Desalination and Water Treatment 317 (2024) 100178

Contents lists available at ScienceDirect

Desalination and Water Treatment

journal homepage: www.sciencedirect.com/journal/desalination-and-water-treatment/

Novel method of poly aluminum chloride extraction from kaolin and its application for wastewater treatment

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ARTICLE INFO

Keywords:
Natural materials
Kaolin
Poly aluminum chloride
Agricultural wastewater treatment

ABSTRACT

Most water resources are consumed by agriculture, particularly in developing countries. Consequently, the volume of wastewater from agriculture accounts for the majority of non-traditional water sources. This work extensively tackled the extraction, production, characterization, and application of leached poly aluminum chloride (PAC) from kaolin as a natural material to inhibit the pollution of water resources. The hydrochloric acid concentration percentage, pH, flux rate of HCl, and contact time were evaluated as factors controlling the output of leached PAC as (Al_2O_3). FTIR, XRD, and SEM were utilized to characterize the produced PAC as a novel material subsequently used in the treatment of agricultural wastewater. The empirical results revealed that the water treated with PAC™ complied with international standards and had no substantial variation in the dissolved ions. While employing PAC™, the maximum removal of turbidity, TSS, TOC, Nitrogen content and COD in agricultural wastewater reached 96.25%, 96.4%, 90%, 87.5%, and 75%, Whereas, the maximum removal of turbidity, TSS, TOC, Nitrogen content and COD in agricultural wastewater reached 95%, 95%, 80%, 84.4%, and 75%, respectively when using C-PAC. In conclusion, the use of PAC™ prepared from kaolin in agricultural wastewater treatment should be expanded as a low-cost and high-quality coagulant that protects the reverse osmosis membranes and mitigates the environmental impact of their disposal.

1. Introduction

Since 1960s, a novel class of water and wastewater treatment reagents known as poly inorganic coagulants (PICs) has been established as they are more cost-effective and efficient than most of conventional coagulants like aluminum or iron salts. PICs are currently being produced and used extensively in Japan, Russia, West Europe, and China for the treatment of water and wastewater [1–3]. IPFs of various types, such as those based on aluminum, iron, inorganic-inorganic composite flocculants, inorganic-organic composite flocculants, and multifunctional composite PICs, have been developed [4–6].

Conventional surface water purification involves a series of steps, including coarse and fine screening, early disinfection, particularly chlorination, coagulation, flocculation, sedimentation, filtering, and final disinfection [2].

Compared with the precursor Al salts, the pre-polymerized aluminum and iron salts have a variety of potential advantages in water treatment. Depending on the characteristics of the raw water, different

Abbreviation: APHA 2022, American Public Health Association 2022; C- PAC, conventional poly aluminum chloride; COD, Chemical Oxygen Demand; FC, Ferric chloride; FTIR, Fourier Transform Infra-Red; mg/l, mili gram per liter; NTU, Nephelometric unit; PAC™, extracted poly aluminum chloride; PAFSi, poly aluminum ferric chloride silicate; PAlFeClSi, poly aluminumferric chloride silicate; SEM, Scanning Electron Microscopy; TEM, Transmittance Electron Microscopy; TKN, Total Kjeldahl Nitrogen; TOC, total organic carbon; TSS, Total Suspended Solids

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https://doi.org/10.1016/j.dwt.2024.100178
Received 20 January 2024; Received in revised form 20 January 2024; Accepted 12 February 2024

degrees of these advantages were observed. Increased efficiency in the coagulation as a whole processes of flocculation and sedimentation enhanced performances in situations where hydrolysis and coagulation are frequently constrained for instance by low pH and alkalinity as well as by the existence of organic pollutants [7,8].

Due to their characteristics of chemical speciation, traditional coagulants and PICs operate differently. Hydrolysis of aluminum salt could result in different byproducts, ranging from monomeric to polymeric hydroxyl complexes, depending on the pH range of naturally occurring fluids. The positively charged $Al(OH)_3$(am) Al(OH)2+, Al(OH)2+, A_{l3}+, Al_2(OH)24+, $A_{l3}(OH)_4{}^{5+}$, and $Al_{13}O4(OH)24^{7+}$ (tridecameric polymer or "Al_{13}"), interact with the negatively charged colloids, leading to destabilization and aggregation of colloidal particles. Commonly, aluminum chloride has been neutralized to produce PACls. PACls typically include high levels of polynuclear aluminum hydrolysis products, such as Al_{13} [5,6,9].

As a polynuclear former, poly aluminum chloride (PAC) is a complex inorganic compound made of aluminum and progressive chlorination of the hydroxyl ion. $Al_x(OH)_yCl_{3x-y}$ or $Al(OH)_aCl\ _b(SO4)_c$ is the chemical formula for PAC, where (a + b + 2c) = 3 and a > 1.05. $Al(OH)_aCl_b$ is the chemical formula for PAC devoid of SO_4, with (a + b) = 3 and a > 1.05 [10]. PAC is a common ingredient in deodorants [11], papermaking [12], and wastewater treatment [13]. Over the past two decades, PAC and other pre-polymerized aluminum solutions have seen an upsurge in demand as coagulant agents. In comparison to the conventional aluminum sulphate, using PAC as a coagulant generally provides benefits such as denser and more compact sludge, high quality, less toxicity [14]. The preferred capability of PAC in water and wastewater treatment plants is the removal of colloids and suspended particles, metal ions, phosphates, organic materials, hazardous metals, and even color [15]. Currently, PAC is available on the market as either liquid or powder product [16].

