

Review Article

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Poly-ferric sulphate as superior coagulant: A review on preparation methods and properties

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Abstract: Iron-based coagulants are widely used in wastewater treatment due to their high positively charged ion that effectively destabilise colloidal suspension, and thus contribute to the formation of insoluble flocs. Ferric chloride, ferrous sulphate, and poly-ferric sulphate (PFS) are examples of iron-based coagulants that are highly available, and are beneficial in producing denser flocs, thereby improving settling characteristics. This work aims to review the preparation methods of PFS and critically discuss the influence of these methods on the PFS properties and performance

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as a chemical coagulant for water and wastewater treatment. In polymeric form, PFS is one of the pre-hydrolysing metallic salts with the chemical formula $[\text{Fe}_2(\text{OH})_n(\text{SO}_4)_{3-n}]_m$ (where, $n < 2$, $m > 10$) and has a dark brownish red colour as well as is more viscous and less corrosive. PFS has an amorphous structure with small traces of crystallinity, containing both hydroxyl and sulphate functional groups. It has been applied in many industries including water or wastewater treatment which is also discussed in this study. It has the ability to remove pollutants contained in water or wastewater, such as turbidity, colour, chemical and biological oxygen demand, phosphorus, and others. This study also provides a review on the combination of PFS with other chemical coagulants or flocculants in the coagulation/flocculation process, and also flocs formed after a more stable treatment process.

Keywords: poly-ferric sulphate, coagulant, coagulation/flocculation, water, wastewater treatment

With the growth of the urbanisation and industrial sectors, water demand has increased as it is essential in many unit operations, thus resulting in a large amount of water discharge. Industrial wastewater may be of low quality due to its high pollutant content, which poses a major hazard to surface and underground water if not treated properly [1]. It is necessary to remove contaminants from wastewater before it is discharged into a watercourse or re-used or recycled for other purposes [2]. Particles or pollutants contained in wastewater come in a wide range of sizes, shapes, and densities, which affect their reactions in water [3]. There are various treatment methods used for water or wastewater including membrane technology [4], adsorption [5–7], filtration [8], ponding system, and others.

Another conventional wastewater treatment method is coagulation/flocculation, where particular chemicals are added to water [9–11]. It results in a physical change in the state of dissolved and suspended particles, allowing them to be removed more easily through sedimentation and filtration [12,13]. This method plays a crucial role in

adsorbing or connecting colloids, lowering the surface charge of colloids [1] to destabilise colloidal suspensions, and remove suspended particles [12,14,15]. Coagulation/flocculation is a key principle in colloid chemistry that allows fine particles suspended in water to clump together and form large flocs [16], which can then be separated from water [17]. As a result, it can reduce numerous pollutants effectively as well as reduce the concentration of chemical and biological oxygen demand (BOD), turbidity, colour, suspended solids (SS), heavy metal, oil and grease, and other organic matters.

Abujazar *et al.* asserted that it is crucial to determine the effectiveness of the coagulant function in specific conditions as coagulation is a complicated process considering numerous aspects [16]. There are three major types of coagulants/flocculants, namely, inorganic, organic, and composite coagulants [1,18,19]. They are also categorised as hydrolysing metallic salts, pre-hydrolysing metallic salts, and synthetic cationic polymers [13], as illustrated in Figure 1, which are more efficient than water-soluble hydrolysing metallic salts [20]. They tend to reduce colour concentration at low temperatures and produce less sludge [13].

Conventional metal coagulants, both polymeric and monomeric forms of aluminium (Al) and iron (Fe) salts, are commonly utilised in the treatment of water or wastewater [11,21,22]. However, aluminium salts are toxic to humans and living organisms [23]. As a result, the use of iron salts has expanded as they are more efficient in removing dissolved organic carbon (DOC) than aluminium-based salts [24]. In addition, iron-based coagulants (Figure 2) are another coagulant of choice, and have been widely used in water and wastewater treatment. Copperas or ferrous sulphate (Figure 2a) is one of the inorganic

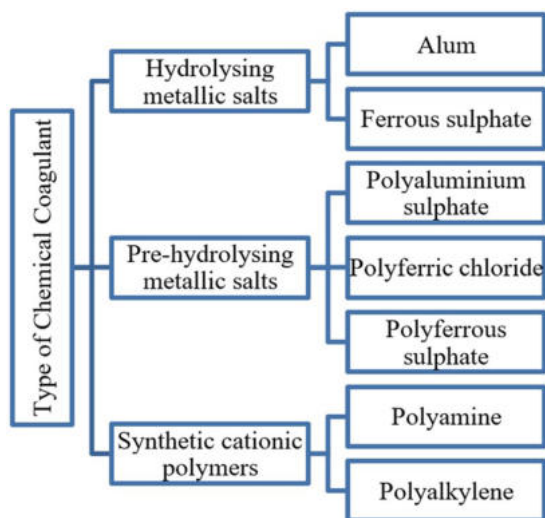


Figure 1: Chemical coagulant classification.

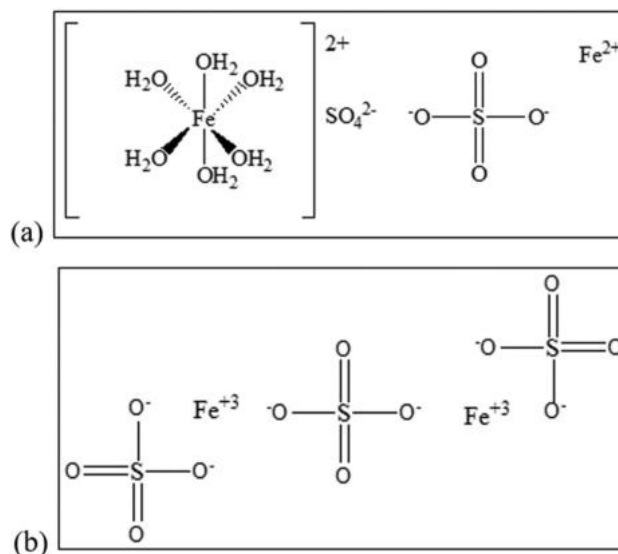


Figure 2: Chemical structures of (a) ferrous sulphate and (b) PFS.

coagulants in the form of iron-based salts that is commercially available, less expensive, and promotes high coagulation efficiency. This type of coagulant has widely been used in rubber processing effluent [25], palm oil mill effluent [11], groundwater [26], and other water or wastewater treatments. It can also control odour and thicken sludge, and can act as a dewatering agent for wastewater treatment [27]. Copperas is a by-product of the titanium dioxide manufacturing industry, which is formed during crystallisation *via* the sulphate process. However, a significant disadvantage of conventional coagulants is their inability to control the nature, temperature, and pH of the water to be treated, and occasionally it may worsen depending on the change in water characteristics.

Therefore, copperas will be partially neutralised with sulphuric acid under high concentration to form pre-hydrolysing coagulants with high molecular weight. Among them are poly-ferric sulphate (PFS; Figure 2b) [28,29] and poly-ferric chloride [30] which are frequently used to treat heavy metals in contaminated water or wastewater. They have received substantial interest due to their several benefits over monomeric versions as iron-based salts can operate in a wider pH range, are less sensitive to temperature [31–33], have lower residual ferric concentrations [31,32], and have low corrosiveness and excellent sewage treatment properties [34]. Gregory and Rossi evaluated the performance of several pre-hydrolysing coagulants for wastewater treatment, and observed that this type of coagulant provided faster flocculation and stronger flocs than hydrolysing salts at equal doses [35]. This is probably because these coagulants are pre-neutralised, have less effect on pH of water, and require less pH correction. In general, ferric ion is more favourable and

effective in removing colour, turbidity, and total carbon, and it has no toxicity concerns [36–38].

