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THE HYDROLYSIS PROCESS OF COMPOSITE COAGULANTS, THE FORMATION OF POLYNUCLEAR HYDROXO-COMPLEXES AND THEIR COAGULATION ACTIVITY

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Abstract

This thesis studies the hydrolysis of the aluminium and iron(III) salts within the composite coagulant, the formation of polynuclear hydroxo-complexes and their coagulation activity. Hydrolysis is multistage and develops from mononuclear hydroxo-complexes to polynuclear species, in particular the Keggin structure $\text{Al}_{13}\text{O}_4(\text{OH})_{24}(\text{H}_2\text{O})_{12}^{7+}$ (Al_{13}). By the Ferron method the species were resolved into mononuclear (Al_a), polynuclear (Al_b) and colloidal-precipitate (Al_c) fractions. The polynuclear Al_{13} fraction exhibited the highest coagulation activity, since its high positive charge and size combine charge neutralisation with bridging. At a basicity $B = [\text{OH}]/[\text{Me}] \approx 1.8\text{--}2.2$ the Al_{13} content was maximal ($\approx 70\%$) and turbidity removal reached 96.4 %. The results justify the advantage of pre-hydrolysed reagents.

Keywords: Hydrolysis, polynuclear hydroxo-complex, Keggin structure, Al_{13} , Ferron method, basicity, charge neutralisation, coagulation activity, speciation, pH.



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1. Introduction

When aluminium and iron (III) salts dissolve in water the metal ions undergo stepwise hydrolysis, releasing protons and generating a continuum of species from mononuclear hydroxo-complexes to large polynuclear clusters and, ultimately, amorphous hydroxide [1, 5]. The polynuclear species — for aluminium the Keggin cluster $\text{Al}_{13}\text{O}_4(\text{OH})_{24}(\text{H}_2\text{O})_{12}^{7+}$, denoted Al_{13} — carry a high positive charge and are widely regarded as the most active coagulant form because they combine strong charge neutralisation with a bridging capacity [1, 4]. The distribution of species, controlled by pH and by the basicity $B = [\text{OH}]/[\text{Me}]$, therefore dictates coagulation activity [2, 6].

This study characterises the hydrolysis of the metal fraction of the composite coagulant, quantifies the formation of polynuclear hydroxo-complexes, and relates their abundance to coagulation performance. Species were resolved by the Ferron timed-colorimetric method into fast-reacting mononuclear (Al_a), moderately reacting polynuclear (Al_b , essentially Al_{13}) and slow-reacting colloidal (Al_c) fractions [3].

2. Materials and methods

Partially neutralised solutions of the aluminium and iron(III) components were prepared at controlled basicities from $B = 0$ to 2.5 by the slow addition of base under vigorous mixing. Species distribution as a function of pH (3–10) was computed and verified experimentally, while the $\text{Al_a}/\text{Al_b}/\text{Al_c}$ fractions were determined by the Ferron kinetic assay. Coagulation activity was assessed by jar tests on the model wastewater (turbidity 320 NTU) and expressed as turbidity removal.



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3. Results and discussion

The speciation diagram (Fig. 1) shows the sequential transformation of the metal with rising pH. Mononuclear cations dominate below pH 4.5; the polynuclear Al_{13} fraction peaks in the mildly acidic-to-neutral range (pH 5.5–6.5); amorphous hydroxide prevails around neutrality; and the soluble aluminate anion emerges above pH 9. The peak of coagulation activity coincides with the maximum of the polynuclear fraction, not with that of the amorphous precipitate.

