm), providing its final concentration 100 ppm, was added to the suspension directly before stability test. After that, the system pH was checked and the measuring vial with the suspension was immediately placed in the thermostated chamber of turbidimeter.

Adsorption experiments were made by the static method in the polymer concentration range 5–120 ppm at the pH values 3, 6, and 9 (±0.1) using 0.05 g of Al₂O₃. Such prepared suspensions were shaken in the water bath OLS 200 Grant for 24 h. After that, the solids were centrifuged using a microcentrifuge (type MPW-223e, MPW Med Instruments). The reaction of polyacrylamide with hyamine proposed by Crummet and Hummel [28] was applied. The solution turbidity was measured after 15 min using the ultraviolet-visible (UV-VIS) spectrophotometer (Carry 1000; Varian) at 500 nm. To determine the amount of adsorbed polyacrylamide, the difference between the initial PAM concentration and that after the adsorption process was calculated (using the calibration curve obtained earlier).

The probes for thermal analysis were prepared by adding 0.1 g of Al₂O₃ to 25 cm³ of NaCl or NaCl with PAM ($C_{PAM} = 200$ ppm) solutions. Due to the fact that anionic PAM shows the greatest adsorption on the alumina surface at pH 3, this value of pH was adjusted in the examined suspensions. Then, they were shaken in a water bath for 24 h, and meanwhile, their solution pH was checked. After this time, these probes were centrifuged and the solid (with or without the polymer) was dried.

Thermal analysis was carried out on a STA 449 Jupiter F1, Netzsch (Germany) under the following operational conditions: heating rate of 10 °C/min, a dynamic atmosphere of synthetic air (50 mL/min), temperature range of 30–950 °C, sample mass ~25 mg, and sensor thermocouple type S TG-DSC. As a reference, empty Al₂O₃ crucible was used. The gaseous products emitted during decomposition of the materials were analyzed by QMS 403C Aeölos (Germany) coupled online to the STA instrument. The QMS data were gathered in the range from 10 to 300 amu.

Results and discussion

The calculated values of TSI stability coefficients are listed in Table 2. Their analysis requires knowledge of the fact that TSI assumes values in the range 0–100. Small TSI values indicate high stability of the examined suspension, whereas increase in the TSI value corresponds

Table 2 Stability coefficient TSI for all examined systems, $C_{PAM} = 100$ ppm

Al ₂ O ₃ suspension	Temperature/°C	TSI		
		pH 3	pH 6	pH 9
Without PAM	15	33.65	19.91	54.23
	35	38.75	55.14	63.45
With PAM 11_5%	15	20.93	16.71	17.09
	35	35.14	29.97	47.20
With PAM 14_20%	15	28.91	8.88	9.48
	35	35.1	20.72	12.47
With PAM 14_30%	15	23.12	3.89	8.97
	35	31.57	19.29	18.59

to deterioration of system stability conditions. As can be seen, alumina suspension without the polymer is successively unstable (for both examined temperatures)—TSI values change in the range 33–63 (with the exception of the system at pH 6 and 15 °C). Generally, the anionic polyacrylamide addition improves stability of the dispersed solid particles (at fixed pH and temperature, the decrease of TSI values for the system containing PAM in relation to that without polymer is observed).

To explain stability changes of the alumina particles dispersed in the polyacrylamide solution, the information about polymer adsorption is necessary. These data are discussed in detail in our previous paper [29], and in the present manuscript, a few relevant results are presented in Figs. 1 and 2. Their analysis indicates that adsorption of anionic PAM on the Al_2O_3 surface increases with the rise of temperature (besides PAM 11_5%) and carboxyl group content in macromolecules, whereas its decrease is observed with the increasing pH.

Solution pH affects both dissociation of the PAM carboxyl groups and alumina surface charge. The pH_{pzc} (point of zero charge (pzc)) of Al_2O_3 in the NaCl solution changes in the range 7.46–8.07 (in the temperature range 15–35 °C) [29]. The greatest adsorption of anionic PAM at pH 3 is a result of electrostatic attraction between the positively charged solid surface and the minimally negatively charged PAM macromolecules (which assumes more coiled conformation assuring their dense packing on the solid surface). The formation of hydrogen bonds between the polymer functional groups (both carboxyl and amide) and the solid surface sites is also possible [30]. The total dissociation of polymer anionic groups at pH 6 and 9 leads to development of polymeric chains, which occupy a larger surface area (adsorption decrease at pH 6) or

