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# Optimization of Flocculation Conditions for Kaolin Suspensions Using [(3-methacryloylamino)propyl]trimethylammonium Chloride and Dimethylacrylamide Copolymer

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## ABSTRACT

The flocculating effect of [(3-methacryloylamino)propyl]trimethylammonium chloride-dimethylacrylamide (TMAPMACh-DMAA) copolymer samples at various molar ratios on a kaolin suspension was studied. Varying the flocculant concentration from 0.01 g/L to 0.24 g/L revealed that 0.04 g/L was the most effective; at concentrations below 0.04 g/L, the copolymer exerted a stabilizing effect on the kaolin suspension. A study of the effect of pH on flocculation revealed that the maximum degree of kaolin suspension clarification was achieved in the pH range of 5.0-7.0. This is due to the neutralizing effect of the ammonium groups of [(3-methacryloylamino)propyl] trimethylammonium chloride on the $\equiv SiO^-$ groups of kaolin particles. Electrophoresis data demonstrated that although kaolin particles possess a negative charge in the pH range from 3.0 to 9.0, the negative ζ-potential values decrease in an acidic medium. Copolymer samples with TMAPMACh:DMAA molar ratios of 50:50 and 60:40 are optimal for flocculation, providing a sufficient number of cationic groups to neutralize the negative charge of kaolin particles. A relationship between the kaolin particle ζ-potential and flocculation efficiency is demonstrated. The role of neutralization and «bridging» flocculation mechanisms in the sedimentation of kaolin particles using the TMAPMACh-DMAA copolymer is substantiated.

## 1. Introduction

Treatment of industrial wastewater remains one of the most pressing environmental challenges due to the increasing discharge of toxic and persistent pollutants into natural ecosystems. Effective purification of wastewater not only reduces the negative impact on flora and fauna but also enables the implementation of water recycling systems, thereby conserving freshwater resources used for drinking and agricultural purposes [1-3]. In this context, flocculation-based methods are widely applied due to their simplicity, cost-effectiveness, and high efficiency in removing suspended particles from aqueous systems [4, 5].

In recent years, considerable attention has been devoted to the development of polymer-based flocculants, which have largely replaced conventional inorganic coagulants due to their superior performance and tunable properties [6, 7]. Among them, polyacrylamide and its derivatives are the most widely used, owing to their high molecular weight and ability to form extended macromolecular structures in solution [3, 8]. The efficiency of polymeric flocculants is strongly influenced by their chemical composition, charge density, and molecular conformation, which determine their interaction with dispersed particles [9, 10].

Kaolin suspensions are commonly employed as model systems for investigating flocculation processes due to their well-defined surface chemistry and predominantly negative surface charge [11, 12]. Numerous studies have shown that flocculation of kaolin particles is governed by a combination of charge neutralization and polymer bridging mechanisms, as well as adsorption interactions between polymer macromolecules and particle surfaces [5, 13-15]. However, despite significant advances in understanding these processes, the relative contribution of electrostatic and structural factors to flocculation efficiency remains a subject of ongoing discussion [8, 10].

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In particular, the influence of composition of the flocculants on the balance between charge neutralization and bridging mechanisms has not been sufficiently clarified. Moreover, the relationship between the ζ-potential of dispersed particles and the efficiency of flocculation under varying physicochemical conditions requires further investigation [10, 15]. A systematic analysis of these factors is essential for optimizing flocculation processes and improving the performance of polymeric flocculants. It should also be noted that the formation of large clay flocs is not always desirable, as they can affect the viscosity of the treated liquid. For example, the formation of kaolinite polymer flocs during oil production hinders oil displacement [16]. There are known cases of flocculation involving the preliminary adsorption of metal ions contained in the water being treated onto the surface of bentonite, followed by its aggregation with acrylamide derivatives [17]. To enhance the flocculating properties of natural polymers-citrus pectin and chitosan-their magnetic composites have been synthesized [18], and known industrial flocculants have been used in mixtures with $Ca^{2+}$ and $Mg^{2+}$ salts [19]. In this regard, when studying the aggregation of dispersed particles by polymer flocculants, it is necessary to account for the influence of electrostatic interactions between the polymer chains and the particles, as well as the possibility of co-aggregation of the formed flocs due to the adhesion of flocculant macromolecules adsorbed onto the particles.