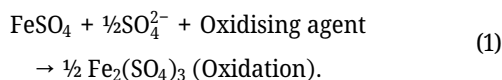
This work reviews and discusses the preparation methods of PFS and their influence on PFS properties. This work also reviews the combination of PFS with other chemical coagulants or flocculants in the coagulation/flocculation process, and also the flocs formed after the more stable treatment process. Overall, a new knowledge base will enhance the quality of the prepared PFS and the treated effluent in various industries.

1 Preparation methods and properties of PFS

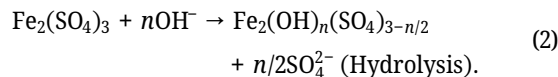
1.1 Preparation methods of PFS

There are several methods available for preparing PFS. Traditionally, PFS is prepared through a simple process of oxidation, hydrolysis, and polymerisation of ferrous sulphate in a highly acidic solution. Graham and Jiang patented the preparation and use of PFS (US Patent, Patent no.: 5785862) [39]. The patent described the PFS preparation process involving a catalytic oxidation method of a solution comprising H_2SO_4 and FeSO_4 under highly acidic conditions (pH 3), including hydrolysis, oxidation, and polymerisation as reported by Cheng, Jiang *et al.*, and Fan *et al.* [31,40,41]. Since then, numerous studies have been conducted on the synthesis and processing of PFS.

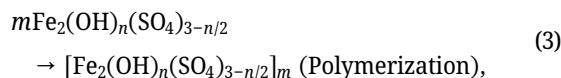
Generally, the reaction in the synthesis of PFS through the oxidation process is achieved by these three steps (as illustrated in Figure 3) [15,36,38,42,43]:



In the formation of basic salt $\text{Fe}_2(\text{OH})_n(\text{SO}_4)_{3-n/2}$, it requires 3/2 molar feed ratio of Fe^{2+} to SO_4^{2-} , where OH^- substitutes SO_4^{2-} [43] as shown in reaction (2) below:



And finally, the polymerisation reaction occurs in generating PFS as indicated in reaction (3) below:



where, m is the function of n ($n < 2$) [41]. Figure 3 illustrates the synthesis process of PFS [30,44].

In previous studies, the synthesis of PFS could be achieved through the addition of ferric sulphate solution to sodium hydroxide or sodium bicarbonate solution [43], and oxidation of ferrous sulphate to ferric sulphate in highly acidic conditions [45]. Nitric acid, hydrogen peroxide, sodium/potassium chlorate, oxygen, and oxygen-enriched air are the most frequently utilised oxidising agents [12,41,43,46]. It may be essential to find other oxidising agents for the synthesis of PFS that may induce high cationic charge in the coagulant, thus improving the charge neutralising capacity.

Several parameters of PFS synthesis have been reported in previous studies, namely, the temperature and duration of each preparation method stage, type of chemical used, and concentration of ferrous sulphate, as tabulated in Table 1. As mentioned above, seeking alternative oxidising agents may be necessary to synthesise PFS that may induce high cationic charge in the coagulant.

On another note, Ke *et al.* reported a modified method to synthesise PFS, as shown in Figure 4, which generated about 9.64–12.70% of total iron content and 0.11% Fe(II) [47]. The operating conditions are tabulated in Table 1.

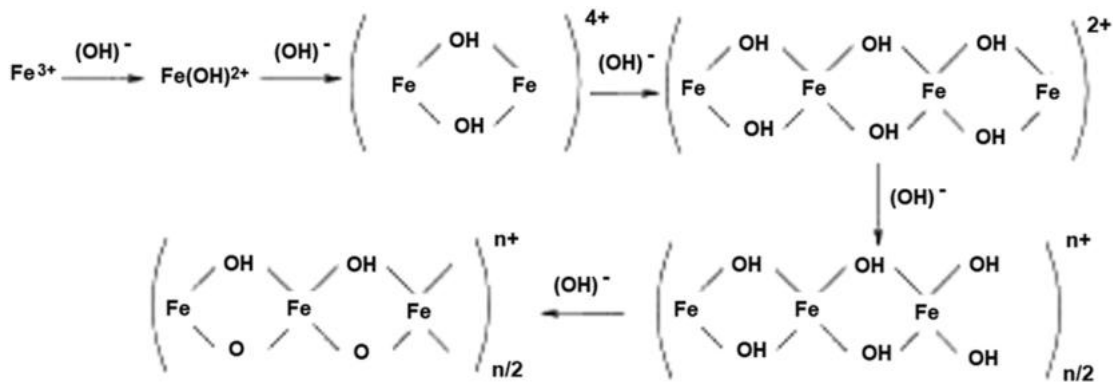
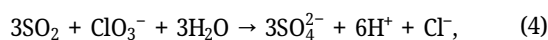


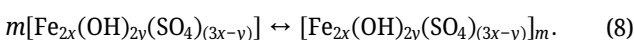
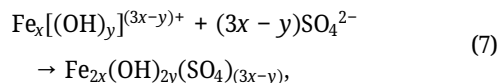
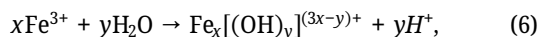
Figure 3: Hydrolysis and polymerisation processes of Fe(III) species.

Another PFS preparation method using microbes and organic waste was opted by Wang *et al.* for water or wastewater treatment [46]. The use of domestic *Thiobacillus ferrooxidans* resulted in high pH value basicity and total iron content [46]. Despite having a high degree of polymerisation, this method required a longer reaction time. On the other hand, Zhang *et al.* employed bipolar membrane electrodialysis to produce highly basic PFS [43].

The synthesis of PFS from residual sulphur dioxide (SO₂) derived from coal in fossil fuel energy plants has been reported by some previous studies [48,49], and as shown in Figure 5 by Zhang *et al.* [50]. This may minimise expenses and the presence of nitrates when using hydrogen peroxide [49]. The reactions are as follows:



Based on the reactions in equations (6)–(8), acid is required for the oxidation of Fe produced *via* the oxidation of SO₂ by sodium chlorate(III) as an oxidant [12]. Sodium chlorate is used to oxidise S(IV) to S(VI), and Fe(II) to Fe(III). The resulting acid and water in each equation play their role in the iron oxidation process, and subsequently for hydrolysis, sulphate inclusion, and polymerisation processes.



According to Butler *et al.*, the basicity of the produced PFS decreased with persistent SO₂ absorption after Fe(II) oxidation was completed due to the increase in free acid during the hydrolysis and polymerisation processes [12].

As previously mentioned, several preparation methods have been reported including the traditional process reactor by researchers as mentioned earlier. Several years ago, polymeric iron salts(III) were developed *via* different techniques [15,41,43,47]. Table 1 summarises the findings from previous studies for each preparation method described above. Different preparation methods resulted in different PFS characteristics. There are several important indexes in the synthesis of PFS including basicity (OH/Fe molar ratio) as the most important index, density, pH (1% sol), total iron content, and others.

