Chemical Engineering Journal 156 (2010) 64–69

Contents lists available at ScienceDirect

Chemical Engineering Journal

journal homepage: www.elsevier.com/locate/cej

Preparation and performance of a high purity poly-aluminum chloride

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ARTICLE INFO

Article history:
Received 12 June 2009
Received in revised form
17 September 2009
Accepted 18 September 2009

Keywords:
Aluminate
Basicity
Bauxite
Coagulation
Poly-aluminum chloride (PACl)
Water treatment

ABSTRACT

Poly-aluminum chloride (PACl) is one of the common coagulants for water and wastewater treatment. A new Chinese national standard of PACl has been implemented, where the concentration of insoluble substances, iron and heavy metals is to be controlled strictly, then researches on the preparation of high-purity PACl are needed. This paper presents a novel method to prepare high-purity PACl which contains high aluminum oxide content (>10%), high basicity (>90%), and low insoluble substance (<0.04%), iron (<300 mg L^{-1}) and heavy metals. Samples taken from the Huangpu River were used to evaluate the coagulation performance of high-purity PACl in comparison with conventional PACl. The results demonstrate that for the whole dose range studied, high-purity PACls removed more turbidity than conventional PACl. On the other hand, when the dose was greater than 4 mg L^{-1} as Al, the high-purity PACl showed great UV_{254}-absorbance removal capacity; which was even much greater at high does (e.g., 10 mg L^{-1}). This could be partly attributed to the charge effect where a relative high zeta potential in the test water was achieved by dosing high-purity PACl.

1. Introduction

Coagulation is an established process for transforming colloidal particles into larger aggregates (flocs) and for adsorbing dissolved organic matter on to particulate aggregates so that these impurities can be removed in subsequent liquid/solid separation processes. The coagulation process consists of three sequential steps: coagulant formation, colloid/particle destabilisation, and particle aggregation. Coagulants used for water and wastewater treatment are predominantly inorganic salts of aluminium and iron. When dosed into water the aluminium or iron ions hydrolyse rapidly and in an uncontrolled manner, to form a range of metal hydrolysis species. A range of factors such as the nature of the water, the coagulation pH and the dose of coagulant together influence the range of species formed and subsequently, the treatment performance. Considerable interest and attention have been paid in last three decades [1–4] to prepare pre-hydrolysed metal-ion coagulants, based on either aluminium (e.g., poly-aluminium chloride) or ferric iron (e.g., polyferric sulfate). These have been shown to perform better in some cases, in comparison with conventional coagulants such as aluminium sulfate or ferric sulfate. The superior performance of pre-polymerized coagulants is attributed to their wider working pH range, a lower sensitivity to low water temperature, lower doses required to achieve the equivalent treatment efficiency, and lower residual metal-ion concentrations [5].

One of the major pre-polymerized coagulants, poly-aluminum chloride (PACl) is used broadly for water and wastewater treatment. PACl contains different amounts of hydroxyl, and the molecular formula of PACl is $[Al_m(OH)_n(H_2O)_x]{\cdot}Cl_{3m-n}$ $(n \leq 3m)$. The application of PACl started more than 30 years ago, and the scope of the application and production has been growing significantly. The annual requirement and production capacity of PACl in the liquid form has reached 2 million tons in China, and the turnover of PACl has been more than 2 billion Chinese Yuan (equivalent to 310 million US dollars).

There are many ways to prepare PACl, such as thermal decomposition, electrolysis, membrane reactor, and acid/alkaline dissolution [6–11]. In general, a national acceptable synthesis method involves a two-step method where the bauxite, hydrochloric acid and calcium aluminate are used as raw materials to prepare a liquid PACl. The bauxite first reacts with hydrochloric acid then calcium aluminate to produce a given Al_2O_3 concentration and a given basicity [12]. The resulting product has Al_2O_3 concentration >10% and the basicity ranging 45–95%. Another preparation method of PACl is that the aluminium hydroxide is used as a major raw material which is mixed with the given amount of 20–30% of HCl and 3–5% sulfuric acid in a reactor under relative high temperature (110–140 °C) and pressure ($2–4 \times 10^5$ Pa) for 2.5–4 h, then after cooling and filtration, a PACl is prepared which has relatively high-purity but low basicity [12]. Due to the high cost of raw materials and operating conditions, this method is relatively expensive.