The aim of this study is to optimize the flocculation conditions of kaolin suspensions using a copolymer of [(3-methacryloylamino)propyl]trimethylammonium chloride and dimethylacrylamide (TMAPMACh-DMAA), and to elucidate the relationship between copolymer composition, suspension pH, ζ-potential, and flocculation efficiency. This study provides new insights into the combined role of charge neutralization and bridging mechanisms in the aggregation of clay particles and will facilitate the development of more efficient flocculation systems for industrial wastewater treatment.

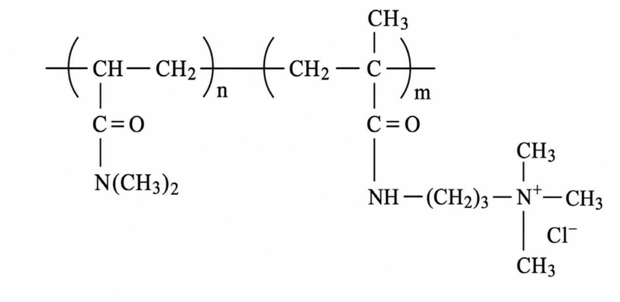
## 2. Experimental section

### 2.1. Materials

#### 2.1.1. Flocculant

The copolymer of [(3-methacryloylamino)propyl] trimethylammonium chloride and dimethylacrylamide (TMAPMACh-DMAA) was obtained by radical copolymerization of [(3-methacryloylamino)propyl] trimethylammonium chloride (TMAPMACh) with N,N-dimethylacrylamide (DMAA) at a temperature of 333 K in the presence of ammonium persulfate as an initiator.

The structural formula of the TMAPMACh-DMAA copolymer is presented below:




The synthesis conditions and methods used for copolymer identification are described in detail in [20].

#### 2.1.2. Kaolin

Kaolin samples from the Alekseevskoe deposit (Akmola region) were used for the preparation of clay suspensions. The mineral composition of the kaolin is as follows (wt.%): quartz – 67.3, kaolinite – 31.4, and muscovite – 1.1 [21]. The clay was preliminarily ground and sieved through a sieve with a pore size of 10 µm. Then, 0.5% kaolin suspensions were prepared by dispersing the powdered clay in deionized water under continuous magnetic stirring for 60 min to ensure homogeneous particle distribution.

### 2.2. Research Methods

#### 2.2.1. Evaluation of the Flocculating Action of the TMAPMACh-DMAA Copolymer

The efficiency of the flocculating action of the TMAPMACh-DMAA copolymer was evaluated by measuring the change in the optical density of kaolin suspensions obtained in the absence and in the presence of the flocculant. Copolymer samples with different molar ratios of TMAPMACh:DMAA were used as flocculants. The concentration of the flocculant introduced into the kaolin suspensions ranged from 0.01 to 0.24 g/L.

The optical density of the kaolin suspensions was measured using a UV-7504 spectrophotometer at a wavelength of 540 nm with a measurement accuracy of ±2%. All experiments were performed at least in triplicate, and the obtained results were averaged. The flocculation process was monitored for 60 min at 5-min intervals. For comparison, the optical density values of the kaolin suspensions obtained in the absence and in the presence of the flocculant were analyzed 60 min after the treatment of kaolin with the flocculant. The pH of the suspensions was adjusted using 0.01 mol/L HCl and NaOH solutions within the range of 3.0-9.0. The pH values were set prior to sedimentation and were not additionally maintained during the 60 min settling period.

#### *2.2.2. Determination of the Charge of Kaolin Particles*

The electrokinetic potential of kaolin particles before and after flocculation was determined by the light scattering method using a Malvern ZetaSizer NanoZS (Malvern, UK) instrument according to the procedure described in [22]. The ζ-potential measurements were performed in deionized water under varying pH conditions without additional control of ionic strength. This is due to the need to avoid the influence of electrolyte ions, introduced to regulate the ionic strength of the medium, on the surface charge of the clay particles.

## 3. Results and Discussion

### *3.1. Effect of suspension pH on flocculation efficiency*

One of the important factors affecting the efficiency of water purification is the pH of the medium. Therefore, the flocculation activity of the TMAPMACh-DMAA copolymer was investigated at different pH values.

