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Evaluation of Alum and Purification Process of Water by Coagulation Method

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Abstract

Alum is a mineral substance that is obtained from nature in pure and impure forms. It is derived from aluminum ore, which is a mineral salt. It is found in many pharmaceutical, cosmetic, and food products. In this study, the three different types of alum were synthesized and evaluated by physical and chemical characteristics. The groundwater from different areas of Chennai was collected and evaluated by semi-quantitative and quantitative methods via Limit Test, Assay by Mohr's Method, Flame Photometry, and Total Hardness Test. All three different alums were treated with groundwater by the coagulation method for the purification of water. The alum-treated water was post-evaluated by semi-quantitative and quantitative methods. In the Limit test, alum-treated water produces less opalescence, turbidity, or colour when compared with untreated water. In the Estimation of chloride by Mohr's method in some water samples, alum reduces the amount of chloride ion concentration. In the Flame photometry analysis, the alum reduces the sodium ion concentration in all areas' groundwater. When compared with all alum, Soda alum is more efficient. In the total hardness test, the hardness of alum-treated water increases due to its interaction with water and the excess production of metal ions. In vitro, antimicrobial activity of alum proved that potash alum produces high antimicrobial activity ammonium and soda alum showed significant anti-microbial activity. We summarized that soda alum is highly efficient in the water purification process and produces higher antimicrobial activity than the other two alums.

Keywords: Alum, Coagulation, Semi-quantitative, Water treatment

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Introduction

Alum is a mineral substance that is obtained from nature in pure and impure forms. It is obtained from aluminum ore (mineral salt). It can be clear, colorless, odorless, and crystalline and tastes like a sweet astringent. It has a molecular weight of 458.28 gm [1]. It is found in many pharmaceutical, cosmetic, and food products. It has a strong astringent quality. It comes in four different colors: white, green, yellow, and red. White is known as phitkari, green is known as heera kasees, yellow is known as kasees, and red is known as surkh phitkari [2]. When heated, it melts and loses its water of crystallization at around 200 degrees resulting in an anhydrous salt. It is frequently contaminated with impurities in markets. It can be made suitable for therapeutic use by dissolving alum in boiling water and the solution is filtered and evaporated to produce crystals which should be stored properly for future use [3].

Alum is a chemical that is used in dyeing textiles by Egyptians in ancient times. Historic people believed that Egyptians hardened the papyruses with alum. Pliny

(Roman historian) stated that alum acts in both the cosmetic and medical fields. Many people believed that alum was only used as a deodorant. In the 12th century, alum was still a common chemical that was used in the medical field as well as in dyeing textiles. It is commonly found in dry and desert regions. Alum was imported from North Africa or in surrounding regions. In the 17th century, the alum extraction method was developed by British people. In the 19th century, British people used alum in a bakery for making bread due to its better appearance. From the 20th century, baking soda was a competitor for alum but alum won [4].

In this study, the three different types of alum were synthesized and evaluated by physical and chemical characteristics.

Properties of alum [3]

Organoleptic character- White and transparent crystalline in nature.

Solubility- Highly soluble in water (especially in hot water).

Boiling point- Around 200 ◦ C

Melting point- 92.5

Density- 1.725 g/cm 3

Application of heat- Alum crystals are liquefied if heat is applied

Action on litmus paper- Blue turns to Red

Types of alum

Potash Alum is commonly named potassium alum. The chemical formula is $KAl(SO_4)_2 \cdot 12H_2O$ and potassium aluminum sulfate is its chemical name. The molar mass is 258.192 g/mol. It is white crystalline and smells like metallic water. Potash alum is also sometimes called white alum. Soda Alum is also called sodium alum or just SAS. Sodium aluminum sulfate is its chemical name and has a molar mass of 458.28 g/mol. It can be in white crystal or powder form and has a smell of metallic water. The chemical formula is $NaAl(SO_4)_2 \cdot 12H_2O$. Ammonium Alum is referred to as ammonium sulfate alum. The chemical formula is $NH_3Al(SO_4)_2 \cdot 12H_2O$. Molar mass of 132.14 g/mol. It exists in a small white crystalline form with a metallic water smell. The common abbreviation of Chrome Alum is CAS. It is commonly called chromium alum and has a chemical formula $KCr(SO_4)_2 \cdot 12H_2O$. It is also known as chromium potassium sulfate and has a molar mass of 283.22 g/mol. Like other alums, it has a metallic water smell but mostly exists in a purple crystal form. Selenate Alum is commonly known as aluminum selenate. In this form of alum, selenite is present instead of sulfate. It acts as a strong oxidizing agent. The molecular formula is $Al_2O_{12}Se_3$ and its molar mass is 482.9 g/mol [5].