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doi:10.1016/j.cej.2009.09.034

Table 1
Characteristic properties of bauxite.

Item	Chemical component (%)							Volume density (g/cm^3)	Water absorption ratio%
	Al_2O_3	Fe_2O_3	TiO_2	SiO_2	CaO + MgO	$K_2O + Na_2O$	Other oxides		
Bauxite (light frit)	30–40	1–5	0.2–0.5	45–50	1.2–2.4	1.4–1.9	0.3–2.4	≥3.15	≤4

Therefore, the method is seldom adopted in preparing PACl for the non-drinking water treatment in China.

The impurity content is high in some ores in China, which are used to prepare PACl. For example, iron content is relatively high; there are certain iron (0.1–3%) remained in the resulting PACl products. It has been known that aluminum coagulants with iron usually boost the coagulation performance, however, in some specific fields, there are strict restrictions for the iron content. In addition, harmful ions such as Pb, Co, Cu and As from the raw materials (bauxite and calcium alumina) are normally remained in the product PACl, which restricts the application of PACl in drinking water treatment. Such PACls are only used for industry effluent and sewage treatment [13]. A high-purity PACl for drinking water treatment is thus required. The aims of this paper are to present a technique to prepare a high-purity PACl using low grade bauxite and calcium aluminate as raw materials and to produce PACl with high aluminium content and basicity, and low contents of iron, heavy metals and insoluble impurities.

2. Synthesis experiment

The process to prepare high-purity PACl can be divided into three steps, namely, bauxite dissolving, basicity promotion and the removal of metal ions and insoluble impurities.

2.1. Dissolving of bauxite with HCl acid

Bauxite with the particle size under 150 meshes, namely light frit, has 30–40% weight of alumina and 1–5% of ferric oxide. The properties of the bauxite can be seen in Table 1. Generally, the higher concentration of hydrochloric acid is used, the higher percentage of alumina dissolves out. In view of high volatilization, hydrochloric acid with a concentration of 20% is usually used. Small amount of sulfate solution can promote the reaction rate in the bauxite dissolving process. Enamel reaction kettle or atmospheric reaction pond is suitable for the bauxite dissolving process. Using the steam to raise temperature to 100–110 °C under atmospheric pressure, the bauxite dissolving process was completed for 3–4 h, which can be seen in Eq. (1). The dissolveout is separated by sedimentation for several minutes, which results in a lucid solution, and then filtered. The filter residues are silicon oxide dominant minerals which cannot react with hydrochloric acid.

$$Al_2O_3 + 6HCl = 2AlCl_3 + 3H_2O \qquad (1)$$

2.2. Adjustment of basicity by calcium alumina

The aluminum content and the basicity are the most important quality control parameters for the production of PACl. Currently, calcium aluminate is widely used to adjust the basicity; the resulting PACl products possess good stability which is suitable for long term storage [14].

Table 2
Properties of calcium aluminate.

Product name	Calcium alumina powder
Appearance	Off white, fine powder
Particle size	120–180 meshes
Main chemical component	
Al_2O_3 (%)	≥55
CaO (%)	≤30
MgO (%)	≤3
Fe_2O_3 (%)	≤2
SiO_2 (%)	≤7
TiO_2 (%)	≤3
Mn (mg L^{-1})	≤110
Cr (mg L^{-1})	≤45
Pb (mg L^{-1})	≤45

Adding calcium alumina into a low basicity of PACl can adjust the basicity of PACl up to 90%. The properties of calcium alumina can be seen in Table 2.