From Table 1, the synthesised PFS contained high concentration of total ion, ferrous ion, and polymerised ion with high acidic conditions. Different concentrations were obtained since the chemicals and reaction conditions applied in synthesising PFS in the laboratory were different. It may be essential to find other oxidising agents for PFS synthesis that may

Table 1: Comparison of PFS and its characteristics according to the relevant literature

Ref.	Conditions	Properties									
		Fe _{total}	Fe(III)	Fe(II)	pH	[OH ⁻]/[Fe ³⁺]	Basicity	Density	Viscosity	Conductivity	z-potential
Catalytic oxidation of ferrous sulphate and sulphuric acid in acidic conditions											
Cheng [31]	<ul style="list-style-type: none"> Molar ratio 1:2 (sulphuric acid to ferrous sulphate) Aging: 5–7 h at 50°C 	-	160 g·L ⁻¹ Fe(III)	<1 g·L ⁻¹	0.56	0.4	-	1.48 g·mL ⁻¹	13.5 cp	-	-
Huang <i>et al.</i> [51]	<ul style="list-style-type: none"> 70 mL of 20% sulphuric acid 200 g FeSO₄·7H₂O, water bath at 60°C stirring speed 120 rpm. Added 5.4 g sodium nitrite and 30 mL of 30% H₂O₂, air of 2.0 L·min⁻¹ was fed with stirring for 2.5 h Drier at 100°C Aging: 45–75°C for 5–7 h 	11.3%	-	-	2.7	-	12.4%	1.45 g·cm ⁻³	-	-	-
Jiang <i>et al.</i> [40]	<ul style="list-style-type: none"> Aging: 45–75°C for 5–7 h 	-	160 g·L ⁻¹ [Fe ³⁺]	<1 g·L ⁻¹	-	0.4	-	0.5–1.0	-	-	-

(Continued)

Table 1: Continued

Ref.	Conditions	Properties									
		Fe _{total}	Fe(III)	Fe(II)	pH	[OH ⁻]/[Fe ⁺³]	Basicity	Density	Viscosity	Conductivity	z-potential
Li and Kang [52]	<ul style="list-style-type: none"> 0.05 mol ferrous sulphate 80 mL deionised water 0.0075 mol H₂O₂ Oxidation at 30°C for 30 min Aging 50°C for 2.5 h r: 0.2–0.5 aging: 50°C for 24 h 	3.3 wt%	-	0.0144 wt%	1.87	-	-	1.073 g·mL ⁻¹	-	-	-
Moussas and Zouboulis [53]	<ul style="list-style-type: none"> Based on the proposed method by Jiang and Graham 1998 5.59 × 10⁻³ M as Fe H₂SO₄, 96 wt% Oxidising agent: 65 wt% HNO₃ Hydrolysis 50°C for 30 min Aging 50°C for 7 h Oxidant: HNO₃ Oxidation: 2 h at 90°C Hydrolysis: 0.5 N of NaHCO₃ for 1 h at 50°C Aging: 2 h at 50°C 	0.74 mol·L ⁻¹	-	-	1.78	0.5	-	1120.71 g·L ⁻¹	-	-	-
Zouboulis <i>et al.</i> [15]	<ul style="list-style-type: none"> 5.59 × 10⁻³ M as Fe H₂SO₄, 96 wt% Oxidising agent: 65 wt% HNO₃ Hydrolysis 50°C for 30 min Aging 50°C for 7 h Oxidant: HNO₃ Oxidation: 2 h at 90°C Hydrolysis: 0.5 N of NaHCO₃ for 1 h at 50°C Aging: 2 h at 50°C 	47 g·L ⁻¹	57%	-	1.4	0.35	-	1132 g·L ⁻¹	-	46 mS·cm ⁻¹	-1.9
Zouboulis <i>et al.</i> [38]	<ul style="list-style-type: none"> Oxidant: HNO₃ Oxidation: 2 h at 90°C Hydrolysis: 0.5 N of NaHCO₃ for 1 h at 50°C Aging: 2 h at 50°C 	52.5 g·L ⁻¹	38%	-	1.5	0.25	-	1182 g·L ⁻¹	-	-	-
Modified PFS using reactor Cheng [36]	<ul style="list-style-type: none"> 40 g·L⁻¹ NaClO₃ (oxidant) and 16.7 g·h⁻¹ NaNO₂ (catalyst) 303–353 K 	-	>150 mh·L ⁻¹	<1 mg·L ⁻¹	-	0–0.6	-	-	-	-	
Ke <i>et al.</i> [47]	<ul style="list-style-type: none"> 0.4 mol·L⁻¹ ferrous sulphate pH 1.5 ± 0.5 250 rpm rotating speed Reaction time 7 h Reaction temperature 90°C 	9.64–12.70%	-	0.11% Fe	-	-	-	-	-	-	
Use of sulphur dioxide (SO₂) Zhang <i>et al.</i> [50]	<ul style="list-style-type: none"> Flue gas desulphurisation (as in Figure 5) Ferrous sulphate as adsorbent SO₂ as raw material Sodium chlorate as oxidant Use SO₂ Sodium chlorate as oxidant Atmospheric pressure Temperature 30–80°C 	25.34 wt%	-	0.029 wt%	2.64	16.04 wt%	-	-	-	-	
Butler <i>et al.</i> [48]	<ul style="list-style-type: none"> Use SO₂ Sodium chlorate as oxidant Atmospheric pressure Temperature 30–80°C 	≥9.0 wt%	-	≤0.1 wt%	2–3	-	8.0–12.0 wt%	≥1.3 g·cm ⁻³	-	-	

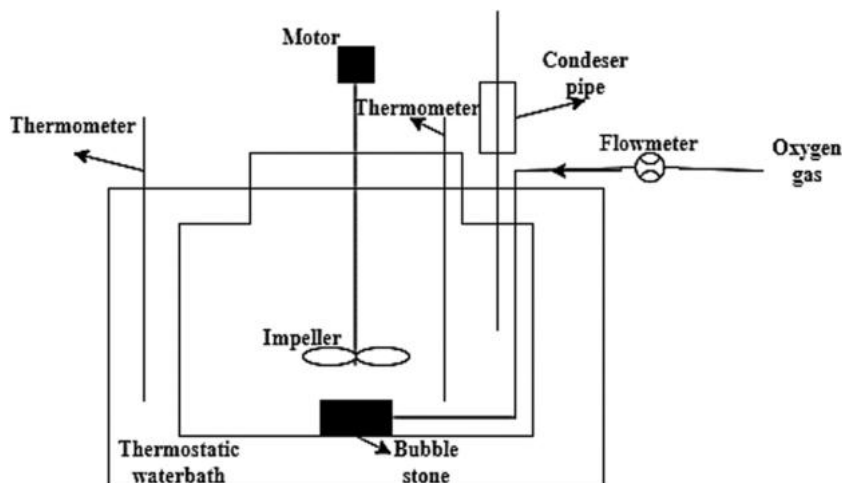


Figure 4: Preparation of experimental equipment for PFS synthesis.

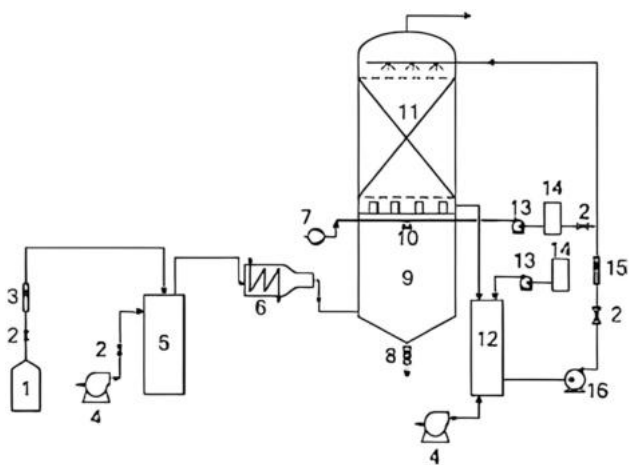


Figure 5: Schematic diagram of the system for SO_2 removal and PFS synthesis as proposed.

induce high cationic charge in the coagulant to remove pollutants contained in selected wastewater.

1.2 Characteristics of PFS

Generally, PFS is dark brownish red in colour [23,31], more viscous than other commercially available inorganic coagulants, less corrosive [31,41,54], and leaves less iron residue over a wide pH range [41,55]. PFS has the chemical formula

$[\text{Fe}_2(\text{OH})_n(\text{SO}_4)_{3-n/2}]_m$, where $n < 2$ and $m > 10$ [38,47]. Table 2 shows the content of analytical grade PFS based on several previous studies.

Referring to Tables 1 and 2, both the analytical grade and the synthesised PFS fully meet the standard even though they were prepared *via* different methods. Moreover, all synthesised PFS have low pH, total iron concentration, and stability that are within the national PFS range.