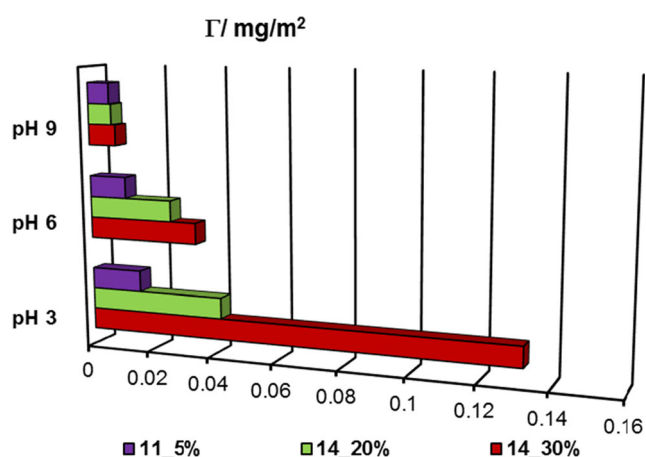


Fig. 1 Adsorbed amounts of PAM on the alumina surface at 15 °C for different solution pH values and anionic group content in polymer chains; $C_{\text{PAM}} = 100$ ppm

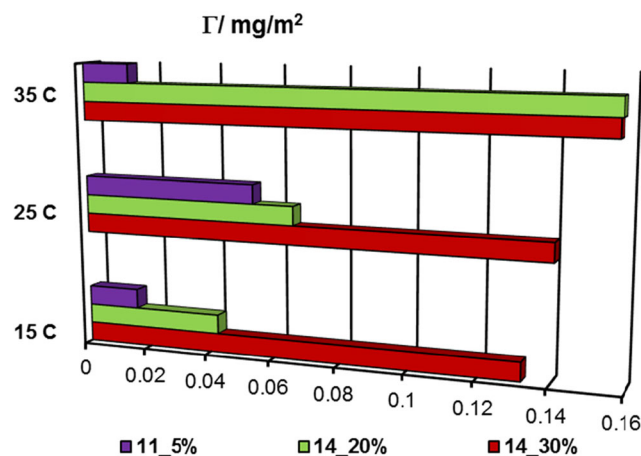


Fig. 2 Adsorbed amounts of PAM on the alumina surface at pH 3 for different temperature values and anionic group content in polymer chains; $C_{\text{PAM}} = 100$ ppm

repulse electrostatically with the negatively charged metal oxide surface (the lowest adsorption level at pH 9).

The temperature increase promotes more extended conformation of polymer chains (increase of hydrodynamic radius of polymer coil) [22]. At a fixed pH value and at higher examined temperature, a thicker adsorption layer, composed of macromolecules expanded perpendicularly to the alumina surface, was formed. As a consequence, the greater number of polymeric molecules can adsorb on the solid surface area unit (adsorption increase).

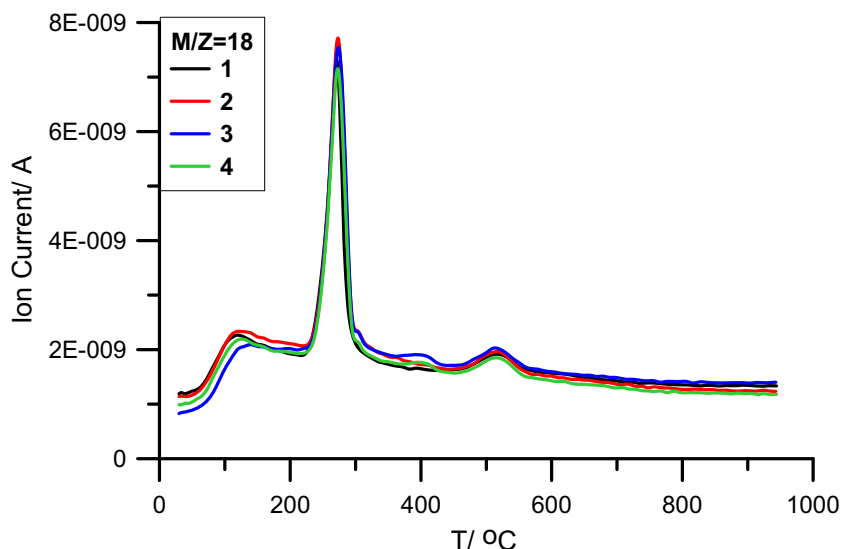
Only in the case of PAM 11.0_5% (whose chains contain the smallest amount of carboxyl groups), the highest adsorption level was observed at 25 °C. Conformation changes of its macromolecules due to temperature increase are not enhanced so effectively by a negatively charged carboxyl groups in polymer chains as is in the case of other polymer samples (with a higher content of anionic groups).

The polyacrylamide-adsorbed amount increase with the rise of carboxyl group content is also observed. More numerous anionic groups cause more expanded conformation of polymeric chains. Such structure of adsorbing macromolecules favors their higher adsorption in the alumina–polymer solution system.