Fig. 1 shows the sedimentation curves of kaolin particles treated with solutions of the TMAPMACh-DMAA copolymer at different concentrations as a function of the pH of the suspension. It should be noted that constant ionic strength was not maintained in the kaolin suspensions. This is due to the complex composition of the suspension, which depends on the ionized groups of the clay mineral, the concentration of $H^+$ and $OH^-$ groups of the introduced acid and alkali, and the concentration of the flocculant, which contains a quaternary ammonium salt with counterions in the form of Cl- ions. Taking into account the low concentration of HCl, NaOH and TMAPMACh-DMAA flocculant used to regulate the pH values of the medium, it can be assumed that the change in strength during the study will be insignificant and, accordingly, its effect on flocculation will also not be so significant. The flocculant concentrations in kaolin suspensions are 0.01-0.24 g/L or $3.0 \cdot 10^{-5} – 7.2 \cdot 10^{-4}$ base-mol/L.

The most pronounced decrease in optical density is observed at copolymer concentrations of 0.04-0.08 g/L. The pH profiles of these curves indicate that the most intensive sedimentation of clay particles occurs in the pH range of 5-7, whereas under more acidic or alkaline conditions the suspensions become more stable. This behavior is associated with the sensitivity of the surface charge of clay particles to changes in the pH of the suspension [8].

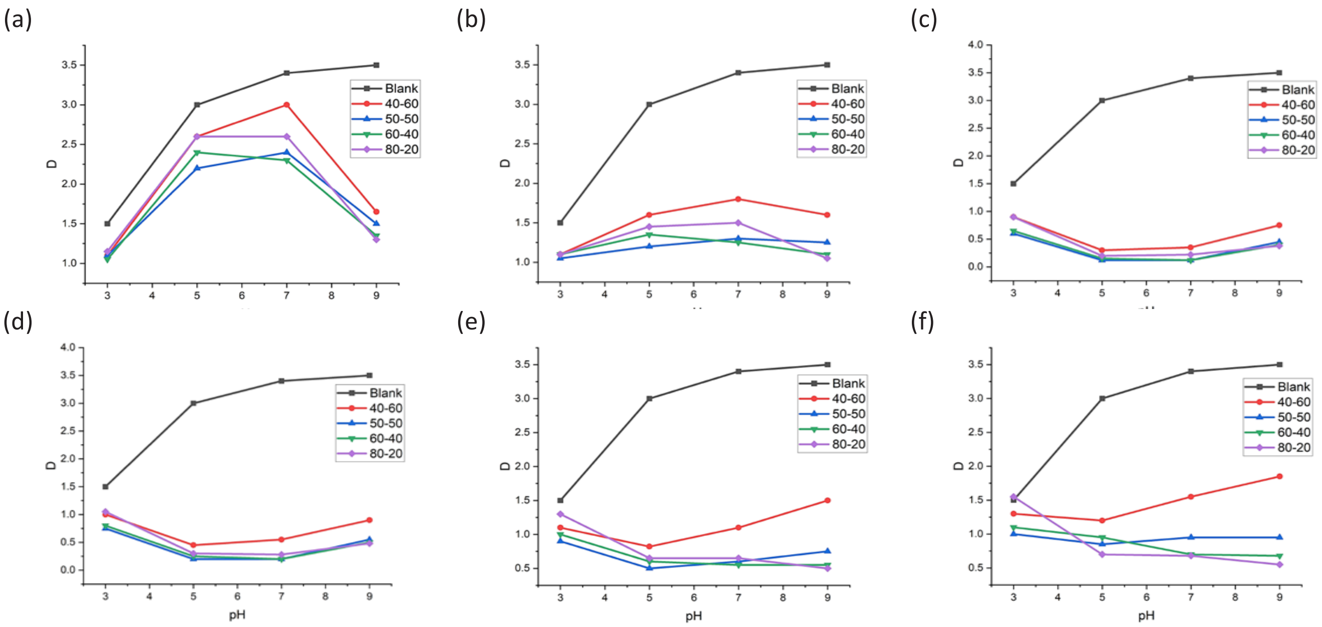



**Fig. 1.** Dependence of the optical density of 0.5% kaolin suspensions on the pH of the suspension in the absence of flocculant and in the presence of TMAPMACh-DMAA copolymers with different molar ratios (TMAPMACh:DMAA = 80:20, 60:40, 50:50, 40:60). The flocculant concentration was (a) 0.01 g/L, (b) 0.02 g/L, (c) 0.04 g/L, (d) 0.08 g/L, (e) 0.16 g/L, and (f) 0.24 g/L.