Apart from the properties discussed earlier, physical characteristics in terms of surface morphology, functional groups, elemental content, and others are also discussed. PFS has the morphology of an amorphous material [17,29,43], which forms many aggregates of various sizes and shapes [29,43,47] and smooth-surfaced [58] branch structure [59] characterised using Scanning Electron Microscopy (SEM). Therefore, Zhang *et al.*, concluded that the synthesised PFS is comparable to that of commercial PFS [43] as depicted in Figure 6.

According to Jia *et al.*, PFS displays a distinct cubic and globular rhombohedra shape [17]. It also has a curl-slice and is characterised by an even distribution of long tube-like structures on the surface [60]. Its particle size is between 5 and 10 μm [42] as shown in Figure 7, and in the range of 10–2 μm [47]. The unique morphology reveals the loose porous nature of PFS and may imply that it has a considerable capacity for adsorption [47].

Table 2: Properties of analytical grade PFS

Ref.	Fe_{total}	Fe(II)	pH	$[\text{OH}^-]/[\text{Fe}^{+3}]$	Density
Tetteh and Rathilal [56]	12.2% w/w	43.7% w/w	–	–	673 $\text{g}\cdot\text{L}^{-1}$
Shen <i>et al.</i> [57]	$25.0 \pm 5.0\%$ (w/w)	–	–	0.25	–
Huang <i>et al.</i> [51]	$\geq 11\%$	$\leq 0.1\%$	2.0–3.0	–	$\geq 1.45 \text{ g}\cdot\text{cm}^{-3}$

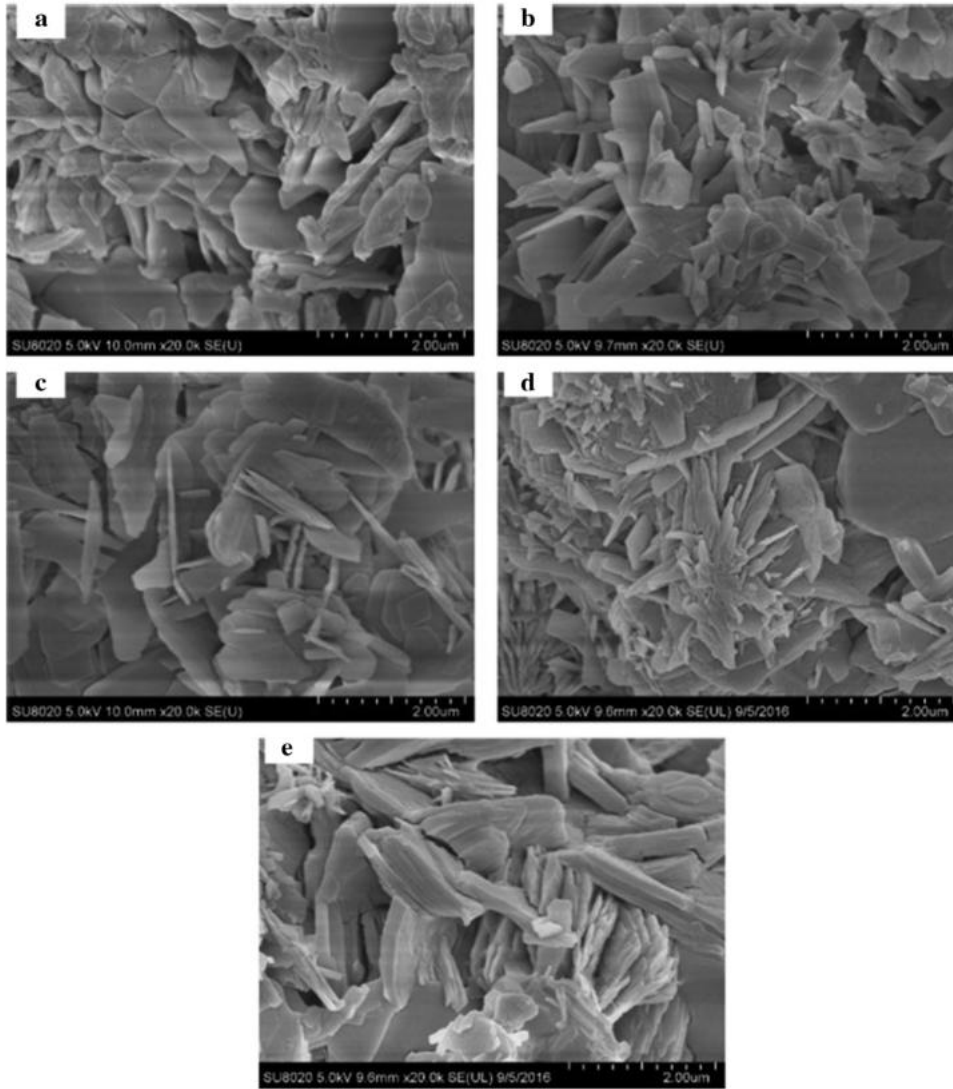


Figure 6: SEM images of (a) analytical grade PFS, and (b–e) synthesised PFS under different operating conditions.

For the X-Ray Diffraction (XRD) patterns, PFS shows an amorphous form with uncertain traces of crystallinity [43,48] at diffraction peaks of 2θ , and peak intensity of 17.51° , 28.57° , and 29.10° [17] as shown in Figure 8 reported

by Zhang *et al.* [43]. In other studies, PFS has been reported to have a crystalline structure at different peaks that varies from a largely amorphous structure to small traces of crystallinity [15,41].

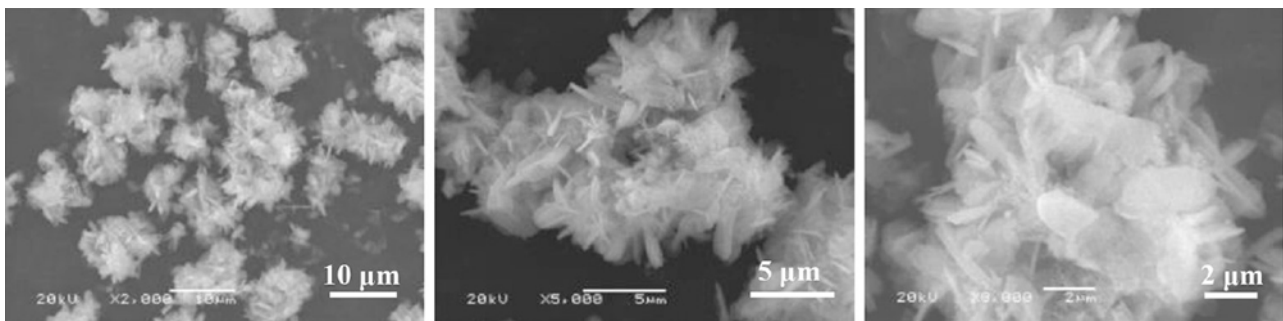


Figure 7: SEM images of PFS synthesised *via* oxidation, hydrolysis, and polymerisation using pure oxygen gas as oxidant.