Based on the turbidimetric and adsorption results, a more probable mechanism of suspension stability in the polymer presence can be proposed.

At pH 3, there is a slight influence of PAM adsorption on the Al_2O_3 suspension stability (for both examined temperatures). It is probably caused by the greatest polymer adsorption and formation of a densely packed polyacrylamide layer on the colloidal particle surfaces. This results in the appearance of steric repulsion between the solid particles which leads to slight improvement of the solid suspension stability in the PAM presence.

Fig. 3 MS profile of H₂O vs temperature for systems: Al₂O₃ (1), Al₂O₃/PAM 11_5% (2), Al₂O₃/PAM 14_20% (3), and Al₂O₃/PAM 14_30% (4)



Improvement of stability of the alumina suspension in the presence of polyacrylamide is more pronounced at two other pH values. TSI coefficient for the samples containing PAM assumes significantly lower values (compared to the oxide suspension without the polymer). The greater the improvement of suspension stability is, the higher content of carboxyl groups in the polyacrylamide chains is found.

At pH 6, the adsorbed macromolecules assume a more extended conformation. Despite less adsorption of the polymer (in comparison to that at pH 3), the alumina suspension stability increases due to the rise of repulsive interactions between the solid particles covered with polymeric layers. The mechanism of stabilization is electrosteric.

A similar effect occurs in the systems at pH 9 at which the adsorption of PAM is significantly lower (electrosteric forces between alumina particles). Because of low adsorption of the polymer, some depletion interactions (caused by unadsorbed PAM chains) can be of importance.

For detailed analysis of processes taking place during the thermal degradation of the examined samples, measurements of the presence of gaseous products by mass spectrometry were performed. The intensity profiles of main decomposition products (H₂O, CO₂) are presented in Figs. 3 and 4. The analysis of $m/z = 18$ (characteristic of H₂O) indicates small changes obtained for the alumina samples modified by PAM in relation to the solid sample without the polymer. This demonstrates that the binding

Fig. 4 MS profile of CO₂ vs temperature for systems: Al₂O₃ (1), Al₂O₃/PAM 11_5% (2), Al₂O₃/PAM 14_20% (3), and Al₂O₃/PAM 14_30% (4)

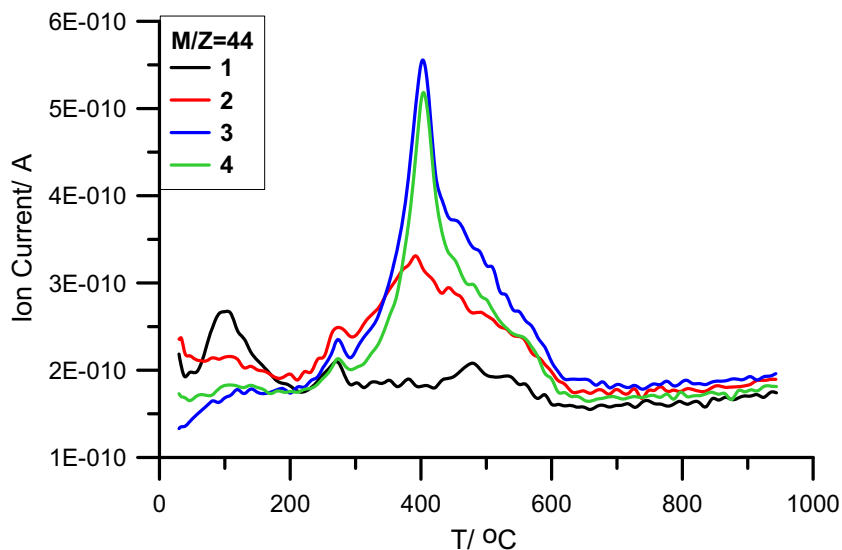


Table 3 The heat values of main stages of thermal degradation of samples determined by DSC analysis

Sample	ΔH_I /J/g	ΔH_{II} /J/g	ΔH_{III} /J/g
Al ₂ O ₃	16.37	64.15	–
Al ₂ O ₃ /PAM 11_5%	18.97	62.42	–1.12
Al ₂ O ₃ /PAM 14_20%	18.79	63.04	–18.9
Al ₂ O ₃ /PAM 14_30%	16.32	61.68	–16.2

process of polymer with solid is of the surface character. This behavior is confirmed by slight differences in enthalpy values calculated from the peak profiles on the DSC curves (Fig. 10, Supplementary Information section)—for desorption of physically (ΔH_I) and chemically (ΔH_{II}) bound water (Table 3). Only in the temperature range 350–450 °C, the appearance of additional peak is observed [31–37]. The presence of this peak is associated with the organic hydrogen oxidation.