The reaction to adjust basicity of PACl can be seen in Eq. (2):

$$10mAlCl_3 + 3mCa[Al(OH)_4]_2 = 8[Al_2(OH)_3Cl_3]_m + 3mCaCl_2 \qquad (2)$$

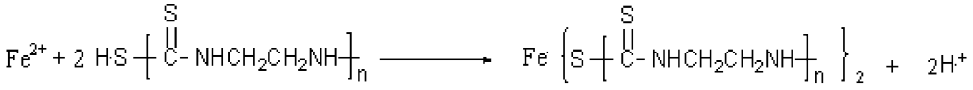
(m is a positive integer).

2.3. Removal of metal ions and insoluble impurities

Most calcium aluminate consists of relatively high heavy metals, and then the removal of heavy metals should be carried out in the preparation process. One method is to add sodium sulfide into the above-mentioned solution after the basicity adjustment. After reaction, precipitate or filter press to remove heavy metals. Take Pb^{2+} as an example, the reaction is shown in Eq. (3):

$$Pb^{2+} + S^{2-} = PbS \downarrow \qquad (3)$$

However, sodium sulfide is not prominent in the removal of iron ions in PACl. The structure of sulfydryl polymer and the reaction with metals (e.g., Fe) can be seen in Eq. (4) which shows a complexation mechanism of Fe and sulfydryl polymer. According to this, a sulfydryl polymer was used to adsorb iron in weak acid surroundings, which removes iron in a high efficiency. Moreover, a previous study [12] found that dosing sulfydryl polymer and modified polyacrylamide (PAM) together would remove Fe and other heavy metals simultaneously. Therefore, a sulfydryl polymer was added with given amount of modified PAM to remove all insoluble impurities in PACl:



$$Fe^{2+} + 2\,HS\left[\overset{S}{\overset{\|}{C}}-NHCH_2CH_2NH\right]_n \longrightarrow Fe\left\{S\left[\overset{S}{\overset{\|}{C}}-NHCH_2CH_2NH\right]_n\right\}_2 + 2H^+ \qquad (4)$$

3. Synthesis and characterization of PACl and jar test procedures

3.1. Synthesis and characterization of PACl

The process of preparing a high-purity PACl is summarized in Fig. 1. After PACl was prepared, the polymers with a sulfydryl group and modified PAM were added into the conventional liquid PACl, this not only removes iron significantly, but also accelerates the sedimentation of insoluble suspended matter.

