

## A STUDY ON PREPARATION AND TESTING OF SOME FERRIC SALTS FOR WATER COAGULATION

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### ABSTRACT

In view of the increasing price of alum, efforts are being made towards local production of a suitable coagulant that could potentially compete with alum in terms of effectiveness and economy. To this end, four ferric salts namely; Ferrosol, Ferrichlor, Ferrifloc and Ferriclear were prepared and tested for coagulation effectiveness. The two salts that were most successful were analysed for cost effectiveness. The salts were prepared by investigating the electrolysis of dilute sulphuric acid. While Ferrosol and Ferrichlor had relatively low performance, Ferrifloc and Ferriclear were found to be almost as effective as alum at water pH 7.5 and 9 and more effective than alum at water pH 10 and 11 causing about 90% turbidity removal dosages of about 50 to 60 mg/l. Furthermore, Ferrifloc and Ferriclear do not depress the pH of water as much as alum. Ferrifloc was found to be superior to Ferriclear in terms of cost effectiveness. Production of Ferrifloc on a larger scale has been recommended towards making it have a significant potential for commercial exploitation.

**Keywords:** Ferric salts, coagulation, alum, cost effectiveness, turbidity and coagulant.

### INTRODUCTION

Potable water is one that can be used for drinking purposes with safety and satisfaction. Potable water standards provide a means for controlling, monitoring and planning water resources. This is to ensure that water does not contain impurities in hazardous concentrations which constitute grounds for rejection of water supply (Hammer, 1977). The purpose of water treatment is to convert the raw water to clear, sparkling and pathogen-free consumable resources. Water is accumulated in a reservoir, where some natural treatments occur. The raw water is pumped by a low lift pump into the aeration unit where dissolved gases are oxidized. The water then proceeds to the coagulation and flocculation unit where a coagulant is added to remove colloids through the processes of coagulation and flocculation. The agglomerated flocs are allowed to settle in a sedimentation basin after which it is filtered and chlorinated. The water is then stored in a clear water tank ready for distribution to consumers. Surface water usually requires coagulation, flocculation, sedimentation, filtration and disinfection prior to distribution (Davis, 2010). The first four steps only remove turbidity and therefore water quality characteristics related to dissolved materials are unchanged. The dissolved inorganic materials are often removed by

precipitation. The most common examples of the use of precipitation are for hardness removal and iron and manganese removal (McTighe, 1911).

Coagulation and flocculation are the most effective unit operations usually employed to remove microscopic impurities in water purification because coagulation aims at destabilizing the ever moving colloids and drives them together while flocculation agglomerates the particles which settles under gravity within the sedimentation basin or get trapped in the pore of filtration media (Sawyer and McCarty, 2005). Due to hydrolysis, coagulating agents have the disadvantage of modifying the physicochemical properties of the liquid (pH – conductivity). In large proportions, they lead to an excess of sludge; this is why use has been made of natural polymers (such as starch, potatoes, alginate, dextrin, gelatine, activated silica) alone or in combination with mineral salts (Degremont, 2009). The effect of Fe (III) and Al (III) on coagulation is not brought about by the ions themselves but by their hydrolysis product (Fair *et al.*, 1971).

Young (2015) stated that most modern surface water treatment plants use alum as a coagulant in their coagulation units. From a technical aspect, alum is suitable for vast majority water treatment; but the choice of coagulant to be used is also influenced by economic consideration and supply situations. Recently, there has been a continuous increase in the price of alum which is not unexpected since alum is being imported from advanced countries. Also there is no local substitute that can compete satisfactorily with alum. This is rather an unhealthy situation as it does not show that the country is taking steps towards self-dependency. In view of these facts, efforts are being made towards the local production of a suitable coagulant (Baghvand *et al.*, 2016).