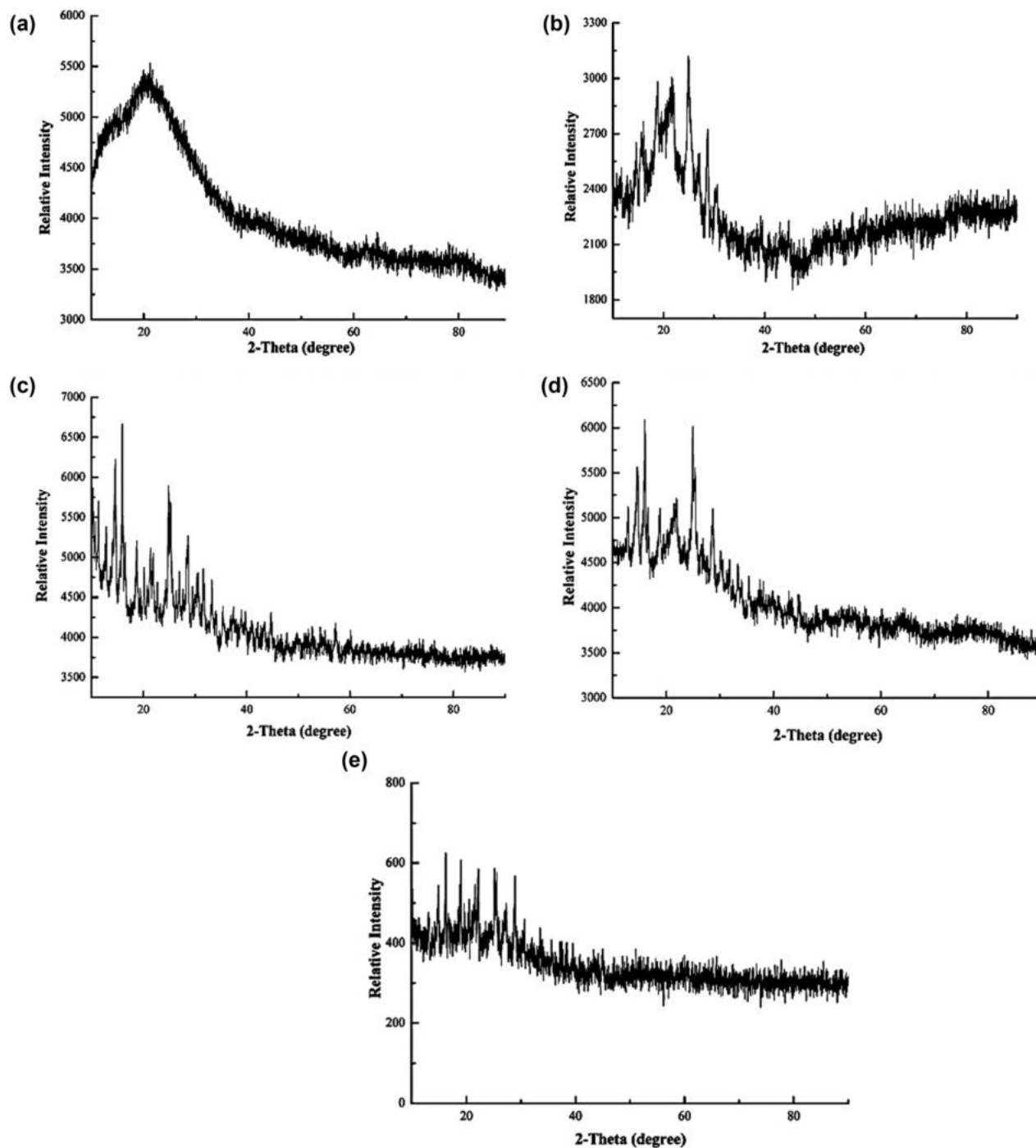


Figure 8: XRD patterns of (a) analytical grade PFS and (b)–(e) synthesised copperas under different conditions.

The chemical composition of the PFS sample is depicted in Figure 9 reported by Wei *et al.* [58], and the Fourier Transform Infra-Red (FTIR) analysis of PFS is summarised in Table 3. PFS contains both hydroxyl and sulphate groups as stated by Zhan *et al.* [61]. Previous researchers have

reported the stretching vibration of -OH group present at $3,300\text{--}3,500$ and $1,638\text{--}1,641\text{ cm}^{-1}$ bands referring to the bending vibration of absorbed water [17,43]. For the sulphate group, peaks in the region of $1,008\text{--}1,010\text{ cm}^{-1}$ and $1,160\text{--}1,120\text{ cm}^{-1}$ indicate the formation of polymer [17].

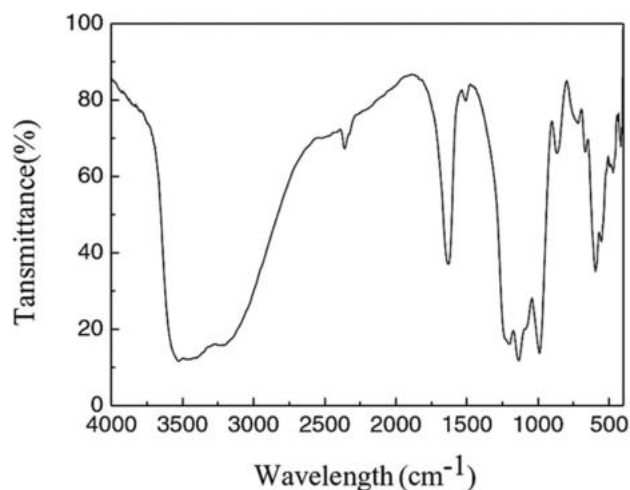


Figure 9: FTIR spectra of PFS.

2 Application of PFS as water coagulant

Numerous studies have been conducted on the use of PFS in controlling the release of arsenic [47], lowering the arsenic concentration that may dissolve in leachate [42], solidifying and stabilising phosphorus, fluorine, and other heavy metals significantly [62], removing silver nanoparticles [63], recovering organic matter [64], and improving acoustic agglomeration efficiency [65]. The application of PFS is extensive in various industries.

In dyeing wastewater treatment, PFS can reduce membrane fouling as it can decrease dissolved organic matter present in the wastewater prior to reverse osmosis treatment [66]. In addition, PFS is able to modify bubble in flotation treatment from distillery wastewater [67], and coagulating mechanism of humic acid [23,31]. In the removal of humic

acid by PFS via coagulation examined by Cheng [31], PFS performed well by decreasing the pH value of coagulation due to charge neutralisation between particles and the coagulant. At lower pH, more positively charged Fe(III) ions are present in the solution [31].

Furthermore, PFS has found its use in industries such as antimony removal in textile wastewater [62,68], phosphorus removal [69,70], arsenic removal [71–73], algae removal [74], iron reduction in phosphate-free conditions [75], and total aluminium removal [76]. It is highly efficient in removing pollutants in municipal wastewater [15], slaughterhouse effluent [3], and meat processing effluent [77]. Butler *et al.*, also reported that PFS performed better than traditional aluminium and iron salts in a wide range of applications [12].

Meanwhile, PFS is suitable for use in water treatment plant considering the chemical and physical features of surface water and seasonal pollution [78]. An illustration of coagulation/flocculation process is depicted in Figure 10.

When PFS reacts with water, it generates $[\text{Fe}_2(\text{OH})_3]^{3+}$, $[\text{Fe}_2(\text{OH})_2]^{4+}$, $[\text{Fe}^3(\text{OH})_6]^{3+}$, and other complex hydroxyl ions [36,47]. The presence of these polymeric species will enhance the charge neutralisation capability as inorganic polymeric flocculants have a higher cationic charge [79], and greater molecular weight; therefore, they are more effective at lower doses [80]. Higher cationic charge leads to increased surface activity and improves their capacity to neutralise the charge of suspended particles [38]. According to Zhang *et al.*, the presence of these ions [43] can decrease chemical oxygen demand (COD) [81], BOD, turbidity [49], and heavy metal [82] in water through hydrolysis, adsorption, and coagulation/flocculation processes. Dissolved organic matter may be removed from wastewater with excellent efficiency using PFS [83].