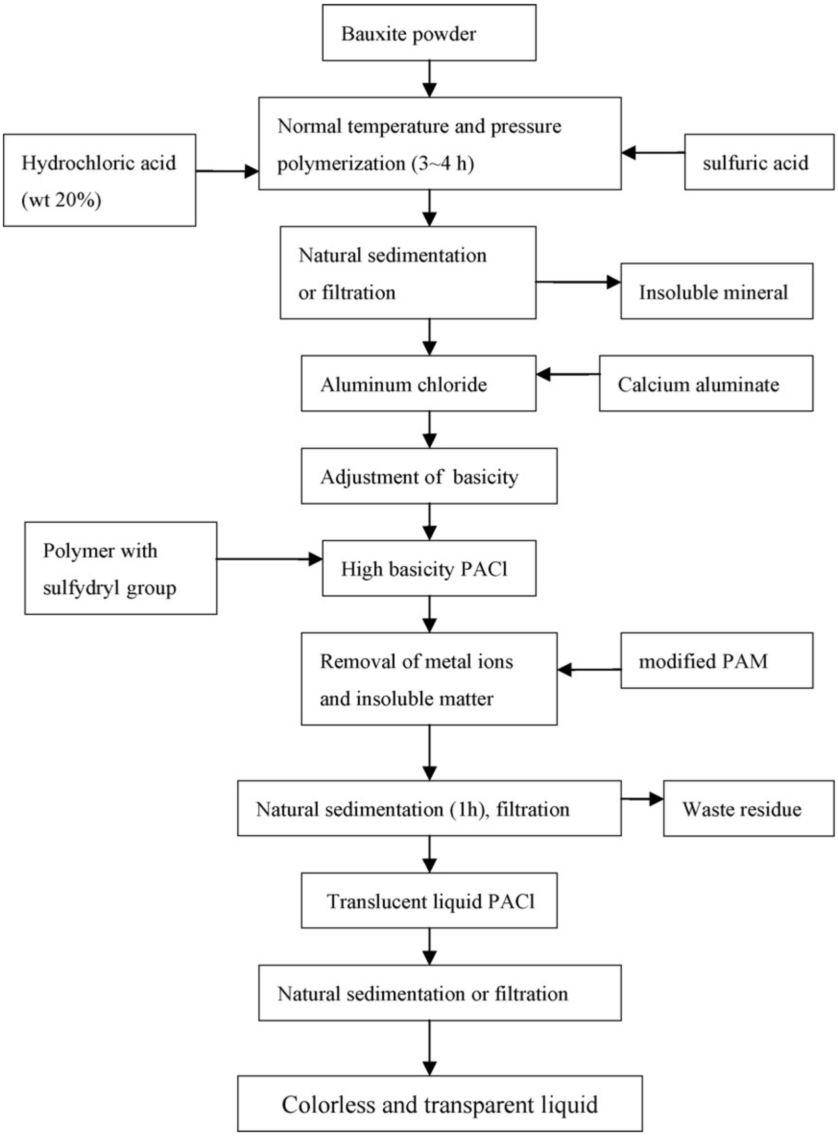


Fig. 1. The technical flow chart of preparation of high-purity PACl with bauxite and calcium aluminate.

The production process is as follows: first, quantitative hydrochloric acid (20 wt%) was added to the reactor, then the bauxite after grinding was added, the reaction was automatic exothermic reaction after heating to 70 °C. Then steam was added to the reactor until the temperature was raised to about 100 °C, the reaction lasted 3–4 h. After the finish of the reaction a small amount of water and calcium aluminate was added to the reactor and mixed for 2–4 h. PACl with given alumina content and basicity was thus prepared.

Polymers with sulfydryl groups and modified PAM were added into the above synthesized PACl, and then the solution was filtered after precipitation for 1 h. The colorless and transparent liquid PACl was obtained after further filtering. After such a treatment, the content of iron in PACl could be decreased to 100–300 mg L^{-1} from above 1000 mg L^{-1}. If the above-mentioned precipitation/filtration process was repeated more times, the content of iron can be reduced to below 100 mg L^{-1}.

The properties of the PACl products were determined according to the national standard methods. Alumina (Al_2O_3) content was determined by the titration with EDTA and zinc chloride; the basicity was measured by titration with potassium fluoride and sodium hydroxide; the insoluble matter was determined by drying-weighing method; and the content of iron and heavy metals ions was determined by ICP method.

3.2. Jar test procedures

The jar tests were conducted using a standard procedure involving a rapid mixing at 400 rpm for 1 min, a slow mixing at 35 rpm for 20 min and 30 min sedimentation. The supernatant after the sedimentation was withdrawn; part of which was used for the measurement of turbidity and the remaining was filtered by a 0.45 μm membrane for the measurement of UV_{254} absorbance. All experiments were duplicated in order to control the data quality and error range was less than ±5%. The standard deviation was presented in the results (tables or figures).

4. Results and discussion

4.1. Adjustment of basicity

The properties of high-purity PACl synthesized by bauxite and calcium aluminate are shown in Table 3.

Table 3
Properties of the high-purity PACl (samples A–C) prepared with bauxite and calcium aluminate in comparison with a conventional PACl.