In the case of $m/z = 44$ (characteristic of CO₂), the appearance of distinct peaks in the temperature range 250–600 °C with the maximum at about 400 °C was observed. This is an evidence of organic substance decomposition. The obtained effects depend mainly on the adsorbate molecule structure and its adsorbed amount (changes in ΔH_{III} values).

The influence of temperature, at which alumina surface modification with anionic PAM was performed, on the thermal characteristics of examined systems is shown in Fig. 5. It represents the TG and derivative thermogravimetric (DTG) curves obtained for alumina with adsorbed PAM 14_30% at 15 and 35 °C. The analysis of these curves indicates that the total mass change is greater in the case of the sample prepared at 35 °C. This is a result of greater adsorption level of PAM obtained at the highest examined temperature.

Conclusions

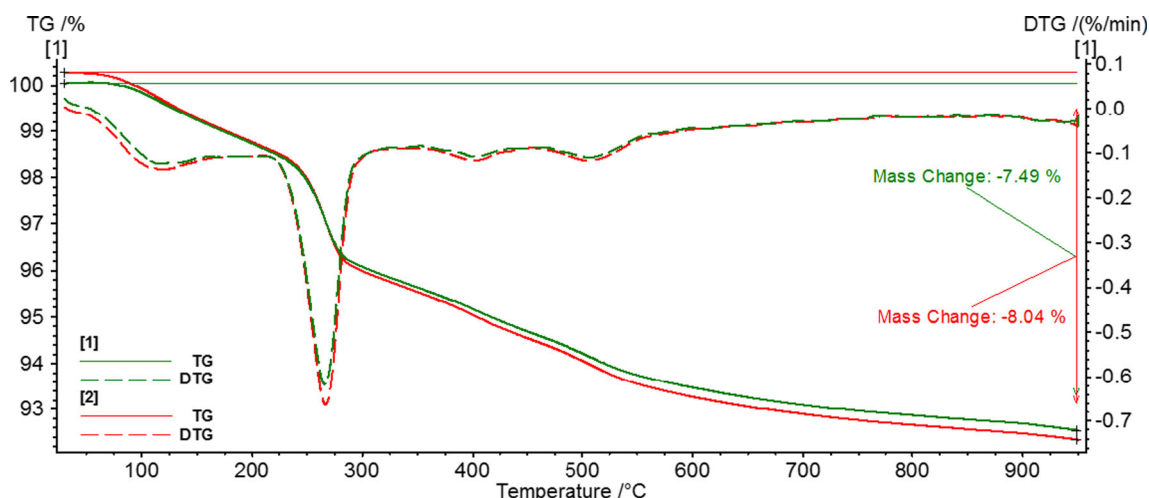
Adsorption of anionic PAM on the surface of aluminum(III) oxide decreases with the rising pH. At pH 3, there is a small dissociation of PAM carboxyl groups, and therefore, the adsorption proceeds by hydrogen bond formation and slight electrostatic attraction. At pH 3, there is a slight influence of PAM adsorption on the Al₂O₃ suspension stability (for both examined temperatures).

Stronger electrostatic attraction between the negatively charged macromolecules and the positively charged Al₂O₃ surface is mainly responsible for the polymer adsorption at pH 6. Despite less adsorption of the polymer (in comparison to that at pH 3), the alumina suspension stability increases due to the rise of repulsive interactions between the solid particles covered with polymeric layers (electrosteric stabilization).

The smallest polymer adsorption at pH 9 is a result of repulsion between the PAM chains and Al₂O₃ particles (both negatively charged). Because of low adsorption of the polymer, some importance of suspension stability improvement can have depletion interactions (caused by unadsorbed PAM chains).

The higher the content of anionic carboxyl groups is in macromolecules and temperature, the larger the amount of adsorbed polyelectrolyte is. This is due to the conformational changes of macromolecules manifested by adopting a more developed structure in the adsorption layer.

The adsorption of anionic PAM causes noticeable decrease in thermal stability of the alumina. The greater the total mass losses are, the higher the polymer adsorption is (i.e., the higher is the temperature of this process and is carboxyl groups' content in the PAM chains). For the alumina samples with the polymer, the additional minimum on the DTG curves appears at about 402 °C (thermo-oxidation of the organic H atoms and carbonized polymer residue).

**Fig. 5** TG and DTG curves for alumina systems modified by PAM 14_30% at pH 3: adsorbed at 15 °C (1) and adsorbed at 35 °C (2)

Compliance with ethical standards

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Conflict of interest The authors declare that they have no conflict of interest.

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