Table 3: FTIR analysis of PFS from previous studies

Group	Wavenumber		
	Jia <i>et al.</i> [17] Sodium chlorate was used as oxidant	Zhang <i>et al.</i> [43] Via hydroxide substitution using a membrane	Wei <i>et al.</i> [58] Commercial PFS
–OH groups	3,300–3,500 cm^{-1} and 1,638–1,641 cm^{-1} (referring to the bending vibration of absorbed water, polymerised and crystallised)	3,250–3,000 cm^{-1} and 1,628 cm^{-1} (referring to the stretching vibration of absorbed water or complexes)	3,381 cm^{-1} (stretching vibration of OH) and 1,634 cm^{-1} (bending vibration of –OH groups in the water molecule)
Fe–O–Fe	1,184–1,190 cm^{-1} and 508–511 cm^{-1}	1,000–980 cm^{-1} (band of Fe–OH)	–
Fe–OH–Fe	1,095–1,099 cm^{-1} (strong adsorption peaks)	–	466 cm^{-1}
Fe–O	–	–	598 cm^{-1}
S–O or the O–S–O bonds	1,008–1,010 cm^{-1} (strong adsorption peaks, indicating polymer was formed)	1,160–1,120 cm^{-1}	–
SO_4^{2-}	626–629 cm^{-1} (weaker bond)	680–610 cm^{-1} (weaker bond)	1,130 cm^{-1}

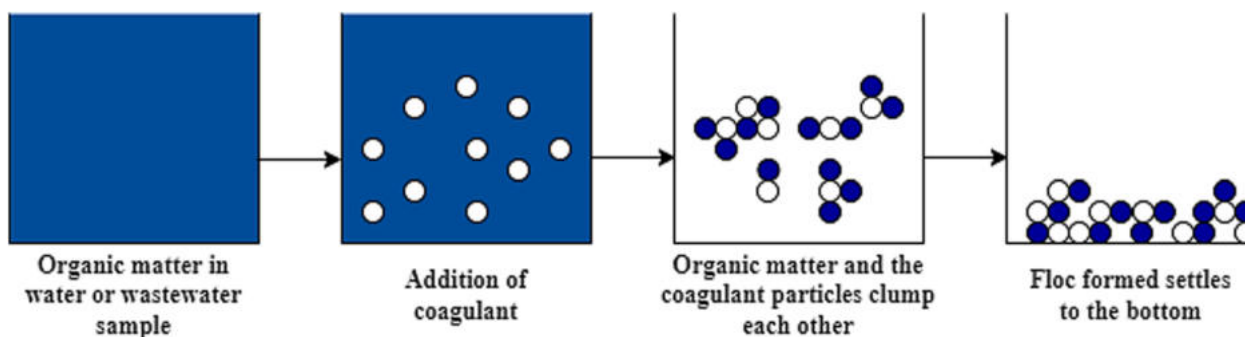


Figure 10: Illustration of coagulation/flocculation process.

The solubility of PFS depends on several factors, including the concentration of PFS, the pH solution, and the presence of other dissolved substances. At low concentrations and/or acidic pH, PFS is highly soluble in water. However, as the concentration of PFS and/or the pH of the solution increases, PFS can become less soluble and may form precipitates. This is because as the pH increases, hydroxide species may form which can combine with PFS to form insoluble precipitates. One study has found that the solubility of PFS decreased with increasing pH, with the solubility dropping from $32.5 \text{ g}\cdot\text{L}^{-1}$ at pH 2 to $0.65 \text{ g}\cdot\text{L}^{-1}$ at pH 11 [84]. This decrease in solubility was attributed to the formation of insoluble hydroxide species at higher pH values. Additionally, the presence of other dissolved substances such as phosphate ions or natural organic matter (NOM) can also decrease the solubility of PFS by causing it to form precipitates. Zhang *et al.* has investigated the effect of other dissolved substances on the solubility of PFS and found that the presence of phosphate ions and NOM could decrease the solubility of PFS, potentially leading to the formation of precipitates [85].

PFS acts by neutralising the charge on suspended particles and destabilising them through the formation of floc in coagulation mechanism. The hydrolysis of PFS leads to the formation of various species, including $\text{Fe}(\text{OH})^{2+}$, $\text{Fe}_2(\text{OH})_2^{4+}$, $\text{Fe}_4(\text{OH})_{12}^{4+}$, and $\text{Fe}(\text{OH})_3$ which can all contribute to the coagulation process [86]. The specific conditions under which hydrolysed soluble compound and $\text{Fe}(\text{OH})_3$ precipitate form will depend on the concentration of PFS and the pH of the solution [86]. At low concentrations and acidic pH, hydrolysed soluble compounds will be the dominant species. As the concentration of PFS and/or the pH of the solution increases, the solubility of PFS will decrease, leading to the formation of $\text{Fe}(\text{OH})_3$ precipitates [84–86]. The exact pH and dosage conditions under which this occurs will depend on the specific characteristics of the water being treated and the desired treatment outcomes.

In the coagulation/flocculation process, PFS can reduce the complex reactions resulting from the iron–salt hydrolysis, allowing easier and more precise control of the coagulation process [23]. PFS is more effective in removing different types of pollutants as summarised in Table 4. It is presumed that the removal of organics by PFS mostly occurs through adsorption, although charge neutralisation is weak in the near neutral region, where the removal is substantially higher [87]. Turhan and Turgut also claimed that PFS is an excellent adsorbent and decolouriser [88]. It is less sensitive to temperature and pH, and it works well over a pH range of 4–11 [47], and changes in dosage [36]. As stated by Xing and Sun, iron salts can coagulate in a wider pH range than aluminium salts, produce heavier flocs, and are less toxic if overdosed [37]. It often gives the best performance when the solution is acidic as revealed by Sahu and Chaudhari [89].

As tabulated in Table 4, PFS has the ability in removing numerous pollutants considering its characteristics which are discussed further in the subsequent sections. In general, PFS consists of high positive charge of iron(III) ions, and also its surface and charge neutralisation capacity. Therefore, these positively charged ions will attract organic matter or suspended particles with opposite charged ions to come into contact, neutralise, and form flocs. The neutralised particles form a larger and denser floc due to the bridging effect of the PFS. PFS polymers form bridges between the neutralised particles, resulting in the formation of larger aggregates. These aggregates can settle faster due to their increased weight [90]. As a result, pollutant present in water and wastewater may be removed.

To enhance the coagulation/flocculation process in wastewater treatment, the combination of different types of coagulants has been attempted. In recent studies and advances in coagulation/flocculation methods to treat polluted water, several researchers have combined inorganic–inorganic, organic–natural, and inorganic–organic coagulants for effective