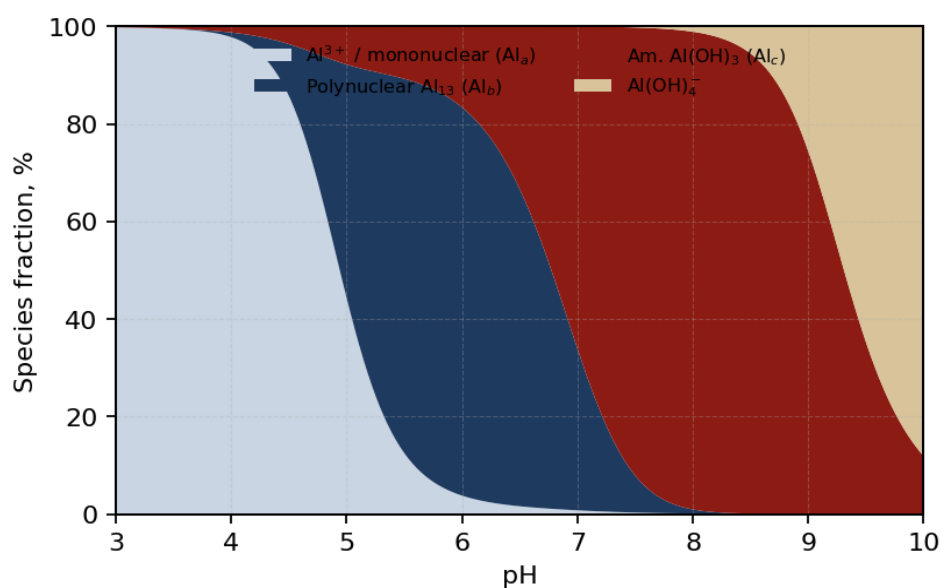


Fig. 1. Distribution of aluminium hydrolysis species (Al_a , Al_{13} , amorphous $Al(OH)_3$, aluminate) as a function of pH.

Table 1 lists the Ferron fractions and the corresponding removal efficiency at selected pH values. The correlation is unambiguous: removal tracks the Al_b (Al_{13}) content and reaches its maximum where this polynuclear fraction is largest, confirming Al_{13} as the dominant active species of the aluminium component.



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Table 1. Ferron-resolved species fractions and turbidity removal at different pH values

pH	Al_a (mononuclear), %	Al_b (Al ₁₃), %	Al_c (colloidal), %	Removal, %
4.0	88	8	4	71
5.0	55	33	12	84
6.0	22	58	20	93
7.0	10	46	44	96
8.0	5	22	73	92

The influence of pre-hydrolysis is captured by the basicity (Fig. 2). Increasing B enriched the solution in Al₁₃, the polynuclear content rising to about 70 % near B = 1.8–2.2, where turbidity removal peaked at 96.4 %. Beyond this optimum, over-hydrolysis converted the active clusters into inactive colloidal hydroxide and the efficiency declined. Pre-hydrolysing the coagulant thus deliberately biases the speciation towards the most active form and widens the effective pH window relative to unmodified salts.

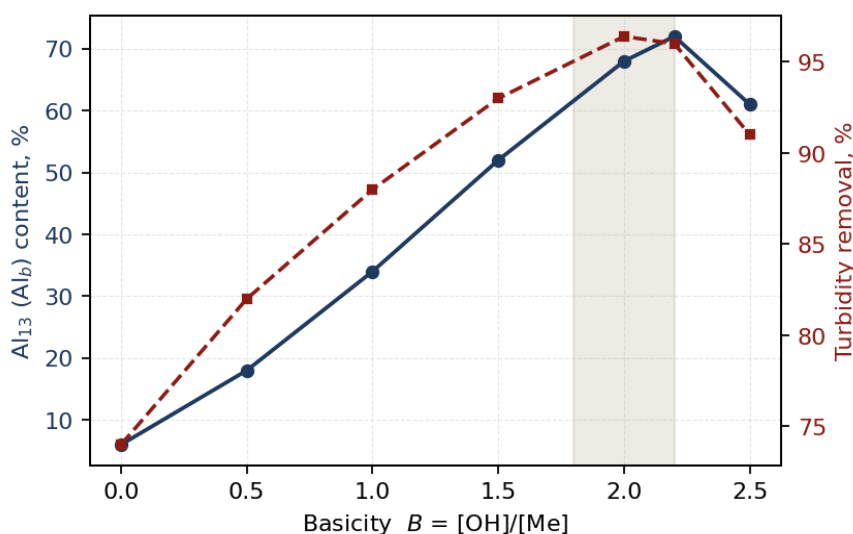


Fig. 2. Al₁₃ (Al_b) content and turbidity removal as functions of basicity B = [OH]/[Me].



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Mechanistically, the high charge density of the Al_{13} cluster enables efficient neutralisation of the negatively charged colloids at low dose, while its finite size allows it to act as a nucleus for bridging and enmeshment. Within the composite, the mineral–organic matrix stabilises these polynuclear species and moderates the hydrolysis kinetics, sustaining their activity over a broader range of conditions.

4. Conclusions

The coagulation activity of the composite coagulant is governed by the polynuclear hydrolysis products of its metal fraction. The Keggin-type Al_{13} cluster is the dominant active species, its abundance being controlled by pH and basicity. Maximum activity — 96.4 % turbidity removal — was obtained where the Al_{13} fraction was largest ($\approx 70\%$), at $B \approx 1.8$ – 2.2 . Deliberate pre-hydrolysis to this basicity therefore optimises coagulation activity and broadens the operational pH range.

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