Because PAC is more expensive than alum on the market, its manufacturing is economically advantageous [17,18].

Many authors stated that poly in organic coagulant is used in turbidity removal as well as biological oxygen demand (BOD) reduction [1,2,4,5,7].

For numerous reasons, including affordability, accessibility, sustainability and eco-friendly, the production of poly aluminum chloride utilizing kaolin and other natural materials is regarded as an innovative technology [19–23].

Therefore, the main targets of this study were dedicated on extraction of aluminum ions from kaolin as natural materials poly aluminum chloride as well as their characterization and applications in pre-treatment of agricultural wastewater, especially turbidity removal. Since the treatment process depends on the role of the poly inorganic coagulants (PIC) in precipitation, coagulation, and adsorption techniques, the treated effluent becomes perfectly suitable for trickle irrigation systems.

2. Materials and methods

Kaolin acquired from AL-Shark Company for treading as the precursor material. Hydrochloric acid (HCl) and sodium hydroxide (NaOH) were supplied by Al-Naser Company for intermediate chemicals, Egypt. All chemicals used are of merchant grade except sodium hydroxide which is a lab grade product. Double distilled water was utilized to prepare all solutions.

2.1. Preparation of poly aluminum chloride from kaolin

10 g of dried kaolin added to 35 ml of HCl (36%), sp.gr. (1.15), reflex for 3 hrs., at 150 °C, then, filtrate of the hot solution through filter paper (Whatman 40), the filtrate has animation as PAC ™. Factors affecting factors for production of PAC™ are studied as flows:

2.1.1. Effect of HCl acid concentration

the concentrations of 5%, 10%, 15%, 20%, 25%, and 30%, were tested under a constant (flow rate (1 ml/min), stirring velocity (500 rpm), contact time (3 hr), kaolin particle size (75 µm), kaolin acid ratio (1: 6) and reaction temperature (170° C).

2.1.2. Effect of reaction time

15, 30, 45, 60, 90, 150, 180, and 240 min were selected time intervals with a constant (flow rate (1 ml/min), stirring velocity (500 rpm), HCl concentration (30%), kaolin particle size 75-micron, kaolin acid ratio (1: 6) and reaction temperature 170° C.

2.1.3. Effect of flow rate

0.5, 1, 2, 3 and 4 ml/min were chosen as different flow rates with a constant (time of reaction is (3 hr), stirring velocity (500 rpm), HCl concentration (30%), kaolin particle size 75 µm, kaolin acid ratio (1: 6) and reaction temperature 170° C.

2.1.4. Effect of temperature

100, 120, 140, 150, 160, and 170° C as different reaction temperatures under a constant (flow rate (1 ml/min), stirring velocity (500 rpm), time of reaction (3 hr) kaolin particle size 75 µm, kaolin acid ratio (1:6) and acid concentration 30%.

2.1.5. Effect of particle size of kaolin

200, 150, 100, 75, and 63 µm under a constant (flow rate (1 ml/min), stirring velocity (500 rpm), time of reaction (3 hr), reaction temperature 170° C, kaolin acid ratio (1:6) and acid concentration (30%).

2.1.6. Effect of kaolin: HCl ratio

1:1, 1:2, 1:3, 1:6, and 1:12 with a constant (flow rate (1 ml/min), stirring velocity (500 rpm), time of reaction (3 hr), reaction temperature 170° C, kaolin acid ratio (1:6) and acid concentration (30%.) and reaction temperature 170° C.

2.1.7. Effect of agitation forces

steering speed of 100, 200, 300, 400, 500,600 and 800 rpm at a constant (flow rate (1 ml/min), time of reaction (3 hr) kaolin particle size 75-micron, kaolin acid ratio (1:6) and acid concentration 30%.

2.2. Characterization of poly aluminum chloride product (PAC ™)

PAC™ was characterized by X-ray diffraction (XRD) using Philips APD–3720 diffractometer with Cu Kα radiation (40 kV, 40 mA) and wavelength (λ) = 1.54 Å., Fourier transform infra red (FTIR) using Jasco FT/IR 460 plus spectrometer in the 400–4000 cm^{-1} of FTIR and 4000–7500 cm^{-1} of NFTIR wave number ranges as well as high resolution scanning electron microscope (JOEL JEM-100S). Turbidity determined using TL2300, HACH/Germany.

2.3. Analysis and Characterization of raw and treated wastewater

Raw and treated wastewater was characterized by total organic carbon using Teledyne. TKN using USA.VELP, Itali. Turbidity using LT2310, Hack, Germany using direct plus, hack, Germany. COD using DR 6000, Hack, Germany. pH and TSS were analysis according APHA 2022.