Table 4: Application of PFS in different industrial effluent treatments

Type of wastewater	Experimental conditions	Removal efficiency (%)	Ref.
Municipal water	Dosage: 2 mg·L ⁻¹ Temp.: 28–30°C pH: 7.6–7.9	Turbidity (86.6%)	Butler <i>et al.</i> [12]
Groundwater sample	Dosage: 0.10 g·L ⁻¹	Arsenic (95%)	Cui <i>et al.</i> [71]
Secondary wastewater effluent	Dosage: 20 mg·L ⁻¹ pH: 7.0	DOC (25.8%) UV ₂₅₄ (32.6%) Endocrine-disrupting chemicals (30%)	Huang <i>et al.</i> [91]
Algae containing wastewater	Dosage: 20 mmol Fe ³⁺ L ⁻¹ pH: 8.5	Total cell (87%) DOC (63%) Turbidity (70%)	Jiang <i>et al.</i> [40]
Upland coloured water	Dosage: 6 mg·L ⁻¹ pH: 4.0	Colour (92%) UV ₂₅₄ (83.2%) DOC (80.3%)	Jiang <i>et al.</i> [92]
Sanitary wastewater	Dosage: 100 mg·L ⁻¹ pH: 6 Stir intensity: 250 rpm Mixing time: 90 s	Total phosphorus (78.99%)	Li <i>et al.</i> [70]
Landfill leachate	Dosage: 1.0 kg·m ⁻³	COD (63%)	Li <i>et al.</i> [93]
Landfill leachate	Dosage: 0.3 g Fe ³⁺ ·L ⁻¹	COD (70%) SS (93%) Turbidity (97%) Toxicity (74%)	Li <i>et al.</i> [28]
Landfill leachate	Dosage: 8 g·L ⁻¹ pH: 6.0	COD _{Cr} (56.38%) Colour (63.38%) Turbidity (89.79%) Humic acid (53.64%)	Liu <i>et al.</i> [94]
Textile wastewater	Dosage: 0.75 mM Fe pH: 5.8–6.2 Temp.: 25°C Stirring: 2 min, 200 rpm and 10 min, 80 rpm Settling time: 30 min	Antimony (77.6%)	Liu <i>et al.</i> [68]
Domestic water treatment	Dosage: 65 mg·L ⁻¹ pH: 4.0	Turbidity (100%)	Lloyd <i>et al.</i> [49]
Kaolin-humic acid	Dosage: 4 mg·L ⁻¹	Turbidity (98%)	Moussas and Zouboulis [60]
Synthetic wastewater	Fe/P molar ratio: 1.61 pH: 7.03 stirring: 3 min, 160 rpm and 5 min, 30 rpm settling time: 30 min	Total phosphorus (97.0%)	Ruihua <i>et al.</i> [69]
Water treatment	pH: 7.5	Silver nanoparticles (76%)	Sun <i>et al.</i> [63]
Oil refinery wastewater	Dosage: 50 mg·L ⁻¹	Turbidity (85%) Total suspended solid (74%) COD (83%) Soap oil and Grease (84%)	Tetteh and Rathilal [56]
Textile wastewater	Dosage: 500 mg·L ⁻¹	COD (41.4%) SS (5.7%)	Tianzhi <i>et al.</i> [83]
Water treatment	Dosage: 0.3 mM	Titanium dioxide nanoparticle (84%)	Wang <i>et al.</i> [95]
Water treatment	pH: 1.5 Dosage: 0.5 g·L ⁻¹	COD (70%) Colour (90%) Zn ²⁺ (99%)	Wang <i>et al.</i> [46]
Kaolin prepared wastewater	Dosage: 16 mg·L ⁻¹	Turbidity (90.36%)	Wei <i>et al.</i> [58]
Surface water	Dosage: 5 mg·L ⁻¹ pH: 7	Turbidity (90%)	Cheng [36]

(Continued)

Table 4: Continued

Type of wastewater	Experimental conditions	Removal efficiency (%)	Ref.
Water-based seed coating wastewater	Dosage: 1.5 g·L ⁻¹ pH: 8.0	Colour (96.8%) COD (83.4%)	Wen <i>et al.</i> [2]
Sewage wastewater	Dosage: 136 mg·L ⁻¹	SCOD: (69.4%) TP (92.9%) TN (45.0%)	Xing <i>et al.</i> [96]
Antibiotic fermentation wastewater	Dosage: 200 mg·L ⁻¹ pH: 4.0	Colour (66.6%) COD (72.4%)	Xing and Sun [37]
Water treatment	Dosage: 40 mg·L ⁻¹	Turbidity (85.1%)	Yang <i>et al.</i> [97]
Sewage wastewater	Dosage: 25 mg·L ⁻¹	UV ₂₅₄ (23.0%) COD (70.3%) TP (91.6%) PO ₄ -P (87.0%)	Zhao and Li [98]
Cotton pulp wastewater	Dosage: 800 mg·L ⁻¹ pH: 7.6	COD (94.85%)	Zhang <i>et al.</i> [50]
Paper making wastewater	Dosage: 500 mg·L ⁻¹ pH: 7.0	COD (82.20%)	
Purple dyeing wastewater	Dosage: 2 mL·L ⁻¹ pH: 7–9	Colour (76.18%) SS (92.23%) COD (71.43%)	Huang <i>et al.</i> [51]
Biologically pretreated molasses wastewater	Dosage: 5.5 g·L ⁻¹	COD (80%) Colour (94%)	Liang <i>et al.</i> [99]

pollutant removal [100]. Table 5 shows the performance of PFS combined with other chemical coagulants. Due to the growing market need for effective wastewater treatment, hybrid coagulants are also used during the coagulation/flocculation phase [16]. The combination of PFS with other chemicals or natural coagulants has high potential in treating water or wastewater. This is due to their greater effectiveness and cheaper cost compared to inorganic coagulants and organic flocculants [101].

For example, PFS-polyacrylamide [53], polyferric iron-based coagulant [102], poly-ferric-titanium-silicate-sulphate [103], polymer + PFS [57,60,104], polyferric silicate sulphate [52,105,106], PFS with flocculant [107], polyferric phosphatic sulphate [108], polymeric aluminium ferric sulphate [59], and polyferric-zinc-sulphate [58] also receive great attention in water or wastewater treatment. They have

shown increased efficiency in removing COD, turbidity, SS, phosphate, and other pollutants. In other applications, PFS was combined with Ca(OH) for the treatment of spent nuclear fuel debris [109], and calcified with CaCl₂ to dissolve inorganic phosphorus of sediment [110].

2.1 Effect of operating parameters on coagulation/flocculation process

The removal of NOM and tiny particles from drinking water is one of the crucial procedures involving the coagulation process [111]. The coagulation/flocculation process is an effective and simple method for water and

Table 5: Previous studies of composite polymeric iron sulphate for different industrial effluent treatments

Coagulant	Optimum dosage	Removal (%)				Ref.
		COD	Turbidity	SS	Phosphate	
PFS + flocculant (FO4440SSH)	(80 mg·L ⁻¹ + 6 mg·L ⁻¹)	–	–	90.51	–	Zhang <i>et al.</i> [107]
Polyferric zinc sulphate	12 mg·L ⁻¹	–	93.42	–	–	Wei <i>et al.</i> [58]
Polymeric aluminium ferric sulphate	45 mg·L ⁻¹	82.8	98.2	–	–	Zhu <i>et al.</i> [1]
Polymeric aluminium ferric sulphate	45 mg·L ⁻¹	83.6	–	–	–	Zhu <i>et al.</i> [59]
Polyferric silicate sulphate	75 mg·L ⁻¹	–	90	–	98–99	Moussas and Zouboulis [105]

wastewater treatment. Some of the factors that influence the effectiveness of the coagulation/flocculation process are the coagulant type and dose, pH, mixing speed, and time as well as temperature and settling time. Optimum conditions for the coagulation/flocculation process result in acceptable discharge limits and eventually can be discharged to nearby watercourses.

2.1.1 Effect of dosage

One of the imperative parameters in the coagulation/flocculation process is the dosage of coagulant. From Table 4, it can be concluded that different water or wastewater samples require different optimum coagulant dosages for treatment. This is due to the differences in organic matter or pollutant contents in the water or wastewater in terms of type and load of impurities. Alazaiza *et al.* described that there are three levels of conditions of coagulant dosage, namely, optimal dosage, under dosage, and overdosage, which are different from each other [112]. The optimal dosage refers to the condition when the coagulant aggregates colloidal particles to achieve a higher pollutant removal efficiency. Meanwhile, a low dosage promotes the formation of colloidal particles, but overdose may pollute the wastewater by increasing the level of pollutants as well as treatment costs [112].

Increasing the dosage will increase the removal efficiency [44]. According to Alazaiza *et al.*, high coagulant dosage results in increased coagulation/flocculation effectiveness for water or wastewater treatment [112]. Moreover, Butler *et al.*, reported higher turbidity removal at lower dosage of PFS [12]. In a study conducted by Saxena *et al.*, it was revealed that PFS only required a lower dosage to remove algae and algal-derived organic matter in a large scale [87]. A lower dosage of PFS is required for wastewater pretreatment process but depends on the source of wastewater [15,113].