Sample	Raw materials	Indicators						
		Al_2O_3 (%)	Basicity (%)	Mn^{2+} (mg L^{-1})	Cr^{6+} (mg L^{-1})	Cu (mg L^{-1})	As (mg L^{-1})	Pb (mg L^{-1})
Sample A	Bauxite 1 from He-Nan province of China	13.7	92.9	1.3	4.3	0.9	1.9	2.1
Sample B	Bauxite from Shan Dong province of China	10.3	91.6	1.7	3.7	1.2	0.54	1.9
Sample C	Bauxite 2 from He-Nan province of China	11.7	92.1	1.9	4.9	1.0	1.8	2.1
A conventional PACl	Bauxite and calcium aluminate	11.1	87.0	42.9	6.6	6.8	4.9	7.9
Chinese national standards		>10.0	45–95	–	<5	–	2.0	10.0

The basicity of high-purity PACl can be maintained at about 92% after the adjustment by calcium aluminate (Table 3), it has exceeded the national and international standards of PACl. It indicated that the method of increasing the basicity in liquid PACl is viable, calcium aluminate reacts with aluminum chloride (zero base) to generate a high basicity PACl (Eq. (2)). The sample A exhibited the highest aluminum content (13.7%) and the highest basicity (92.9%). Therefore, the bauxite 1 from He-Nan province which was used to prepare sample A is to be used as the raw material to prepare high-purity PACl.

4.2. *Removal of metal ions and insoluble impurities*

The results of removal of heavy metal ions by the method of adding polymer with sulfydryl groups were shown in Table 3. It indicated that the concentrations of heavy metals in high-purity liquid PACl (samples A–C) are all lower than the required values in the national PACl standard and were much lower than that in the conventional PACl. The content of heavy metals of sample A was the lowest.

The results of removing iron ion by adding polymers with sulfydryl groups were shown in Fig. 2. The content of iron in PACl (samples A–C) significantly decreased after precipitation treatment with sulfydryl group polymers, regardless of the type of bauxite used. In comparison with conventional PACl which has iron content ranging between 0.1% and 3%, the iron content in samples A–C was lower than 300 mg L^{-1} after sedimentation, indicating the added sulfydryl group polymers can effectively remove iron ions from PACl.

Chinese national standards have strict requirements for the insoluble matter in PACl. After the preparation, the mixture of liquid PACl was generally stored for 4–10 d, or was filtered by the plate and frame filter press in order to reduce the content of insoluble matter.

The insoluble matter of PACl can be reduced to 2% after sedimentation for 60 min by adding and mixing with sulfydryl group polymers. The transparent solution was obtained after filtering the above mixture solution.

It can be seen from Fig. 3, that the treated liquid PACl, samples A–C, possessed less color than that of the conventional PACl. Because the particle sizes of bauxite, calcium aluminate powder as the raw material were different, the sedimentation velocity of large particle is faster, it is easy to settle; the sedimentation velocity of small particles is relatively slow. If the liquid product is in high basicity and large viscosity, the sedimentation of small particles will be more difficult. It resulted in many insoluble matters in liquid PACl.

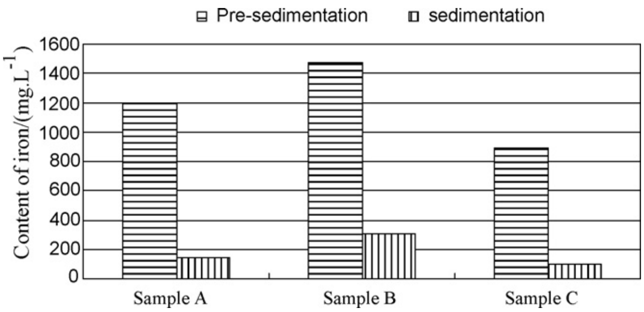


Fig. 2. The content of iron in PACl after adding polymers with sulfydryl groups.

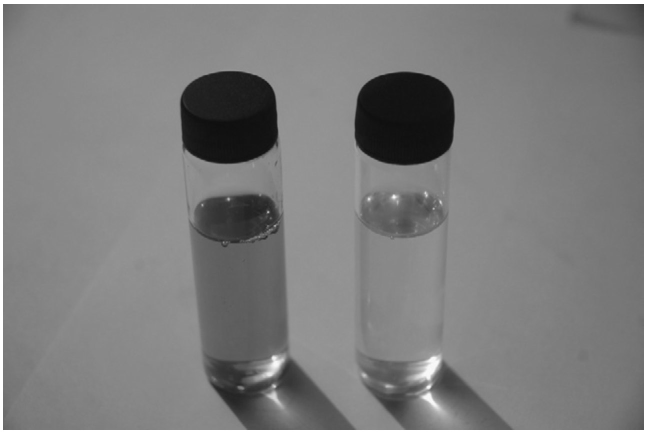


Fig. 3. Appearance of PACl samples. Left: PACl without treated by sulfide/PAM polymer (Abs_{977} = 0.325) and Right: PACl treated by sulfide/PAM polymer (Abs_{977} = 0.276).