As can be seen from Fig. 2, in an acidic medium the kaolin particles possess a relatively low negative charge, which increases with increasing pH. At pH < 5, protonation of a portion of the silanol groups present in the clay structure may occur, leading to a decrease in their negative charge:

$$\equiv SiOH + n\ H^+ \rightarrow \equiv SiOH_2^+$$

In neutral and alkaline media, the abundance of $OH^-$ groups leads to the dissociation of ≡SiOH groups on the surface of kaolin particles:

$$\equiv SiOH + n\ OH^- \rightarrow \equiv SiO^- + n\ H_2O$$

Subsequently, the efficiency of flocculation will depend on the interaction of the flocculant with the silanol groups on the surface of the clay particles [23-25].

Similar pH-dependent flocculation behavior has been reported for kaolinite dispersions treated with cationic polymeric flocculants [5, 26]. Sun et al. [5] emphasized that electrostatic interactions between negatively charged clay surfaces and cationic functional groups of polymer chains play a dominant role in the initial stage of aggregation. Deng et al. [26] also demonstrated that the conformational flexibility and molecular characteristics of polymeric flocculants significantly affect the aggregation efficiency of kaolinite particles. The results obtained in the present work are therefore in good agreement with recent studies on clay-polymer flocculation systems.

The stabilization of suspensions at pH < 5 may be attributed to the mutual electrostatic repulsion between positively charged $\equiv SiOH_2^+$ groups of kaolin and [(3-methacryloylamino)propyl] trimethylammonium chloride groups within the macromolecules of the flocculant adsorbed on the surface of clay particles [9].

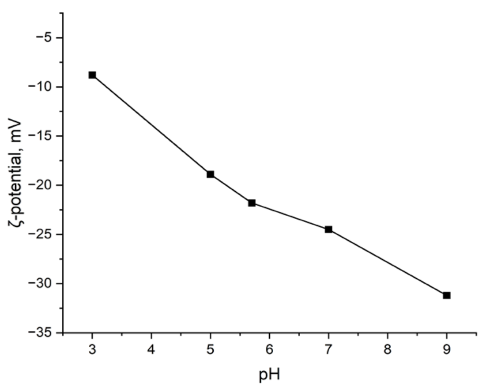



**Fig. 2.** Effect of the pH of a 0.5% kaolin suspension on the ζ-potential of kaolin particles.

The high flocculation efficiency in the pH > 5 region can be explained by electrostatic attraction between oppositely charged $\equiv SiO^-$ groups of the clay and the ammonium groups of the TMAPMACh-DMAA copolymer. At pH > 7, stabilization of the suspensions is again observed, which is likely associated with electrostatic repulsion between the $\equiv SiO^-$ groups of the clay and the ionized groups of DMAA.

### *3.2. Effect of the Concentration and Composition of the TMAPMACh-DMAA Copolymer on Flocculation*

Analysis of the curves describing the dependence of the optical density of kaolin suspensions on the pH of the suspension obtained at different flocculant concentrations shows that their general profile corresponds either to increasing curves in the pH range of 3.0-5.0 followed by a decrease in D values as pH increases from 7.0 to 9.0, or to decreasing curves followed by a subsequent increase in the turbidity of the system.

At TMAPMACh-MAA concentrations of 0.01 g/L and 0.02 g/L, the optical density reaches maximum values in the pH range of 5.0-7.0. This indicates that both acidification and alkalization of the suspension medium reduce the flocculation efficiency. Such behavior of the system at pH > 7.0 may be explained with the enrichment of the surface of kaolin particles with functional groups of the DMAA units of the copolymer ($-CONH_2$), which in the presence of increasing amounts of $OH^-$ groups in aqueous medium may be converted into $-COOH$ groups. The dissociation of carboxyl groups enhances the electrostatic repulsion between kaolin particles, thereby preventing their aggregation and leading to stabilization of the system.