2.4. Preparations of working solutions of PAC ™

One gram of PAC dissolved in 1000 ml of double distilled water (each ml ≈ 1 mg/l), and then used in jar test experiments.

2.5. *Treatment of agricultural wastewater*

The prepared solution of PAC™ was employed for treatment of representative sample of agricultural wastewater (Tal-Naroz- Beni-Suef city, Beni-Suef Governorate).

Each single liter of raw water was mixed with 0.5 - 3 ppm PAC under continuous rapid stirring (200 rpm) for one-minute followed by slow mixing for 5 min (30 rpm) and 20 min standing time. The concentration of each parameter before and after treatment was measured by suitable equipment or according to APHA, 2022 [25], then the optimum conditions were recorded.

3. Results and discussion

3.1. *Factors controlling the productivity of Al_2O_3*

3.1.1. *Effect of HCl concentration*

The relation between the HCl concentration and the percentage of the produced Al_2O_3 was illustrated in Fig. 1. The result showed that as the power of acid increases, the productivity content of PAC increases. The optimum productivity limit was at HCl concentration of (30%). The optimum concentration of HCl was achieved as 30–36% [19].

3.1.2. *Effect of reaction time*

The effect of the contact time on the percentage of the produced Al_2O_3 was shown in Fig. 2. The results revealed that as the contact time increases, the Al_2O_3 production increases where the upper productivity limit was at time 3 hrs of direct contact between the reagents, whereas

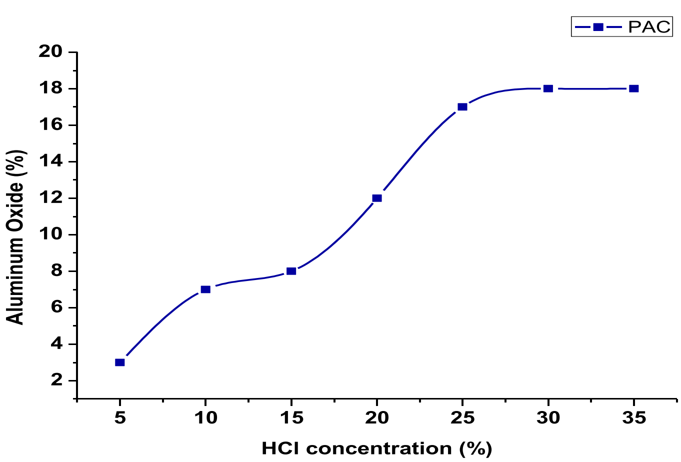

Fig.1. The effect of HCl conc. on Al_2O_3 productivity.

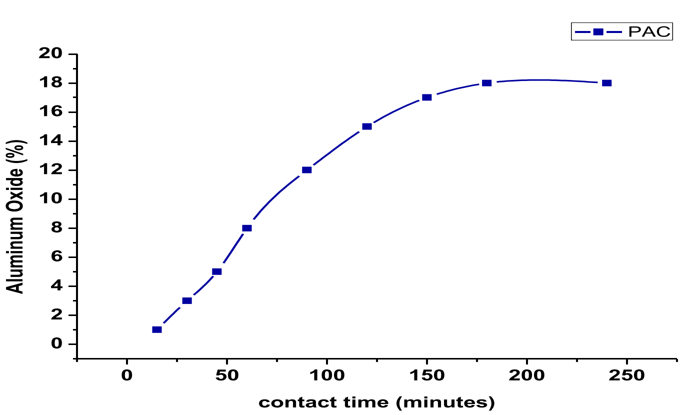

Fig.2. The contact time effect on Al_2O_3 productivity.

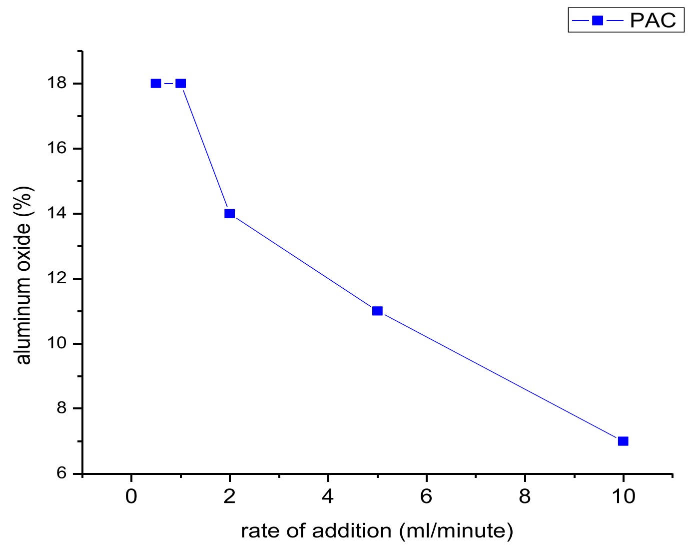

Fig.3. The effect of flow rate on Al_2O_3 productivity.

the optimum concentration of HCl was achieved as 2hrs [19] and 2.5 hrs [26].