Fig. 4 is the curve changes of insoluble impurities on sample A in the condition of 80 °C by adding a given amount of liquid modified PAM. After adding a small amount of polymer with sulfydryl groups and modified PAM, the sedimentation velocity is very quick. After sedimentation for 5 min and 10 min, the content of insoluble matter was only 1.4% and 0.5%, respectively. The content of insoluble matter can be reduced to 0.04% after 60 min. Transparent PACl solution can be obtained by sedimentation for another 1 h. If the temperature of the solution was raised to 60–90 °C, the solution

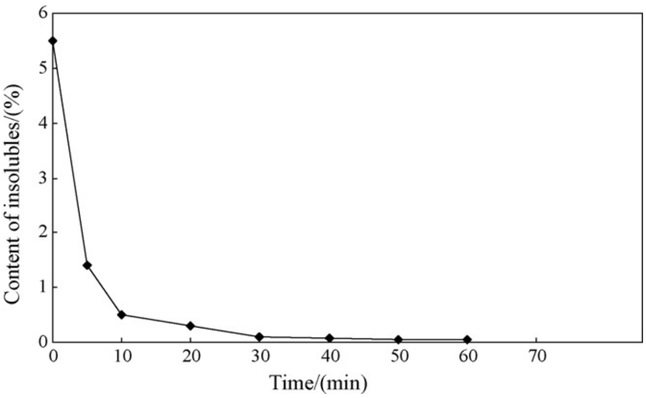


Fig. 4. The change of the insoluble substance in liquid PACl by polymer with sulfydryl group and modified PAM.

Table 4
Comparative water treatment performance of high-purity and conventional PACl.

Coagulants (PACl)	Test quality parameters	PACl doses as Al_2O_3 ($mg\,L^{-1}$)					
		0	2	4	6	8	10
Sample A (bauxite 1 from He-Nan province of China)	Turbidity (NTU) UV_{254} abs. (cm^{-1})	9.53 ± 0.03 0.051 ± 0.003	5.76 ± 0.03 0.048 ± 0.003	1.86 ± 0.03 0.043 ± 0.003	1.14 ± 0.03 0.039 ± 0.003	1.04 ± 0.03 0.037 ± 0.003	0.86 ± 0.03 0.036 ± 0.003
Sample B (bauxite from Shan Dong province of China)	Turbidity (NTU) UV_{254} abs. (cm^{-1})	9.53 ± 0.03 0.051 ± 0.003	7.39 ± 0.03 0.048 ± 0.003	3.57 ± 0.03 0.045 ± 0.003	1.97 ± 0.03 0.042 ± 0.003	1.61 ± 0.03 0.041 ± 0.003	1. 27 ± 0.03 0.036 ± 0.003
Sample C (bauxite 2 from He-Nan province of China)	Turbidity (NTU) UV_{254} abs. (cm^{-1})	9.53 ± 0.03 0.051 ± 0.001	7.97 ± 0.03 0.046 ± 0.001	3.10 ± 0.03 0.044 ± 0.001	1.80 ± 0.03 0.043 ± 0.001	1.56 ± 0.03 0.039 ± 0.001	1.11 ± 0.03 0.039 ± 0.001
A conventional PACl	Turbidity (NTU) UV_{254} abs. (cm^{-1})	9.53 ± 0.03 0.051 ± 0.002	8.88 ± 0.03 0.048 ± 0.002	4.82 ± 0.03 0.045 ± 0.002	2.60 ± 0.03 0.043 ± 0.002	2.14 ± 0.03 0.042 ± 0.002	1.60 ± 0.03 0.041 ± 0.002

