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Cationic acrylamide copolymers and terpolymers as flocculants for model aqueous suspensions

Summary — Cationic acrylamide copolymers with diallyldimethylammonium chloride and terpolymers of the both monomers with *N*-vinylformamide were synthesized, characterized by capillary viscometry and their flocculation efficiency evaluated by extinction, sludge volume and solid matter recovery measurements. The polymerization reactions were carried out either as thin-layer processes in aqueous solutions or as microsuspension polymerization. The results from the numerous flocculation tests performed on model aqueous suspensions of fine coal, colloidal silica and talc have been presented and discussed. Flocculation efficiency was determined taking into account the type and dose of the used copolymer.

Key words: acrylamid cationic copolymers and terpolymers, diallyldimethylammonium chloride, *N*-vinylformamide, flocculants, flocculation efficiency.

KATIONOWE KOPOLIMERY I TERPOLIMERY AKRYLOAMIDU JAKO FLOKULANTY MODELOWYCH SUSPENSJI WODNYCH

Streszczenie — Kationowe kopolimery akryloamidu z chlorkiem diallilodimetyloamoniowym oraz terpolimery tych dwóch monomerów z *N*-winyloformamidem (schemat A) otrzymywano dwoma metodami: w cienkiej warstwie roztworu wodnego bądź w postaci mikrosuspensji (tabela 1). Scharakteryzowano je metodami wiskozymetrii kapilarnej (tabela 2) a także oceniono ich efektywność flokulacyjną na drodze pomiaru ekstynkcji oraz objętości i masy osadu poflokulacyjnego. Przedstawiono i przedyskutowano wyniki licznych testów flokulacyjnych przeprowadzonych na modelowych wodnych zawiesinach miazgi węglowej (tabela 3, rys. 1), krzemionki koloidalnej (tabela 4, rys. 2) oraz talku (tabela 5, rys. 3). Efektywność flokulacyjną porównywano uwzględniając typ i dawkę użytego flokulantu.

Słowa kluczowe: akryloamid, kationowe kopolimery i terpolimery, chlorek diallilodimetyloamoniowy, *N*-winyloformamid, flokulanty, efektywność flokulacyjna.

Flocculation is a process in which a particular finely dispersed matter in aqueous systems, such as colloid, is converted into larger structures (flocs) that settle easily on the influence of hydrophilic high molecular weight polymers — flocculants. This is brought about by the bridging action of the flocculant. Colloidal particles can absorb onto long chain hydrophilic macromolecules in such a way that a particular chain actually acts as a bridge between attached two or more particles thus “bridging” them (*i.e.* few macromolecules and some particles) to form flocs [1, 2]. Neutralization of the surface electric charges of the suspended matter by the counter-charged ionic groups of the polymer chains supports flocculation substantially [2, 3].

Flocculant efficiency depends on polymer structure (branching), molecular weight, charge density, dose, amount and also the type of suspended particles. The optimum dose of polymer flocculant is often determined

by jar tests. Relevant data is obtained on the basis of such relations as: interface height [2], extinction [4] versus settling time, or between settling velocity and the radius of gyration of the flocculating polymer [2]. The use of polymeric flocculants leads to rapidly-settling large dense flocs. An increase in the flocculant dose can result in a re-agglomeration of the attached particles [5].

Cationic copolymers based on acrylamide are widely used in water treatment. Acrylamide (AAm) and diallyldimethylammonium chloride copolymers are among the most important macromolecular compounds in that category with a high flocculation efficiency [6–8].

Acrylamide copolymers are synthesised by suspension [9, 10], solution [11, 12] and inverse emulsion polymerization processes [13, 14]. An alternative method can be the radical polymerization carried out as a thin-layer process. The latter polymerization reaction can be initiated by UV or γ radiation as well as conventional radical initiators [15–18]. The products obtained can be used directly after crushing. By thin-layer polymerization, the amount of

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chemicals normally used in the process (organic solvents applied as oily phase in suspension or inverse emulsion polymerization, or the non-solvents used for polymer precipitation) can be decreased or eliminated.