3.1.3. *Effect of flow rate*

The flow rate has a significant effect on the production of $Al2O_3$ (Fig. 3). The result showed that with the increase in the flow rate the production of Al_2O_3 decreases. It became clear from the results that the best flow rate to obtain the highest production rate is 1 m/min.

3.1.4. *Effect of kaolin grain size*

The large, exposed surface area of the Kaolin enhances the reaction and then the production of Al_2O_3 increased (Fig. 4). The result revealed that as the grain size increases, the production of Al_2O_3 decreases where the best size for the optimum condition is 75 µm. Productivity increases as the particle size decreases due to the increase in the surface area of the kaolin.

3.1.5. *Effect of kaolin: HCl ratio*

Various proportions were tested to reach the best ratio to obtain the highest production rate (Fig. 5). The result showed that as ratio increases the production of Al_2O_3 increases, where the optimum condition was at 1: 6 ratios, whereas the optimum condition is 4.5 m/g as was shown [26].

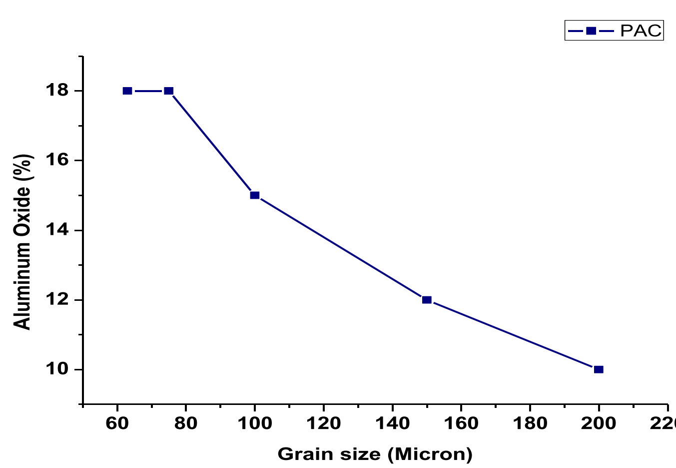

Fig.4. Effect of grain size(micron) on Al_2O_3 productivity.

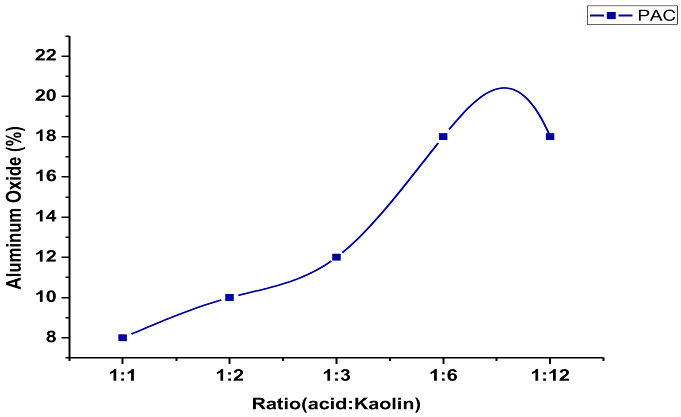

Fig.5. The effect of Acid: Kaolin ratio on Al_2O_3 productivity.

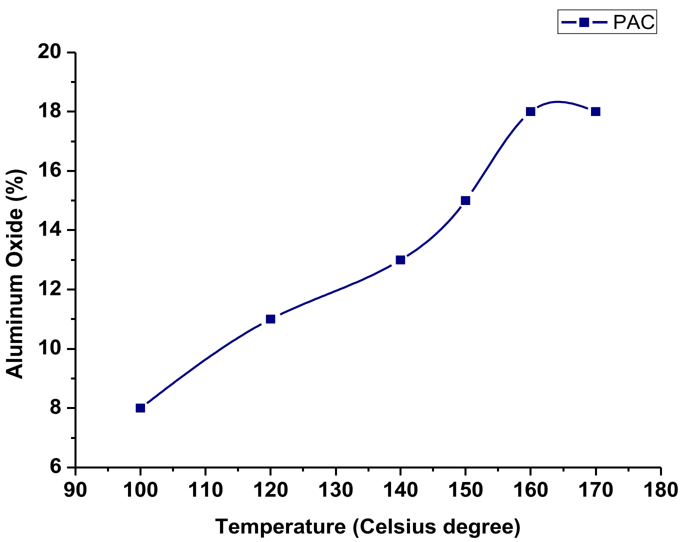

Fig.6. The effect of temperature on Al_2O_3 productivity.

3.1.6. Effect of reaction temperature

The endothermic nature of the reaction is clearly indicated (Fig. 6). The increase of temperature raises the production of Al_2O_3. The results indicated that as the increase in the reaction temperature, the produced percentage of Al_2O_3 increases. The convenient temperature for optimum production condition is 160°C, whereas the optimum temperature was recorded 80–100°C [26] and 105°C.