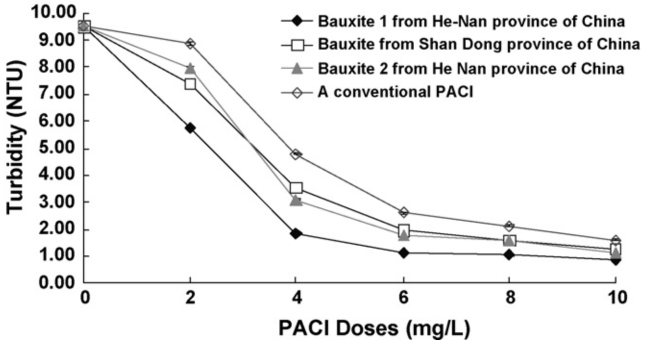


Fig. 5. Comparative turbidity removal performance with high-purity PACls and a conventional PACl.

will become less viscous, it will speed up the settlement of insoluble matter. The high temperature, the better separation efficiency. It should be noted that the excessive dosage of modified PAM will increase the solution viscosity, causing the precipitation more difficult, the removal performance of the insoluble matter was not as good as that under a natural sedimentation. As a high temperature favors to the removal of insoluble matter, the PACl solution was kept at 80–90 °C which also saves the energy in the process of drying. Therefore, high-purity PACl was obtained by adding polymer with sulfydryl groups and modified PAM in the precipitation process. It is then no need to change the current preparation process.

4.3. The comparative coagulation performance between high-purity and conventional PACl

Raw water in the Wu Lake was used as test water, the coagulation performance of high-purity and conventional PACl was comparatively studied.

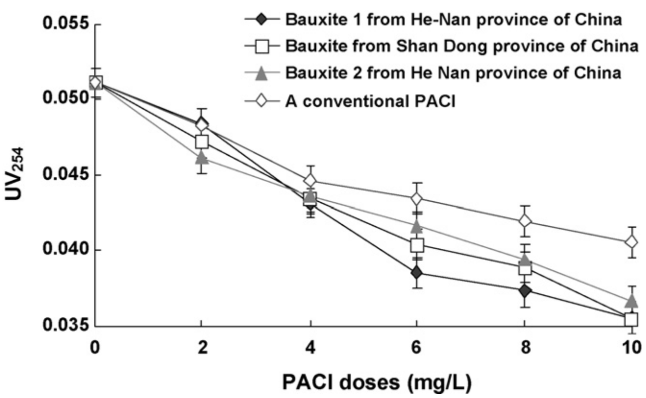


Fig. 6. Comparative UV_{254}-absorbance removal performance with high-purity PACls and a conventional PACl.

As shown in Table 4 and Figs. 5 and 6, for the whole dose range studied, three high-purity PACls removed more turbidity than conventional PACl, especially; high-purity PACl sample A reduced the turbidity to the lowest level, showing the excellent turbidity removal capacity (Fig. 5). On the other hand, at lower doses (<4 $mg\,L^{-1}$ as Al), the high-purity PACl did not show significant advantage for the UV_{254}-absorbance removal. However, when the dose was greater than 4 $mg\,L^{-1}$ as Al, the high-purity PACl showed great removal capacity; for example, for a dose of 6 $mg\,L^{-1}$, UV_{254}-absorbance removal by high-purity PACl sample A was 24.0% whilst that by the conventional PACl was 15.0% (Table 4 and Fig. 6). The UV_{254} removal performance was even much greater at high does (e.g., 10 $mg\,L^{-1}$). The observed performance could be explained by the charge effect. As shown in Fig. 7, for the coagulant dosages of less than 4 $mg\,L^{-1}$, the zeta potential of the test water treated by high-purity PACl, A, B and C, was similar to that treated by the conventional PACl. When the doses were in the range of 5–30 $mg\,L^{-1}$, the zeta potential of the test water treated by high-purity PACls increased faster than that with conventional PACl. The effect of the charge on the overall treatment performance with various coagulants has been studied previously, where both algal cells and natural organic matter were removed by polymerized coagulants at low doses and this was partly attributed to a strong charge neutralization caused by the property of the polymerized coagulants [3,4] and fast rate of chemical complexion [15,16]. Therefore, the observed results from this study are consistent with the published work.