Acrylamide is considered to be a mutagenic monomer and significantly influences the nervous system of living bodies (can cause paralysis and cancer) [19, 20]. For this reason, it is required that the residual amounts of AAm has to in the (co)polymers used for water treatment be limited. According to European Council Directive 98/83/EC regarding all water for domestic consumption, the permissible concentration for AAm in drinking water is 0.10 µg/l. The amount of acrylamide in the polymeric flocculants used in water treatment can be limited by substituting it with an isomeric monomer — *N*-vinylformamide (NVF), that is nontoxic and very reactive in homopolymerization as well as copolymerization reactions [21–25].

The copolymerization of diallyldimethylammonium chloride (DADMAC) with acrylamide is a well known reaction. The reactivity ratios (*r*) for the copolymerization of DADMAC with AAm depend on the content of DADMAC in the initial comonomer solution as well as on the total concentration of both comonomers and differ substantially ($r_{DADMAC} = 0.02\text{--}0.04$, $r_{AAm} = 4.6\text{--}6.7$ [26]).

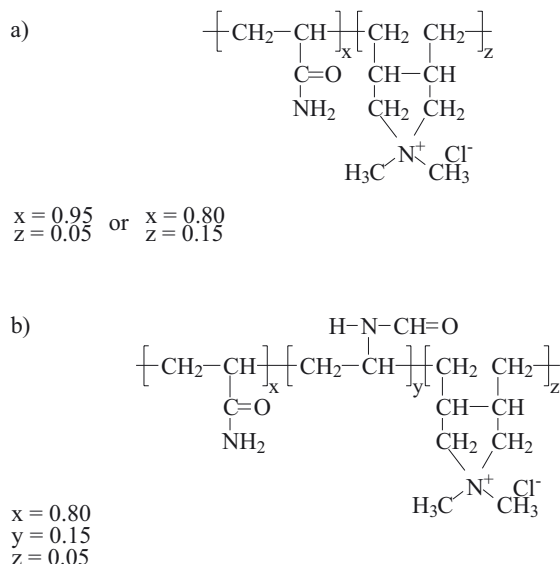
According to Pelton *et al.* [27], the polymerization rate of NVF is higher than in the case of DADMAC, however, the difference in *r* values is less than in the case of the former monomer pair ($r_{DADMAC} = 0.13$, $r_{NVF} = 1.92$). The presence of diallyldimethylammonium chloride in copolyacrylamide flocculants improves the sludge filtration process. DADMAC is also used as a comonomer of AAm in other applications, such as in the paper industry and electro-conducting coatings where antibacterial properties are required [28].

The aim of this work has been to determine the influence of *N*-vinylformamide on the flocculation properties of cationic copolymers of AAm and DADMAC synthesized either by thin-layer solution or microsuspension polymerization.

EXPERIMENTAL

Materials and polymer synthesis

AAm (~99 %, Fluka) and DADMAC (65 % water solution, Aldrich) were used for the synthesis of cationic



Scheme A. Scheme illustrating cationic acrylamide co- and terpolymer chains: a) AAm/DADMAC, b) AAm/NVF/DADMAC

copolymers. The molar ratio of AAm/DADMAC in monomer mixtures were 95/5 (base copolymer) and 85/15. In the synthesis of cationic terpolymers, the 15 mol. % of AAm in the base copolymerizable mixture (95/5) was replaced with *N*-vinylformamide (NVF) (~98 %, Aldrich). Chemical structure of the respective copolymer and terpolymer chains are presented in Scheme A.

Thin-layer (2 mm thick) 50 wt. % solution polymerization was carried out in poly(tetrafluoroethylene) form, while microsuspension polymerization was performed in a glass reactor (both types of polymerizations at 50 ± 2 °C, for 4 h) [29]. The reaction mixtures were purged with nitrogen before the reactions were initiated. Ammonium persulfate (POCH, Poland) was used as a radical initiator (0.5 wt. %) in copolymerization reactions. 2,2'-Azobis(2-amidinopropane) dihydrochloride (~97 %, Aldrich) (0.5 wt. %) was used as a radical initiator for terpolymerization processes. The initiators were dissolved in a 50 wt. % aqueous solution of the monomers.