At copolymer concentrations below 0.04 g/L, incomplete surface coverage of kaolin particles by copolymer macromolecules may lead to insufficient charge neutralization and preservation of relatively high negative absolute ζ-potential values. Under these conditions, electrostatic repulsion between particles remains significant, resulting in stabilization of the suspension. In addition, adsorbed polymer fragments may form hydrated surface layers that contribute to steric stabilization of the dispersion. Therefore, the reduced flocculation efficiency at low copolymer concentrations can be attributed to the combined effect of electrostatic and steric stabilization mechanisms.

When the concentration of the copolymer introduced into the suspension increases to 0.04-0.24 g/L, the D = f(pH) curves in the pH range of

3.0-7.0 exhibit a decreasing trend. However, the D values tend to increase again as pH rises from 7.0 to 9.0, which is particularly noticeable for copolymer samples with the highest DMAA content, i.e., with a TMAPMACh:DMAA molar ratio of 40:60.

As noted above, the most pronounced flocculating effect is observed at copolymer concentrations of 0.04 g/L and 0.08 g/L, which is associated with an increase in the number of introduced alkylammonium groups of the copolymer and a higher number of $\equiv SiO^-$ groups on the surface of clay particles. However, an increase in the concentration of $OH^-$ groups at pH > 7 leads to a higher degree of ionization of acrylamide groups in the copolymer structure, which results in a stabilizing effect on the suspension. This is confirmed by the fact that the strongest stabilizing effect on the system is observed for the copolymer sample with the highest content of dimethylacrylamide.

As for the composition of the flocculant, for copolymer samples with TMAPMACh:DMAA molar ratios of 50:50 and 60:40, a decrease in D values down to 0.11 is achieved at a concentration of 0.04 g/L, indicating effective flocculation. Micrographs of the suspensions obtained after treatment with the flocculant solution show the formation of large aggregates of clay particles with water layers between them (Fig. 3b, c). In the absence of the flocculant, kaolin particles are distributed uniformly and do not exhibit a tendency to aggregate (Fig. 3a).

Thus, the most pronounced flocculating effect on the kaolin suspension is exhibited by TMAPMACh-DMAA copolymer samples with TMAPMACh:DMAA molar ratios of 50:50 and 60:40 at a concentration of 0.04 g/L.

### *3.3. Effect of the Ratio of TMAPMACh and DMAA Monomer Units on Flocculation*

Analysis of the relationship between optical density and ζ-potential values revealed a clear correlation between the surface charge of kaolin particles and flocculation efficiency. The minimum values of residual optical density are achieved when the ζ-potential is close to zero, whereas at high negative or positive ζ-potential values an increase in optical density is observed, indicating insufficient aggregation or stabilization of the dispersions (Fig. 4).

The high flocculation efficiency observed for the copolymer with TMAPMACh-DMAA molar ratios of 50:50 and 60:40 is attributed to the optimal ratio of its monomer units, which results in an appropriate balance of cationic and anionic groups in the system.

[(3-methacryloylamino)propyl]trimethylammonium chloride contains a quaternary ammonium group and serves as a source of a permanent positive charge that does not depend on the pH of the suspension [24]. The main function of TMAPMACh is electrostatic interaction with the negatively charged surface of kaolin particles and the reduction of the ζ-potential, which leads to a decrease in electrostatic repulsion between similarly charged clay particles. At the same time, an excess of such groups results in excessive charge neutralization and inversion of the surface charge sign [25].

The presence of DMAA units in the macromolecule provides chain flexibility and increases the effective radius of the macromolecular coil. This promotes the formation of extended free chain segments capable of forming so-called «bridges» through hydrophobic interactions of the «loops» and «tails» of copolymer macromolecules adsorbed on the surface of clay particles [26].

At a TMAPMACh:DMAA copolymer composition of 50:50, the content of cationic groups is sufficient to bring particles closer together through partial charge neutralization, while the significant fraction of DMAA prevents complete adsorption of the macromolecule on the particle surface and ensures effective bridge formation between particles. As a result, a synergistic action of the charge neutralization and «bridging» mechanisms of clay particle aggregation is realized,

(a) (b) (c)

**Fig. 3.** Micrographs of kaolin suspensions obtained in the absence (a) and in the presence of a 0.04% solution of the TMAPMACh-DMAA copolymer with TMAPMACh:DMAA molar ratios of 60:40 (b) and 50:50 (c). Scale bar = 100 µm.