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## **METHODOLOGY**

### **Experimental Setup**

The experimental setup consisted of an electrolytic cell with dilute sulphuric acid as the electrolyte. The electrodes were made of mild steel rods of length 280 mm and diameter 25 mm, they were centrally pierced 2 mm deep longitudinally to allow for good electrical contact. A glass tube was used to pass air from a gas cylinder into the solution for aeration. The depth of immersion and separating distance of the electrodes were maintained at 10.2 cm and 4.0 cm respectively and the solution was electrolysed in a one litre beaker using a soresen battery charger.

### **Factorial Design**

The factor which were considered to be relevant to the optimum performance of the resulting solution were identified; these included time of electrolysing, current, concentration of acid and aeration. Other factors such as the volume if the container, separation distance between the electrodes and surface area of the electrodes were kept constant for the purpose of this research.

A 2<sup>4</sup> factorial experiment was designed to determine the relative effect of the variables identified. Table 1 shows the experimental setting adopted. For each combination of

electrolysing time, current and concentration of acid; 800 ml of acid was electrolysed with the separation distance and surface area of the electrodes being kept constant. As the battery charger was switched on, the valve of the gas cylinder was opened or left closed depending on the desired level of aeration. After each experimental run, 200 ml of the resulting solution was oven dried and weighed. The resulting solution and the solution made from the solid were used to coagulate water samples. The percentage turbidity removal was calculated using the equation (1).

$$\text{Percentage turbidity removal} = \frac{\text{Initial Turbidity} - \text{Lowest residual turbidity}}{\text{Initial Turbidity}} \quad (1)$$

The responses measured included the weight of the solid formed, percentage turbidity removal by the electrolysed solution and percentage turbidity removal by the solid coagulant.

### Preparation of the Ferric Coagulants

1000 ml of one Normal sulphuric acid solution was prepared from 98% pure concentrated sulphuric acid solution and stored in a round bottom flask for use. Based on the results of the factorial experiment, 800 ml of N/30 H<sub>2</sub>SO<sub>4</sub> solution was electrolysed for 40 minutes. The solution was left unstirred throughout the process of charging (0.21 – 0.5 amps DC) and the voltage across the electrode was 4.4 V. the resulting product was named Ferrosol.

The 2<sup>4</sup> Factorial Experiment Setup

Experiment No.	Time of electrolysing (mins.)	Current (amps)	Concentration of acid (Normal)	Aeration
1	20	0.5	1/50	off
2	40	0.5	1/50	off
3	20	2.0	1/50	off
4	40	2.0	1/50	off
5	20	0.5	1/5	off
6	40	0.5	1/5	off
7	20	2.0	1/5	off
8	40	2.0	1/5	off
9	20	0.5	1/50	on
10	40	0.5	1/50	on
11	20	2.0	1/50	on
12	40	2.0	1/50	on
13	20	0.5	1/50	on
14	40	0.5	1/5	on
15	20	2.0	1/5	on
16	40	2.0	1/5	on

For the Ferrichlor, 40 ml of 1N H<sub>2</sub>SO<sub>4</sub> solution and 0.8 g of tropical chloride of lime (TCL, obtained from Opa dam) were mixed into a well stirred solution with about 200 ml of distilled water in a one litre beaker. More distilled water was added to the solution, making it up to 800 ml with continuous stirring. Other combinations by volume of 1N H<sub>2</sub>SO<sub>4</sub> solution and TCL were similarly prepared and tested. The resulting solution in each case was electrolysed and the solution was left unstirred throughout the process of electrolysing (0.21 – 0.5 amps DC) which lasted for 40 – 45 minutes. The voltage across the electrodes was 4.1 V.

For the production of Ferrifloc, 40 ml of 1N H<sub>2</sub>SO<sub>4</sub> solution was made into a well stirred solution with 200 ml distilled water in a one litre beaker. More distilled water was added to the solution, making it up to 800 ml with continuous stirring. The resulting solution in each case was electrolysed after which 0.8 g of TCL was added with continuous agitation. Other combinations of electrolysed acid and varying amounts of TCL were similarly prepared and tested. The current of the electrodes was 0.21 – 0.5 amps DC passing with the voltage across the electrodes being 3.3 V.