The organic phase in microsuspension polymerization reaction contained Span 80 (sorbitan monooleate, Sigma-Aldrich) and a mixture of toluene and heptane (3:1 wt./wt.). Polymeric products from this kind of polymerization have been dissolved in distilled water, precipitated in methanol and dried at 60 °C after filtration.

Table 1. Methods of synthesis and product acronyms

Method of synthesis	Product acronym	Kind of initiator
Microsuspension copolymerization	P(AAm/DADMAC) 95/5	ammonium persulfate
Solution thin-layer copolymerization	P(AAm/DADMAC) 95/5*	
Microsuspension co- and terpolymerization	P(AAm/DADMAC) 85/15	ammonium persulfate
	P(AAm/NVF/DADMAC) 80/15/5	2,2'-azobis(2-amidinopropane) dihydrochloride
Solution thin-layer co- and terpolymerization	P(AAm/DADMAC) 85/15*	ammonium persulfate
	P(AAm/NVF/DADMAC) 80/15/5*	2,2'-azobis(2-amidinopropane) dihydrochloride

* From text

The methods of synthesis and acronyms of the obtained products are presented in Table 1. The acronyms of the solution thin-layer co- and terpolymerization products are marked with an asterisk, whereas those from microsuspension processes are not.

Fine coal (< 0.22 mm; Hossa, Poland), colloidal silica Aerosil 150 (nanoparticulate Conlac, Chemia Partner, Poland) and talc (grain size <6 μm ; Alfa, II Quality, Slovakia) were used as inorganic pollutants.

Measurements

Capillary viscometry

The intrinsic viscosity values of the AAm copolymers and terpolymers in aqueous solutions were compared on the basis of capillary viscometric measurements carried out in an Ubbelohde's viscometer. The measurements were performed at 30 ± 0.2 °C, repeatedly for five times for each polymer solution.

Flocculation tests

A model suspension was prepared by mixing 5 g of the fine pollutant material with 1 dm³ of distilled water in a beaker. The required amount of the tested copolymer solution was added into the beaker while stirring. The stirring was continued for 10 s at 400 rpm after the addition of the copolymer and then reduced to 50 rpm for another 10 min. The obtained suspension was transferred to a calibrated Imhoff funnel and left to settle down.

The extinction of the aqueous phase was measured using Spekol 11 spectrophotometer (Carl Zeiss Jena; wavelength 470 nm) every 10 minutes over a period of 2 hours. The volume of the settled deposit and its mass after drying were measured. Similar measurements (under the same experimental conditions), but without the flocculant (blind test) were carried out for comparison purposes.

RESULTS AND DISCUSSION

Co- and terpolymers

The intrinsic viscosity $[\eta]$ values of AAm/DADMAC copolymers and AAm/NVF/DADMAC terpolymers obtained using the both polymerization methods are presented in Table 2. A comparison of the values for the basic copolymers [P(AAm/DADMAC) 95/5] from the two polymerization methods reveals that the intrinsic viscosity of product obtained in microsuspension polymerization is almost twice as much for the copolymer synthesized by thin-layer method. An increase in the cationic comonomer content in the copolymerization system up to 15 mol. % of DADMAC caused a significant increase in the intrinsic viscosity values of the copolymers irrespective of the polymerization method ($[\eta] > 3000$ cm³/g). The observed differences in $[\eta]$ values

were caused most probably by the polyelectrolytic effect — a repulsion of the positively charged segments of the copolymers with a higher density of ammonium cations leading to higher coil sizes of the macromolecular chains and consequently to higher intrinsic viscosities. This is on the assumption, that molecular weights of the copolymers with higher DADMAC content are rather lower than those of P(AAm/DADMAC) 95/5 copolymers and the respective chain stiffnesses are similar [30].