3.1.7. Effect of agitation forces

The stirring process is very necessary to complete the reaction and obtain the highest product (Fig. 7). The result indicated that as the RPM increases, the content of Al_2O_3 increases. The highest concentration of Al_2O_3 produced was at steering speed of 500 rpm, which causes more collision between acid and kaolin particles.

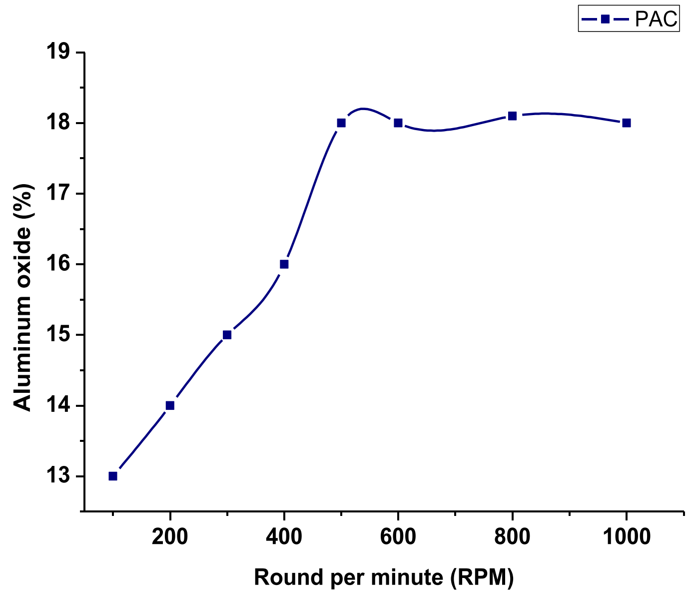

Fig.7. The effect of RPM on Al_2O_3 productivity.

Table 1
physicochemical characteristics of PAC versus commercial PAC.

Parameters	(PAC™)	Conventional PAC
Specific gravity(sp.gr)	1.40	1.40
Solid content %	50	52
Al_2O_3%	18.1	17.5 ± 1
Cl %	18	18 ± 1
Basicity	40	40-50
pH 5%	4.0	3.5-5
Al conc. %	9.2	9.0
Fe conc. %	1.01	0.01
Insoluble HCl % (max.)	0.1	0.1

Based on the above results of lab experiments, the optimum conditions which give the best concentration of Al_2O_3(18.2%) are 30% HCl concentration, 1 mil/min, flow rate, 3 hrs reaction time, 170 °C reaction temperature, 75 µm gran size of kaolin, and 500 rpm steering.

3.2. Characterization of PAC

3.2.1. Physico-chemical characterization

Table (1) shows that, there is no significant variation between the extracted PAC PAC ™ and the conventional one, except for the presence of iron in PAC ™ with a concentration up to 1.01%. The iron by the way enhances the coagulation process.

3.2.2. FITR characterization

FT-IR analysis of kaolin and PAC™ (Fig.8) displayed the stretching of the hydroxyl vibration bands at 3700 and 3600 cm^{-1}, which corresponds to the inner surface –OH stretching vibration of kaolinite. Band

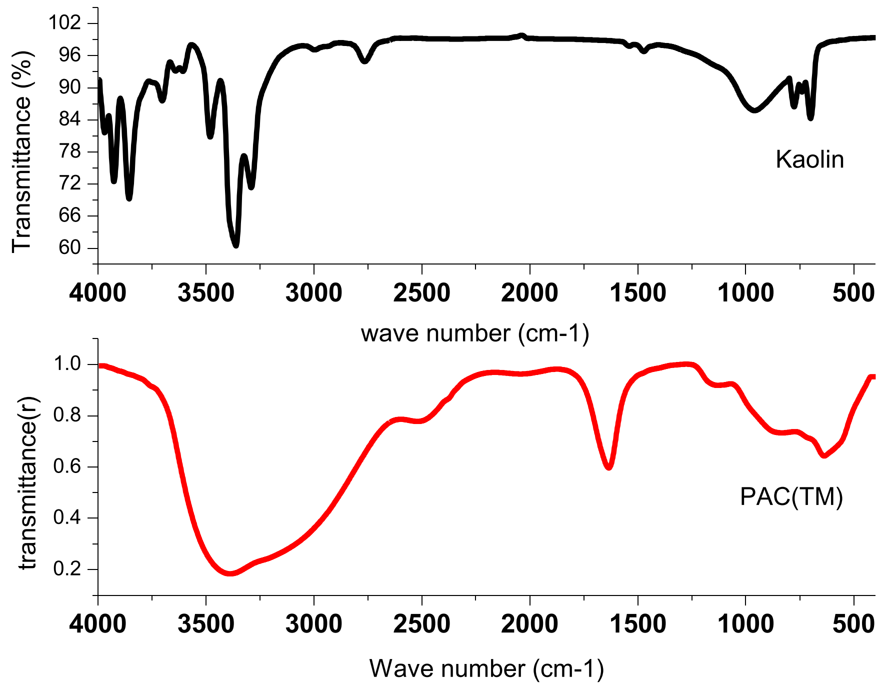

Fig.8. FTIR spectra of kaolin and PAC.