Furthermore, electrolysed acid was prepared as described in the preceding paragraph and 20 ml of sodium hypochlorite was added with continuous stirring. Other combinations of electrolysed acid and sodium hypochlorite were similarly prepared and tested. The resulting product was named Ferriclear.

### Coagulation of Water Samples

0.5 litre of synthetic raw water was treated with varying amounts of the ferric coagulants at different pHs. The synthetic raw water was prepared by dissolving 2 g of clayey laterite passing 300 µm sieve and rubbed between the palms (to obtain ultrafine colloidal particles) in 2 litres of tap water. The resulting suspension was stirred for 5 seconds and allowed to settle for 10 minutes after which it was diluted 3 times to obtain a turbidity of about 12.5 – 14.5 NTU. Other samples of the raw water were similarly treated with varying amounts of alum. Treatment in each case was done using the jar test method. Coagulation effectiveness was determined by measuring turbidity of the water samples before and after treatment using a Hach's model 2100A turbidimeter. Blank experiments on un-dosed water samples were run concurrently with dosed samples.

## RESULTS

### Factorial Design Experiment

Results of the factorial experiments are given in Table 2; here it can be seen that for weight, N/50 is not good with or without aeration, aeration gives higher weight and there is no great difference between 20 and 40 minutes. N/50 is also not good for percentage turbidity removal.

Results of the Factorial Experiment

Experiment No.	% turbidity removal by solution	% turbidity removal by solid coagulant	Weight of solid coagulant (g)
1	67.69	82.31	0.32
2	56.15	57.96	0.92
3	74.62	57.69	0.96
4	81.54	51.54	1.32
5	87.69	90.00	5.92
6	80.77	83.08	6.96
7	84.62	83.85	7.12
8	90.00	86.92	10.12
9	75.38	79.23	0.84
10	63.08	78.46	1.04
11	57.69	86.00	1.16
12	80.00	76.15	1.12
13	90.00	78.46	10.12
14	92.85	83.85	8.16
15	80.00	83.08	12.76
16	80.00	86.92	10.20

Table 3 shows the results of the analysis of the experimental data given in Table 2; this table shows that the effect of interaction between increasing time of electrolysis from 20 – 40 minutes and increasing current from 0.5A to 2.0A is to increase the efficiency of the solution by removing turbidity by 7.9%.

Effect of factors on efficiencies of the electrolysed solution			
Factor	% turbidity removal by solution	% turbidity removal by solid coagulant	Weight of solid coagulant
Time of electrolysis (1)	2.3*	5.72*	1.13*
Current (2)	1.94*	4.41*	1.30*
Concentration of acid (3)	16.14*	14.11*	4.62*
Aeration (4)	-0.60	3.61	1.40*
Interactions <sup>+</sup> 1, 2	7.9*	6.61*	2.30*
Interactions 1, 3	-0.60	3.07	0.95
Interactions 1, 4	1.29	4.74*	0.78
Interactions 2, 3	-0.60	3.81	0.65
Interactions 2, 4	-1.68	4.48*	0.44
Interactions 3, 4	0.37	3.93	0.45
Interactions 1, 2, 3	-0.83	0.84	0.25
Interactions 1, 2, 4	0.21	0.45	0.13
Interactions 1, 3, 4	00.60	0.61	0.18
Interactions 2, 3, 4	0.61	-0.45	0.11
Interactions 1, 2, 3,4	0.21	-0.84	-0.31

\* Significant at 95% confidence level

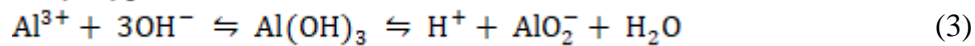
<sup>+</sup> signifies interaction between time of electrolysis (1) and current (2); similarly 1, 3, 1, 4 etc.