Table 2. Intrinsic viscosities ($[\eta]$) of acrylamide co- and terpolymers

Copolymer/terpolymer	$[\eta]$ value, cm ³ /g
P(AAm/DADMAC) 95/5	1360
P(AAm/DADMAC) 95/5*	770
P(AAm/DADMAC) 85/15	3550
P(AAm/NVF/DADMAC) 80/15/5	340
P(AAm/DADMAC) 85/15*	3290
P(AAm/NVF/DADMAC) 80/15/5*	660

^{*)} Description of copolymers — see Table 1.

A replacement of the 15 mol. % AAm in the basic comonomer composition [P(AAm/DADMAC) 95/5] with NVF causes a significant decrease in the $[\eta]$ values for terpolymers obtained by both polymerization methods, as compared with those for the base AAm/DADMAC copolymers (Table 2). This is probably related to the diminishing molecular weight of the AAm/NVF/DADMAC terpolymers in comparison with the AAm/DADMAC copolymers. NVF usually polymerizes to products with lower molecular weights than AAm polymerization counterparts [24]. In the investigated system, an introduction of NVF to the monomer mixture cause a reduction of the $[\eta]$ value of the terpolymer P(AAm/NVF/DADMAC) 80/15/5 by a factor of 4 (340 cm³/g) in comparison with copolymer P(AAm/DADMAC) 95/5 (1360 cm³/g) for microsuspension polymerization products. The difference of $[\eta]$ values for the respective pair of solution thin-layer polymerization products: P(AAm/NVF/DADMAC) 80/15/5* (660 cm³/g) and P(AAm/DADMAC) 95/5* (770 cm³/g) is significantly lower.

Fine coal suspension flocculation

The highest suspension clarification was observed for lower flocculant dose (0.75 $\mu\text{g}/\text{cm}^3$) using the microsuspension polymerization terpolymer P(AAm/NVF/DADMAC) 80/15/5 (even though its $[\eta]$ value is the lowest among the tested copolymers) and P(AAm/DADMAC) 85/15* copolymer (Fig. 1a and Table 3). The application of the former terpolymer led to a reduction in extinction by 91 % as compared to the blind test after 10 minutes of flocculation. An increase in the charge density of the copolymers caused an increase in flocculation efficiency. A comparison between the floccu-