at 3400 cm^{-1} belongs to the stretching vibration of the outer-surface of the hydroxyl groups, indicating the presence of kaolinite in raw kaolin clay. Absorption band at 1627 cm^{-1} assigned for the -OH bending vibration and C=O stretching vibration as confirmed by [27–30]. The bands at 1110 cm^{-1} and 1026 cm^{-1} correspond to Si-O bending and stretching vibrations and attributed to the asymmetric stretching vibration of Fe–OH–Fe or Al–OH–Al [19]. Absorption band at 914 cm^{-1} corresponds to the Al-O bending vibration. Bands at 540 and 470 cm^{-1} represent to Al-O-Si skeletal vibration.

The spectrum exposed the characteristic bands for OH-stretching, H_2O bend Al-OH_2 bend, Al-O symmetric stretch, and Al-O bending at 3372, 1671, 1135, and 505 cm^{-1}, respectively whereas these transmittance bands confirmed by [4,5]. furthermore, there were two peaks at 770 cm^{-1} and 578 cm^{-1} for PAC™, which were attributed to bending vibrations of Fe– OH and Al–OH, respectively [31–33].

3.2.3. SEM characterization

Fig. (9a) illustrates the SEM micrograph of kaolin. The figure shows a well observation of hexagonal particles and the flaky texture of clays [24]. SEM micrograph obtained for PAC sample indicates the cave form, which refers to Al_{13} of PAC (Fig. 9b). The black spots could be iron oxide. SEM-EDAX also represents ratios of Al, Cl and O which compatible with elemental analysis of PAC [4,5].

3.2.4. XRD characterization

The kaolin clay contains quartz-SiO_2 (JCPDS number 085–0504) and kaolinite-$Al_2SiO_5(OH)_4$ (JCPDS number 029–1448) [27]. According to XRD analyses, raw kaolin clay is composed mainly of kaolinite and metal oxides [28,30]. X-ray diffractogram of poly aluminum chloride indicates the existence of a broad band around 2θ equal to 25°. The abroad peaks at in the range of 2θ from 5 to 25° are assigned for Al_{13} species in PACl–Al_{13} and the amorphous character of the sulphate salt [4,5] (Fig. 10).

3.3. Chemical treatment

3.3.1. Agricultural wastewater treatment

The variations of TDS, Turbidity, COD, TOC and Total nitrogen of agricultural wastewater before and after purification of the representative samples are displayed in Table 2. The variation and removal percentage of each parameter using extracted PAC and commercial PAC is also presented. It was found that the maximum removal percentage of turbidity, TSS, TOC, Nitrogen content and COD in agricultural wastewater reached 96.25, 98, 75, 90,96.25%, 98%, 75%, 90%, and 97%, respectively using PAC™, while the maximum removal conventional PAC reached 95%, 97%, 80%, 84.4%, and 75%, respectively. Now variation of Ph before and after treatment. The maximum COD removal efficiency of 95.4% at a dosage of 25 mg/l and pH of 7 using PAFSi [34]. The removal percentages of COD and TSS are 92%, and 93% using PAlFeClSi [4] whereas the removal percentages of COD and TSS are 99.7%, and 99% using PAlFeCl + Si [7]. The turbidity after purification with Al coagulants is within the permissible limits of Egyptian standards (1NTU), where as the rest of parameters are with limits according to Egyptian