4.4. The comparative production cost of high-purity and conventional PACl

The preparation process of high-purity PACl and conventional PACl is different as stated previously, where raw materials used and

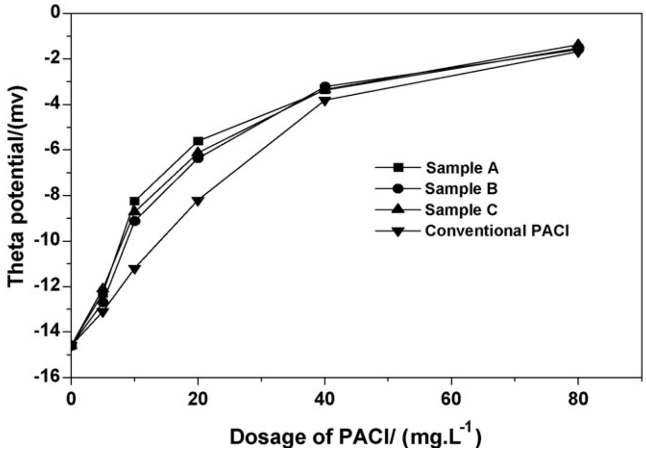


Fig. 7. The zeta potential in the test solutions with high-purity PACl, A, B and C, and conventional PACl.

Table 5
Cost of raw materials of preparing PACls.

Raw materials	Aluminum hydroxide	Bauxite	Calcium aluminate	HCl (30%)	Sulfydryl/PAM polymer	Production cost (RMB per ton)
Conventional acid dissolving of $Al(OH)_3$ process	100 kg/200 RMB		95 kg/95 RMB	350 kg/95 RMB		490
High-purity PACl-bauxite process		180 kg/72 RMB	95 kg/95 RMB	350 kg/95 RMB	50 RMB	342

the operating conditions are different. As shown in Table 5, $Al(OH)_3$ is relatively expensive than bauxite but sulfydryl/PAM polymer is cheaper. In addition to this, conventional acid dissolving of $Al(OH)_3$ process requires the operation performed at relative high pressure, 20,000–50,000 Pa, whilst the new bauxite process needs operated at ambient pressure; the energy consumption demand is lower than the acid dissolving process.

5. Conclusions

The preparation process of high-purity PACl with calcium aluminum and bauxite is developed, and the production cost is lower than that of acid dissolving of aluminum hydroxide. The metal ions can be effectively removed from the liquid PACl solution at both room and high temperatures. The preparation process is adapted to a wide range of conditions without cooling or heating, which can be beneficial to save the energy. The developed method has also avoided viscous flocs to be formed when using modified PAM. The most important feature of the new technology is that the preparation of high-purity and transparency PACl was conducted in the use of conventional natural sedimentation tank without changing the existing production process, and no additional equipment was required. The high-purity PACl prepared by the process possesses high-purity, low iron, low heavy metals, and few insoluble matters. The product meets the requirements of drinking water treatment and paper industrial application. The coagulation results also demonstrated that for the whole dose range studied, high-purity PACls removed more turbidity than conventional PACl. On the other hand, when the dose was greater than $4\,mg\,L^{-1}$ as Al, the high-purity PACl showed great UV_{254}-absorbance removal capacity; which was even much greater at high does (e.g., $10\,mg\,L^{-1}$). Relative low dose requirement of high-purity PACl indicates the potential of the reduction of operating cost.

Acknowledgments

The authors acknowledge the financial support of this study by the State Key Special Funds for Water Program, Ministry of Science and Technology, China (2008ZX07421-002). The research is also sponsored by Shanghai Science & Technology Commission and the Knowledge Transfer Awards Scheme of the UK Engineering and Physics Science Research Council (UK EPSRC).

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