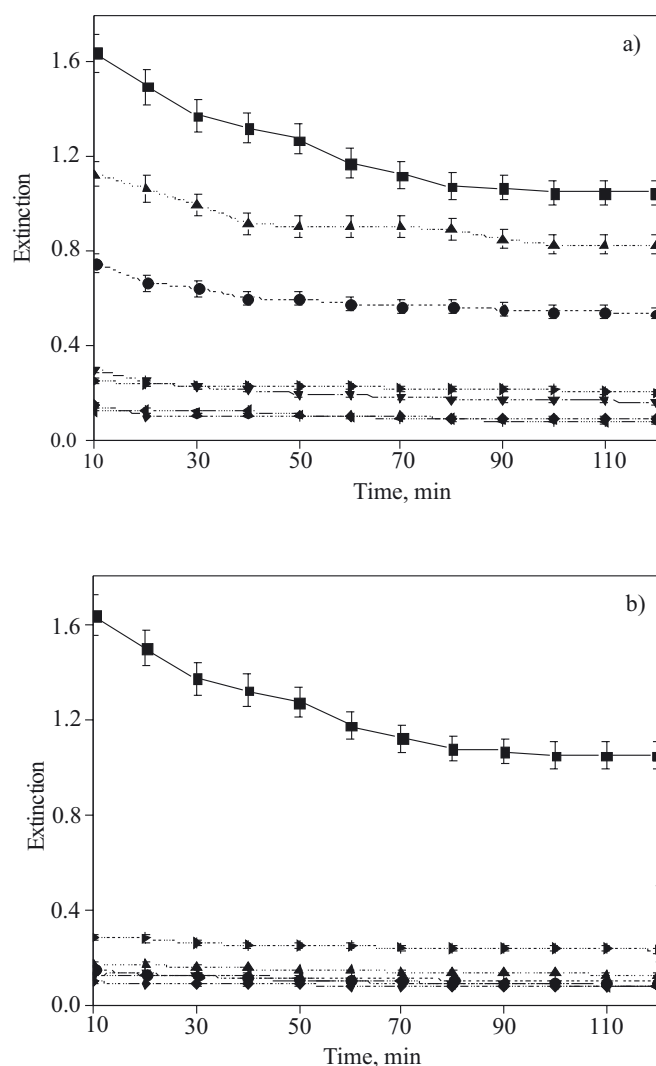


Fig. 1. Extinction of fine coal suspension as a function of time for the tested products: a) dose $0.75 \mu\text{g}/\text{cm}^3$, b) dose $3.0 \mu\text{g}/\text{cm}^3$; ■ — blind test, ● — P(AAm/DADMAC) 95/5, ▲ — P(AAm/DADMAC) 95/5*, ▼ — P(AAm/DADMAC) 85/15, ◆ — P(AAm/NVF/DADMAC) 80/15/5, ◀ — P(AAm/DADMAC) 85/15*, ▶ — P(AAm/NVF/DADMAC) 80/15/5*

lation efficiency of copolymers obtained using thin-layer solution and those obtained by micro-suspension copolymerization confirms the better results generated by the latter products. A four-fold increase in flocculant dose (Fig. 1b) caused an additional reduction of extinction for all the applied AAm copolymers. This is especially noticeable for thin-layer polymerization products. The sludge volume as well as mass recovery of inorganic suspended matter increased with an increase in the flocculant dose (Table 3). A distinct influence of type of the tested products on fine coal sludge volume was not observed. The recovery of suspended coal particles was better for products synthesized by micro-suspension polymerization (irrespective of the dose used) and for higher doses it was typically in the 90–95 wt. % range.

Silica suspension flocculation

The results obtained in the use of synthesized copolymers as flocculants for colloidal silica suspension were not as good as those obtained in the case of fine coal suspension. This may have been caused by the rather poor interactions between cationic copolymers and colloidal silica particles. At a lower flocculant doses (Fig. 2a, Table 4) only AAm/DADMAC 95/5 copolymers (from both types of copolymerization) slightly improved the clarity of silica suspension in comparison with the blind test. At a higher dose ($3.0 \mu\text{g}/\text{cm}^3$, Fig. 2b), the extinction was improved using the copolymer from micro-suspension method P(AAm/DADMAC) 85/15 (over 80 % extinction reduction in comparison with untreated system, acceptable mass recovery but high sludge volume) and to a lower degree with the copolymer AAm/DADMAC 95/5 and terpolymer P(AAm/NVF/DADMAC) 80/15/5* (worse extinctions but better results of sludge volumes and mass recovery, Table 4). In the other tests, the deflocculating effect of colloidal silica particles was observed. For the P(AAm/DADMAC) 85/15 copolymer, the recovery mass was exceptionally significant (above 82 wt. %).

Table 3. Flocculation parameters measured for fine coal suspension

Flocculant dose, $\mu\text{g}/\text{cm}^3$	0.75	3.0	0.75	3.0	0.75	3.0	0.75	3.0
Flocculant	Flocculation parameters							
	Extinction reduction, % ¹⁾				Sludge volume, cm^3		Mineral-mass recovery, g	
	after 10 min		after 120 min					
Blind test	0		36		8.0		3.90	
P(AAm/DADMAC) 95/5	54	91	67	94	11.5	14.0	4.45	4.66
P(AAm/DADMAC) 95/5*	31	90	50	92	11.0	14.0	4.02	4.51
P(AAm/DADMAC) 85/15	82	92	90	95	15.0	17.0	4.57	4.74
P(AAm/NVF/DADMAC) 80/15/5	91	94	94	95	13.0	14.5	4.57	4.64
P(AAm/DADMAC) 85/15*	77	92	91	96	12.0	13.0	4.40	4.51
P(AAm/NVF/DADMAC) 80/15/5*	85	83	88	86	13.0	16.0	4.39	4.46

¹⁾ Extinction reduction as a ratio of extinction values after 10 or 120 min of particular test to the blind test extinction after 10 min.