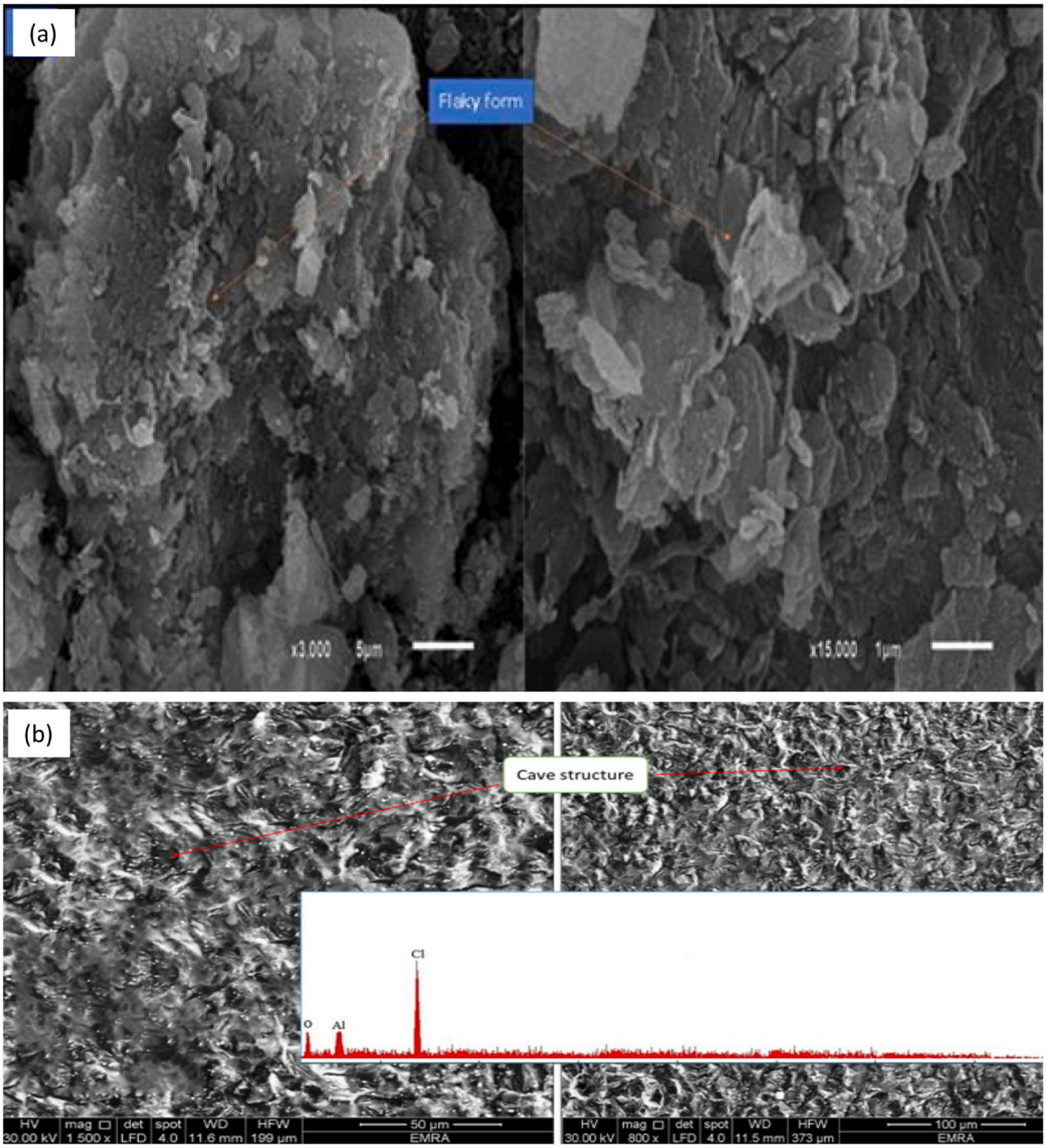

Fig. 9. SEM micrograph of kaolin and PAC.

minster of irrigation no 48/1982. From the results the efficiency of PAC ™ is more than conventional PAC.

From the calculation of the results, the cost of a ton of locally manufactured PAC™ reached $250, compared to $1000 for an imported PAC, while the cost of a cubic meter of treated water using locally manufactured PAC™ reached ($2.5 \times 10^{-7}$$). Numerous organic and suspended compounds found in seawater are known to foul reverse osmosis membranes, shorten their lifetime, degrade desalinated water quality, and increase losses. Consequently, incorporating PAC™ into the pre-treatment stage.

According to the efficacy of PAC ™ for removal of organic and suspended materials, PAC ™ can be used for pretreatment for desalination by reverse osmosis which leads to prevent of foul reverse osmosis membranes, extend their lifetime, improve desalinated water quality, and decrease losses of water. In the following diagram you can find a diagram showing the use of PAC™ in combination with reverse osmosis.