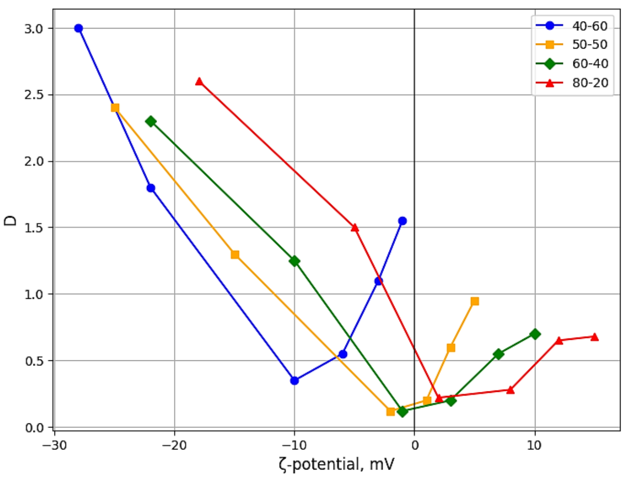



**Fig. 4.** Dependence of the optical density of 0.5% kaolin suspensions treated with flocculant solutions containing different TMAPMACh-DMAA ratios on the ζ-potential of the particles. T = 60 min.

leading to maximum flocculation efficiency [27]. When the composition shifts toward a lower TMAPMACh content (TMAPMACh:DMAA = 40:60), flocculation becomes limited due to the persistence of a high negative ζ-potential. Conversely, with an increased content of cationic units (TMAPMACh:DMAA = 80:20), the contribution of the charge neutralization mechanism increases, which may lead to stabilization of the system due to overcharging of the dispersed particles, thereby narrowing the range of effective flocculation.

The obtained results indicate that efficient flocculation is achieved only under conditions where partial charge neutralization is accompanied by sufficient extension of polymer chains capable of forming interparticle bridges. Excessive charge compensation or insufficient polymer adsorption may result in restabilization of the suspension due to electrostatic or steric effects. Similar behavior has been reported for other cationic polymer–clay systems, where the balance between electrostatic interactions and polymer conformation determines the aggregation efficiency [5, 16, 25, 26].

Thus, flocculation of kaolin suspensions in the presence of TMAPMACh-DMAA copolymers is determined not only by the concentration of the flocculant but also by its composition and the pH of the medium, which control the ζ-potential of the system, the conformation of macromolecules, and the balance between charge neutralization and bridging mechanisms. In terms of its influence on flocculation efficiency, the process of charge neutralization of aggregating kaolin particles is comparable to the stage of «bridge» formation between copolymer macromolecules adsorbed on the surface of clay particles.

## 4. Conclusion

This study systematically investigated the flocculation behavior of kaolin suspensions in the presence of TMAPMACh-DMAA copolymers, with particular attention to the effects of copolymer composition, concentration, and suspension pH. The results demonstrate that flocculation efficiency is governed by the interplay between electrostatic interactions and macromolecular conformation.

Optimal flocculation conditions were identified at copolymer concentrations of 0.04-0.08 g/L and within a pH range of 5-7, where a significant reduction in optical density was achieved. Copolymers with TMAPMACh:DMAA molar ratios of 50:50 and 60:40 exhibited the highest performance, indicating that an optimal balance between cationic and nonionic units is critical for effective particle aggregation.

A clear correlation between the ζ-potential of kaolin particles and flocculation efficiency was established. Minimum residual turbidity was observed at ζ-potential values close to zero, confirming the dominant role of charge neutralization in destabilizing the suspension, while polymer bridging contributes to the formation of stable aggregates.

This study provides new insights into the combined influence of copolymer structure and physicochemical conditions on flocculation mechanisms. It is shown that neither charge neutralization nor bridging alone is sufficient to achieve maximum efficiency; instead, their synergistic action determines the overall performance of the system.

From a practical standpoint, the identified optimal conditions and copolymer compositions can be used in the design of efficient flocculants for industrial wastewater treatment, particularly for systems containing fine clay particles.

Future work should focus on extending the proposed approach to real multicomponent wastewater systems, as well as on evaluating the influence of ionic strength and competing ions on flocculation performance.

## Author Contributions

**Mirzhan Tursynbetov:** Writing – original draft preparation, experimental investigation and data analysis; **Sagdat Tazhibayeva:** Conceptualization of the study and scientific supervision; **Kaldibek**

**Abdiyev:** Conceptualization of the study and data analysis; **Baltabay Sadykov:** Methodology and investigation; **Kuanyshbek Musabekov:** Project administration and final approval of the version to be published.