Table 4. Flocculation parameters measured for colloidal silica suspension

Flocculant dose, $\mu\text{g}/\text{cm}^3$	0.75	3.0	0.75	3.0	0.75	3.0	0.75	3.0
Flocculant	Flocculation parameters							
	Extinction reduction, % ¹⁾				Sludge volume, cm^3		Mineral-mass recovery, g	
	after 10 min		after 120 min					
Blind test	0		12		120		4.23	
P(AAm/DADMAC) 95/5	31	42	39	58	125	125	4.24	4.45
P(AAm/DADMAC) 95/5*	14	defloc.	33	8	120	120	4.39	4.20
P(AAm/DADMAC) 85/15	defloc.	82	defloc.	89	130	195	4.04	4.85
P(AAm/NVF/DADMAC) 80/15/5	defloc.	defloc.	defloc.	defloc.	120	130	3.96	4.04
P(AAm/DADMAC) 85/15*	defloc.	defloc.	defloc.	defloc.	110	110	3.92	3.90
P(AAm/NVF/DADMAC) 80/15/5*	0	39	17	61	105	125	4.22	4.46

¹⁾ Extinction reduction as a ratio of extinction values after 10 or 120 min of particular test to the blind test extinction after 10 min.

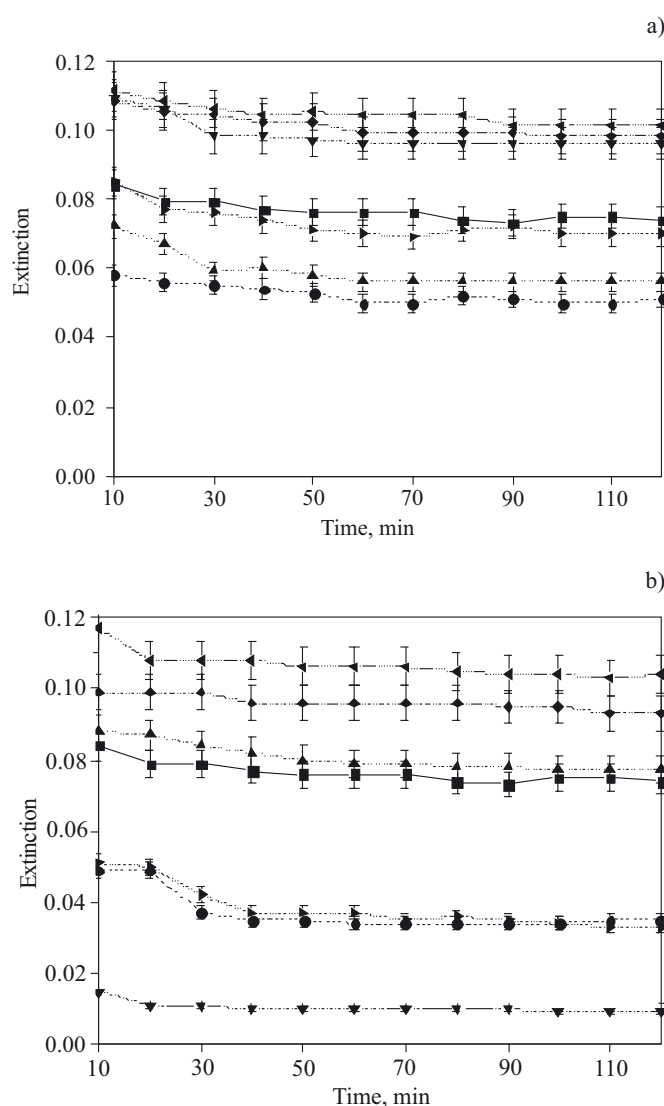


Fig. 2. Extinction of colloidal silica suspension as a function of time for the tested products: a) dose $0.75 \mu\text{g}/\text{cm}^3$, b) dose $3.0 \mu\text{g}/\text{cm}^3$; symbols — see Fig. 1