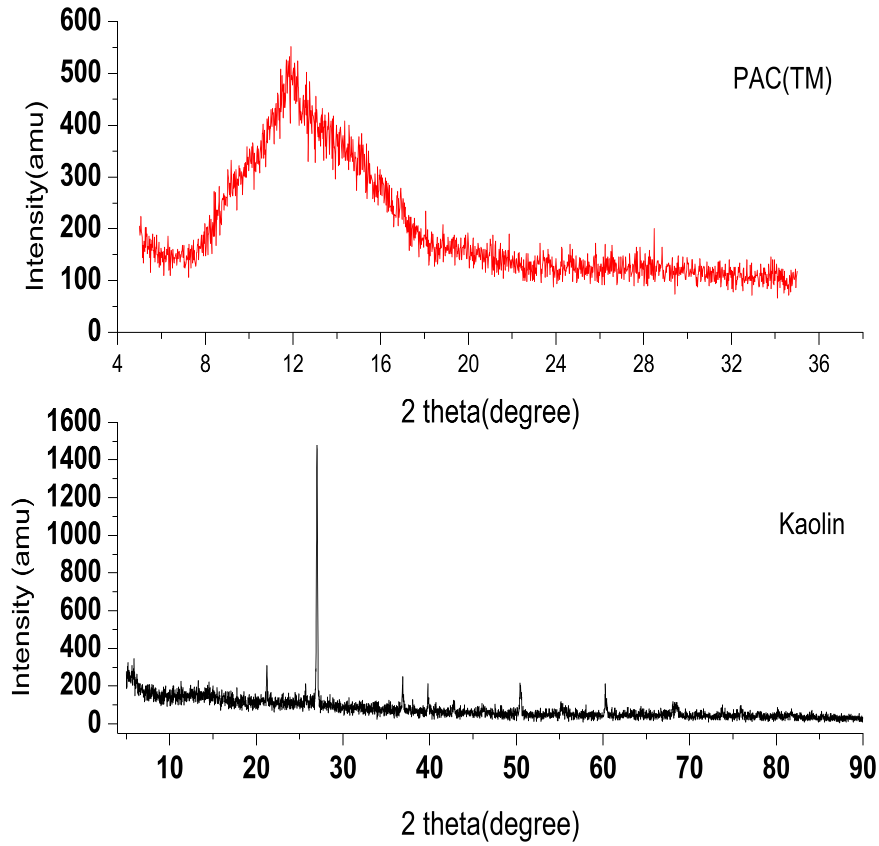

Fig. 10. XRD pattern of Kaolin and PAC TM.™.

Table 2
Variations of some physicochemical items of water before and after purification of for extracted PAC and commercial PAC.

Coagulant Parameters	Before Purification	Concentration after purification		Removal percentage		Permissible limits
		PAC™	Commercial PAC	PAC™	Commercial PAC	
Turbidity (NTU)	4	0.15	0.2	96.25	95	< 1
TSS (mg/l)	9	0.18	0.25	98	97	5
COD (mg/l)	12	4	4	75	75	5
TOC (mg/l)	10	1	2	90	80	5
TKN (mg/l)	16	0.5	2.5	97	84.4	1
pH	7.65	7.5	7.52	-	-	6.5-8.5

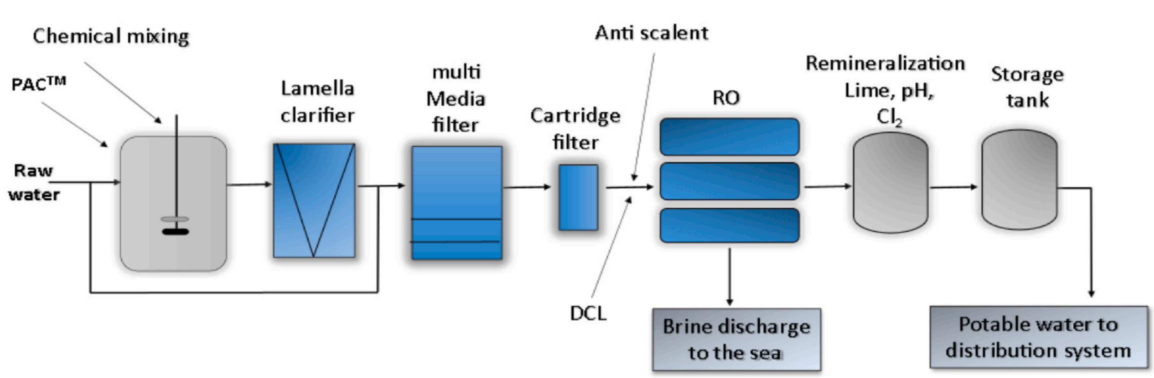

Diagram 1. The utlization of PAC ™ in combination with Reverse osmosis.

4. Conclusion

Based on the current study of lab experiments, the optimum conditions which give the best concentration of Al_2O_3(18.2%) are 30% HCl concentration, 1 mil/min, flow rate, 3 hrs reaction time, 170 °C reaction temperature, 75-micron gran size of kaolin, and 500 rpm steering, which is very close to the Egyptian standard for commercial PAC. PAC™ was used in removal of some pollutants such as turbidity, COD, TOC, TSS and TKN from agricultural wastewater. There is no variation of removal percentages in the case of PAC™ and C-PAC. The maximum removal of turbidity, TSS, TOC, Nitrogen content and COD in agricultural wastewater reached 96.25%, 98%, 90%, 97%, and 75% respectively using PAC™. Whereas, the maximum removal of turbidity, TSS, TOC, Nitrogen content and COD in agricultural wastewater reached 95%, 97%, 80%, 84.4%, and 75%, respectively as using C-PAC. Finally, this study found an economic and effective way to exploit the low-cost resources available in Egypt in large quantities to manufacture ecofriendly materials for the treatment of agricultural wastewater. The cost of a ton of locally manufactured PAC™ reached $250, compared to $1000 for an imported PAC, while the cost of a cubic meter of treated water using locally manufactured PAC™ reached ($2.5 \times 10^{-7}$$). Numerous organic and suspended compounds found in seawater are known to foul reverse osmosis membranes, shorten their lifetime, degrade desalinated water quality, and increase losses. Consequently, incorporating PAC™ into the pre-treatment stage. By adopting this method in a large scale, the agricultural drainage water will be reused safely as one of the important water resources in light of what Egypt is suffering from a decline in domestic water sources to meet the increasing demand.

Funding

This work was funded by the Science and Technology Development Fund (STDF) under Grant Number (46896).

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgement

The authors are thanks to Science and Technology Development Fund (STDF) for funding this project and Faculty of Earth Sciences, Beni- Suef University, Egypt.

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