2.1.2 Effect of initial pH value

Another important parameter in the coagulation/flocculation process is the initial pH value. In this process, pH needs to be adjusted as it affects the removal of pollutants in water and wastewater [27]. As summarised in Table 4, the optimum pH value in treating water or wastewater is in the range of pH 5–7. Cheng asserted that PFS has a wide range of optimum pH values [36]. Increasing the initial pH value will increase the removal of pollutants using poly-ferric based coagulants [103].

The removal of pollutants using PFS can decrease the coagulation pH owing to the availability of more positively charged Fe(III) ions with more neutral sites on the surface of humic acid at lower pH levels [31]. PFS can also coagulate at low pH through charge neutralisation [23]. When the pH value is increased, the complex formation of PFS by hydroxide ions will decrease, thus causing the pollutant adsorption onto the flocs of Fe(OH)₃ or Fe(OH)₄⁻ become difficult [23].

2.1.3 Effect of mixing speed and time

In the coagulation/flocculation process, preferable mixing speed and time can ensure that the coagulants used coagulate well with the suspended particles contained in the water or wastewater samples. In a study by Yukselen and Gregory, it was reported that rapid mixing was in the range of 70–75 rpm for 0.5–3 min, while slow mixing required a range of 30–150 rpm for 5–30 min to promote floc formation [114]. If the speed is too high, the flocs formed will break and result in a high turbidity content in the water [115]. It is necessary to identify the optimum mixing conditions for removing pollutants. A study on wastewater treatment revealed that the phosphorus removal was higher when PSF was subjected to rapid mixing at the coagulation speed and time of 160 rpm and 3 min, respectively, and slow mixing at 30 rpm speed and 5 min time [69]. The PFS disperses equally all over the solution and then collide with each other and form flocs [16].

2.1.4 Effect of temperature

Temperature directly influences floc size, strength, and ability to reassemble after a shear break-up [16]. The influence of temperature may raise the kinetic energy of iron particles, promoting further collisions with organic particles containing negatively charged ions, and therefore boosting the effectiveness of the coagulation process [116]. Low temperature is not ideal for flocculation [117], mostly because the viscosity of the solution increases as the temperature decreases [16]. An increase in temperature is favourable for the formation of larger flocs [118].

2.2 Production of flocs during coagulation/flocculation

The generation of flocs using PFS as coagulant is more stable [31], with floc breakdown occurring less often during

the growth phase, as stated by Zhao *et al.* [119]. This induces faster floc development resulting in faster settling rates [40], and it will be easily removed during the filtration process as less iron residue is formed [36]. The flocs formed from the use of ferric salts are larger than those using aluminium salts [120], and are more compact [68]. The amount of sludge formed during the sedimentation process is small in the range of 0–22 g·kg⁻¹ of total solid sludge [121,122].

Several studies have been conducted on the structure and elemental content of flocs formed using PFS as a coagulant. For instance, Liang *et al.* conducted SEM analysis and found that the flocs formed were mostly amorphous and random, and the coagulated flocs have uneven sheet-like forms of various sizes (Figure 11) [99]. Figure 12 displays the SEM image of precipitates form with larger aggregates compared to other iron-based coagulants, and the precipitates have the largest surface area, and thus can adsorb various metal ions such as C, O, Na, and other elements [90].

As discussed earlier, the flocs formed from PFS are stable and larger in size at optimum treatment conditions. Thus, PFS can be used as coagulant in treating various types of water or wastewater due to its effectiveness in removing pollutants, and the flocs formed will be easily disposed of.

3 Limitation and future works

PFS has been discovered for its efficiency in the removal of phosphorus, arsenic, algal, total aluminium, turbidity,

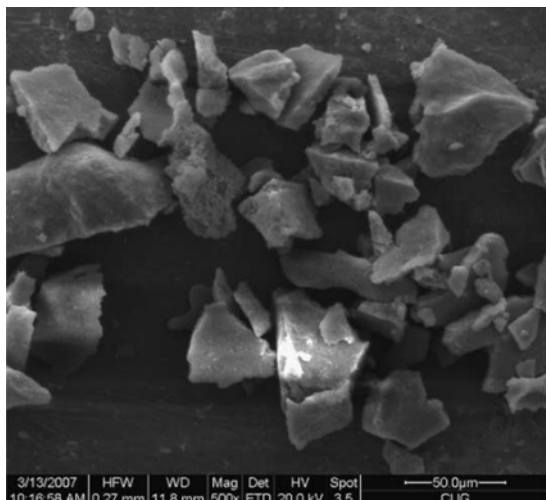


Figure 11: SEM image of the coagulated floc formed from PFS (synthesised *via* catalytic oxidation using sodium chlorate as oxidant) at 500× magnification.

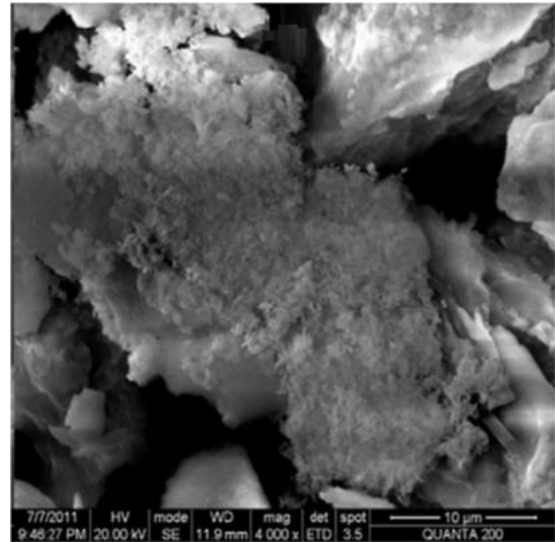


Figure 12: SEM image of commercial PFS precipitates at 4,000× magnification.

colour, and heavy metals. The synthesis of PFS from acid waste from the steel and dyestuff industries allows a significant reduction in production costs, which is another benefit of PFS application [36]. However, PFS has certain drawbacks resulting in a smaller market share [17]. Since PFS has high acidity, it may easily lower the pH of the water, further damaging the coagulation process, and possibly contaminating the equipment [17]. PFS-based water treatment processes may require the use of chemicals, such as pH adjusters and cleaning agents, which can have negative environmental impacts if not properly managed. Chemical use can contribute to water pollution and the accumulation of harmful chemicals in the environment [123]. The addition of PFS can affect its pH and potentially lead to changes in water quality that can negatively impact aquatic ecosystems and wildlife [124]. PFS-based water treatment processes require energy to operate, which can contribute to greenhouse emissions and climate changes. The energy required for water treatment can vary depending on the specific process and location but can be significant [125]. In terms of cost, it may vary depending on various factors such as supplier, quantity, and location. In 2023, the average cost of PFS ranges from \$0.70 to \$1.50 per kilogram, depending on the specific grade and quantity purchased [126]. The cost of using PFS in water treatment can vary depending on the specific application and system design. However, some general cost estimates for PFS-based water treatment processes including coagulation and flocculation were \$0.05–0.10 and \$0.10–0.30 per 1,000 gallons treated, respectively [127]. Overall, while PFS can be an effective coagulant for water and wastewater treatment application, its use

should be carefully considered in light of its economic and environmental impacts. More research is needed to synthesise PFS that is promising and acceptable for use in industry.

4 Conclusion

Polymeric iron-based coagulant or PFS is reviewed in this study in terms of its preparation methods and their effects on the coagulation/flocculation process for treating various types of water or wastewater. Based on previous studies, PFS exhibits an amorphous structure with small traces of crystallinity, and the presence of hydroxyl and sulphate as functional groups. PFS has the potential to be used as a coagulant as it contains higher polymeric species that can aid the coagulation/flocculation process. As a result, this polymeric form of iron-based coagulant is effective in removing pollutants in the wastewater. It is only required in small dosages, and can operate over a wide range of pH values depending on the nature of the water or wastewater. The use of this coagulant solely or in combination with other types of coagulants has a promising potential in the removal of pollutants.

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