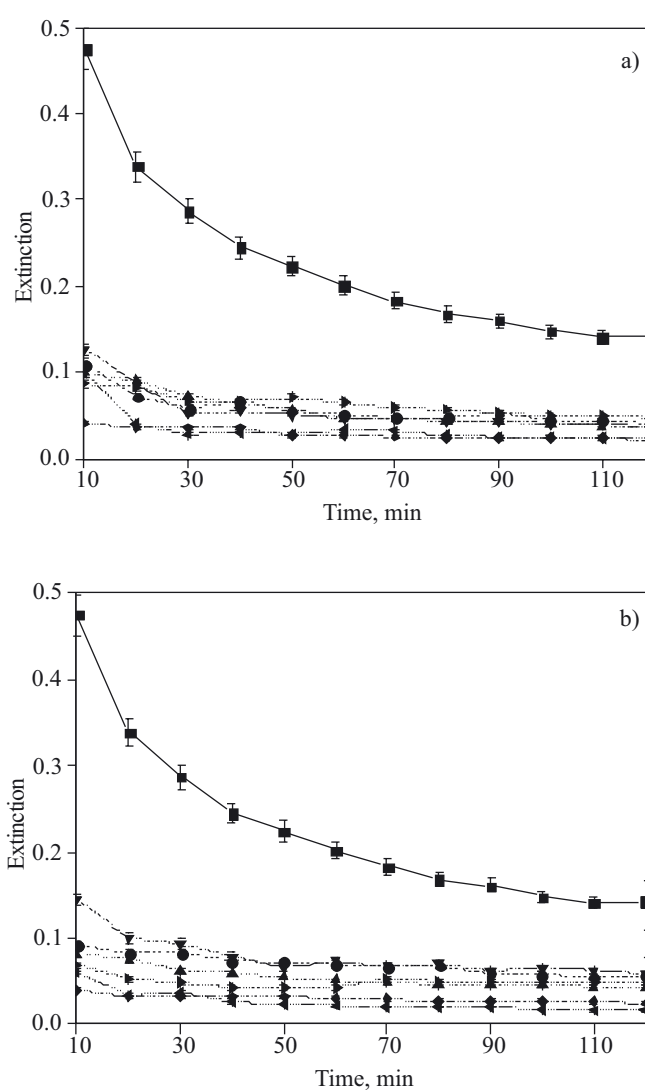


Fig. 3. Extinction of talc suspension as a function of time for the tested products: a) $0.75 \mu\text{g}/\text{cm}^3$ dose, b) $3.0 \mu\text{g}/\text{cm}^3$ dose; symbols — see Fig. 1

Talc suspension flocculation

All the tested products exhibited flocculation activity for talc suspensions, independent of the applied dose

(Fig. 3 and Table 5). The best extinction reduction was observed for P(AAm/NVF/DADMAC) 80/15/5 and P(AAm/DADMAC) 85/15* (ca. 95 % after 120 minutes

Table 5. Flocculation parameters measured for talc suspension

Flocculant dose, $\mu\text{g}/\text{cm}^3$	0.75	3.0	0.75	3.0	0.75	3.0	0.75	3.0
Flocculant	Flocculation parameters							
	Extinction reduction, % ¹⁾				Sludge volume, cm^3		Mineral-mass recovery, g	
	after 10 min		after 120 min					
Blind test	0		70		8.5		4.40	
P(AAm/DADMAC) 95/5	77	81	91	88	11.0	12.0	4.41	4.15
P(AAm/DADMAC) 95/5*	73	69	92	88	12.5	13.0	4.81	4.75
P(AAm/DADMAC) 85/15	79	83	92	91	12.0	10.5	4.74	4.38
P(AAm/NVF/DADMAC) 80/15/5	91	92	95	95	14.0	15.0	4.92	4.87
P(AAm/DADMAC) 85/15*	79	87	95	97	11.5	13.0	4.85	4.90
P(AAm/NVF/DADMAC) 80/15/5*	82	85	90	90	15.0	17.0	4.85	4.86