## Conflict of interest

The authors declare no conflicts of interest.

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## Оптимизация условий флокуляции суспензий каолина с помощью сополимера [(3-метакрилоиламино)пропил]триметиламмония хлорида и диметилакриламида

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### АННОТАЦИЯ

Изучено флокулирующее действие образцов сополимера [(3-метакрилоиламино)пропил] триметиламмония хлорида и диметилакриламида (ТМАПМАХ-ДМАА) при их различных мольных соотношениях на суспензию каолина. Изменение концентрации флокулянта от 0,01 г/л до 0,24 г/л показало, что наиболее эффективной является концентрация 0,04 г/л, при концентрации менее 0,04 г/л сополимер оказывает стабилизирующее действие на суспензию каолина. При исследовании влияния pH среды на флокуляцию установлено, что максимальная степень осветления суспензии каолина достигается в интервале pH 5,0-7,0. Это обосновано нейтрализующим действием аммониевых групп [(3-метакрилоиламино)пропил] триметиламмония хлорида на $\equiv SiO^-$ группы частиц каолина. Методом электрофореза показано, что хотя частицы каолина и имеют отрицательный заряд в интервале pH от 3,0 до 9,0, в кислой среде значения отрицательного ζ-потенциала снижаются. Оптимальными для флокуляции являются образцы сополимера с мольным соотношением ТМАПМАХ:ДМАА 50:50 и 60:40, обеспечивающие наличие достаточного количества катионных групп для нейтрализации отрицательного заряда частиц каолина. Показана взаимосвязь между значением ζ-потенциала частиц каолина и эффективностью флокуляции. Обоснована роль нейтрализационного и «мостичного» механизмов флокуляции при осаждении частиц каолина с помощью сополимера ТМАПМАХ-ДМАА.

**Ключевые слова:** флокуляция, полимерные флокулянты, суспензия каолина, очистка воды, дзета-потенциал, нейтрализация заряда, мостичный механизм

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## [(3-метакрилоиламино)пропил]үшметиламмоний хлориді және диметилакриламид сополимерінің қатысында каолин суспензияларының флокуляция жағдайларын оңтайландыру

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### АҢДАТПА

[(3-метакрилоиламино)пропил]үшметиламмоний хлориді-диметилакриламид (ТМАПМАХ-ДМАА) сополимері үлгілерінің каолин суспензиясына флокуляциялық әсері әртүрлі мольдік қатынастарда зерттелді. Флокулянт концентрациясын 0,01 г/л-ден 0,24 г/л-ге дейін өзгерту 0,04 г/л ең тиімді екенін көрсетті; 0,04 г/л-ден төмен концентрацияларда сополимер каолин суспензиясына тұрақтандырғыш ретінде әсер етті. pH-тың флокуляцияға әсерін зерттеу каолин суспензиясының мөлдірленуінің максималды дәрежесіне 5,0-7,0 pH диапазонында жетуге болатындығын көрсетті. Бұл ТМАПМАХ аммоний топтарының каолин бөлшектерінің $\equiv SiO^-$ топтарына бейтараптандырғыш әсеріне байланысты. Электрофорез әдісі каолин бөлшектерінің pH 3,0-9,0 аралығында теріс зарядты болғанымен, қышқыл ортада теріс ζ-потенциал мәндерінің төмендейтінін көрсетті. ТМАПМАХ:ДМАА мольдік қатынасы 50:50 және 60:40 болатын сополимер үлгілері флокуляция үшін оңтайлы болып табылады, олар каолин бөлшектерінің теріс зарядын бейтараптандыру үшін жеткілікті катиондық топтарды қамтамасыз етеді. Каолин бөлшектерінің ζ-потенциалы мен флокуляция тиімділігі арасындағы байланыс көрсетілген. ТМАПМАХ-ДМАА сополимерінің қатысында каолин бөлшектерінің шөгуіндегі бейтараптандыру және «көпірлік» флокуляция механизмдерінің рөлдері негізделген.

**Түйін сөздер:** флокуляция, полимерлі флокулянттар, каолин суспензиясы, суды тазарту, дзета-потенциал, зарядты бейтараптандыру, көпірлік механизм