### Ferric Coagulants Versus Alum

When varying amounts of Ferrosol, Ferrichlor, Ferrifloc, Ferriclear and 1% solution of alum were used to treat 0.5l sample of water, it was observed that all the ferric coagulants were effective at pH 7.5 and pH 9. Alum was the most effective giving maximum turbidity removal of approximately 90% at a dosage of 80 mg/l. this was closely followed by Ferrifloc and Ferriclear, both removing maximum turbidity of approximately 88% at the a dosage of 50 mg/l. Ferrichlor was next at approximately 75% turbidity removal at 125 mg/l while Ferrosol removed about 52% turbidity at a dosage of 120 mg/l. The low value of turbidity removal achieved by Ferrosol as compared to other ferric salts and alum could be attributed to the ferrous ion,  $Fe^{2+}$ , it contained as revealed by the addition of sodium hydroxide to a small portion of Ferrosol. Ferriclear and Ferrifloc showed better performances than alum at pH 10 and 11 (Vok *et al.*, 2016). It was observed that both of these coagulants removed about 89% turbidity at 50 – 60 mg/l while alum achieved a turbidity removal of 80% and 60% at pH 10 and 11 respectively; this was achieved at a dosage of 100 mg/l at both pH.

Table 4 shows the effect of varying dosages of Ferrifloc, Ferriclear and alum on turbidity removal and on pH of treated samples at pH 7.5. From the table, it can be seen that one glaring advantage of the two most successful synthesized product, Ferrifloc and Ferriclear, is that they do not depress the pH of treated water as much as alum does. The use of alum at water treatment plants is always complemented with the use of a pH booster such as lime ( $Ca(OH)_2$ ) which is a source of added expense and operational steps. Ferrifloc and Ferriclear will require little or no complementary pH booster depending on the pH of the raw water (Eaton *et al.*, 2015).

This can be due to the production of hydrogen ion that depresses the pH of water to a certain extent when aluminium or ferric salt is added to water. Insoluble ferric hydroxide dissolves to a limited extent to produce  $\text{Fe}^{3+}$  and  $\text{OH}^-$  ions while insoluble aluminium hydroxide exists in equilibrium with  $\text{Al}^{3+}$  and  $\text{OH}^-$  ions on one hand and with  $\text{H}^+$  and  $\text{AlO}_2^-$  ions on the other hand (Equation 2 and 3). The extra hydrogen ion introduced into water due to the acidic characteristics of aluminium hydroxide might be responsible for further depression in pH of water treated with alum.



Effect of varying dosage of Ferrifloc, Ferriclear and alum on turbidity removal and pH of treated water samples at pH 6.5

Coagulant	Dosage (mg/l)	Blank reading (NTU)	Residual turbidity (NTU)	% turbidity removal	Final pH
Ferrifloc	10	13.6	9.4	31.0	7.4
	20	12.8	5.8	55.0	7.3
	40	13.0	2.0	82.5	7.0
	50	13.5	1.7	87.2	6.9
	60	12.8	1.9	84.5	5.7
	75	12.5	2.4	81.0	6.2
	100	13.0	3.4	74.5	5.9
Ferriclear	10	12.5	9.5	24.0	7.4
	20	13.0	7.2	44.5	7.3
	40	13.5	3.1	77.2	7.1
	50	13.5	1.9	85.5	6.9
	60	12.5	2.1	83.0	6.7
	75	13.6	3.0	77.5	6.3
	100	13	3.5	73.2	6.0
Alum	20	13.5	6.7	50.4	7.4
	40	14.0	2.2	80.3	7.0
	60	13.5	1.6	88.0	6.6
	80	13.0	1.3	89.6	6.1
	100	13.5	1.7	87.3	5.7
	140	12.5	2.5	80.0	5.4

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## CONCLUSIONS

The results of this research showed that Ferrifloc and Ferriclear perform as well as alum and better than alum at certain pH although they do not depress the pH of water as much as alum does. This means there will be little need for lime or none at all to aid them in the production of potable water to depending on the pH of the raw water. The solution of both Ferrifloc and Ferriclear are stable at room temperature. On the other hand, Ferrosol and Ferrichlor do not compare satisfactorily with alum.

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