¹⁾ Extinction reduction as a ratio of extinction values after 10 or 120 min of particular test to the blind test extinction after 10 min.

for both flocculant doses). The use of the tested cationic AAm co- and terpolymers usually caused an increase in the sludge volume in comparison with the blind test. Generally, the flocculant sludge volume increased with the higher dose. An introduction of NVF into the copolymer generated an increase in the sludge volume in comparison with two-component AAm/DADMAC copolymers. Over 96 wt. % of the suspended talc was recovered over most of the tested copolymers with the exception P(AAm/DADMAC) 95/5 and P(AAm/DADMAC 85/15).

CONCLUSIONS

The conclusions can be summarised as follows:

— The introduction of *N*-vinylformamide to the AAm/DADMAC mixture causes a decrease in the intrinsic viscosities for the AAm/NVF/DADMAC terpolymers in comparison with AAm/DADMAC copolymers synthesized in microsuspension and solution thin-layer radical polymerization.

— Clarification by self-sedimentation of suspended matter (blind tests evaluated by extinction changes after 120 min) increases in the order: colloidal silica (~93 %) < fine coal (~68 %) < talc (~34 %).

— The most effective cationic acrylamide co- and terpolymer flocculants cause a decrease in the final extinctions of the aqueous systems in the following order: colloidal silica (~12.5 %) < talc (~8.5 %) < fine coal (~6.0 %).

— The best flocculation results are observed when AAm/DADMAC 85/15 copolymer and AAm/NVF/DADMAC 80/15/5 terpolymer are used for fine coal and talc suspensions and AAm/DADMAC 95/5 copolymer used for colloidal silica.

— The sludge volumes for recovered fine coal and talc lay in the range of about $3.0 \text{ cm}^3/\text{g}$, whereas the respective values for nanoparticulate silica are almost one order magnitude higher (*ca.* $28 \text{ cm}^3/\text{g}$).

— The recovery of minerals from deposited sludges was rather high for fine coal and talc and in optimal cases reached level above 93 wt. %.

The presented results confirm that acrylamide cationic copolymers with diallyldimethylammonium chloride or terpolymers containing *N*-vinylformamide can be applied for the effective flocculation of mineral suspensions such as fine coal and talc.

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W kolejnym zeszycie ukaza się m.in. następujące artykuły:

- M. Żenkiewicz, A. Szach — Wybrane problemy termoformowania materiałów polimerowych
- K. German — Wybrane zagadnienia pirolizy odpadów tworzyw polimerowych. Cz. I. Problem obecności chloru w oleju pirolitycznym w świetle aktualnego stanu wiedzy
- D. Filip, D. Macocinschi, G. E. Ioanid, S. Vlad — Multiblokowe poliuretany i poliuretano-moczniki o budowie segmentowej na podstawie makrodioli poli(tlenek etylenu)/poli(tlenek propylenu)/poli(tlenek etylenu). Otrzymywanie i charakterystyka (*j. ang.*)
- K. M. Tomczyk, P. G. Parzuchowski, J. Kozakiewicz, J. Przybylski, G. Rokicki — Synteza oligowęglanodioli z „zielonego monomeru” — węglanu dimetylu — jako segmentów miękkich elastomerów poli(uretano-mocznikowych) (*j. ang.*)
- J. Paciorek-Sadowska — Modyfikacja pianek PUR-PIR związkami boroorganicznymi na podstawie bis(hydroksymetylo)mocznika (*j. ang.*)
- P. Jakubowska, T. Sterzyński, B. Samujło — Badania reologiczne kompozytów poliolefinowych o wysokim stopniu napełnienia z uwzględnieniem charakterystyk $p-v-T$
- S. Frąckowiak, M. Kozłowski — Polimerowe kompozyty elektroprzewodzące jako materiały o potencjale sensorycznym
- J. Jaglarz, J. Cisowski, D. Bogdał, A. Kassiba, J. Sanetra — Badanie cienkich warstw polimerowych modyfikowanych nanocząstkami SiC (*j. ang.*)