Uses of alum

Potash alum is used in baking powder, pickling leather, water clarifiers, aftershave, and fireproof clothes. Soda alum is most commonly used in baking soda and the food industry it acts as an acidulant. Ammonium Alum is commonly used in tanning, dyeing in textiles, flame retardant processes for textiles, and in the manufacture of certain types of cement, glues, and deodorants. It may also be used in water purification systems. Chrome alum has a deep violet color, and is commonly added to other types of alum to grow purplish crystals. It is most commonly used in leather tanning. Selenite alum is used in the preparation of antiseptics. Apart from these uses, alum also has therapeutic properties like Anti-microbial, Anti-platelet, Anti-obesity, Anti-haemorrhagic, Anti-inflammatory, Anti-Dandruff, and Anti-asthmatic [6].

Role of alum in water treatment

Alum can be used to treat contaminated water in the water purification processes and to act as a coagulant in the Coagulation-Flocculation processes. It is a chemical Water treatment technique used before the sedimentation and filtration process. It is used to enhance the ability of water purification by removing large sedimented particles. Purification is done by Coagulation and Flocculation. It destabilizes the charge of the particles present in the sample. Coagulants(alum) with opposite charges (positive) of the suspended solids are added to the water to neutralize the negative charges on dispersed solids (clay and organic substances). Once the charge is neutralized, the small suspended particles stick together and microflocs are formed. Micro flocs are nothing but slightly large particles that formed and they are still too small to be visible to the naked eye. In other words, it is a chemical Water treatment technique done before the sedimentation and filtration process to strengthen the proficiency of the treatment and its ability to remove polluted and dirty particles. Flocculation is the process where a chemical coagulant is added to the water to stimulate bonding between different types of particles. This leads to the creation of larger aggregates that can separate easily [7].

Materials and Methods

Chemicals required Potassium sulfate, Ammonium sulfate, Sodium sulfate, Ammonium sulfate, concentrated sulphuric acid, and Distilled water.

The apparatus required a Beaker, China dish, glass rod, tripod stand, and funnel Equipment required a Hot plate.

Preparation of potash alum [8]

15 grams of potassium sulfate was weighed and transferred into a beaker. To this, 60 ml of distilled water was added and stirred continuously. Slightly warm the solution. Keep the solution aside (Beaker 1). In Beaker 2, 60 g of aluminum sulfate and 90 ml of distilled water were

taken, 6 ml of concentrated sulphuric acid was added and the solution was stirred continuously to get a clear solution (Beaker 2). Both the solutions were mixed and transferred into the china dish after passing them through filter paper. The solution was heated continuously until it reached the crystallization point. To check the crystallization point, take the stirring rod out of the china dish and blow some air on it. The formation of crystalline crust on a glass rod after blowing air is a sign of reaching crystallization.

After this process, the china dish was removed from the burner and kept in an ice bath after covering it with a watch glass overnight. After crystals are formed, crystals are filtered from the mother liquor and dried using filter paper. The product was weighed and the percentage of yield was calculated and reported.

Preparation of ammonium alum

47.4 g of aluminum sulfate was Weighed and transferred into the beaker, and 45 ml of hot water was added to dissolve and make up the volume of 75 ml (Beaker 1). Weigh 23.1 g of ammonium sulfate and transfer it to another beaker, 45 ml of distilled water was added to dissolve and make up a volume of 75 ml (Beaker 2). Both solutions were mixed to form ammonium alum. The solution was kept overnight at room temperature to get small crystals. Reheat and cool to obtain large crystals. After 1 hour crystals are formed. The product was weighed and the percentage of yield was calculated and reported.

Preparation of soda alum

31.6 g of aluminum sulfate was weighed and transferred into the beaker, and 30 ml of hot water was added to dissolve and make up to 50 ml with distilled water. 13.2 g of sodium sulfate was transferred to another beaker, and 30 ml of distilled water was added and made up the volume. Two solutions were mixed in the china dish and boil the solution on the burner. After boiling, the solution was kept in an ice bath overnight. Crystals are formed, dry, and weigh the product.

Evaluation tests for alum

A. Solubility:

Add 0.5 g of solid sample to 1 ml of distilled water in a test tube. Stir gently with a glass rod. Record the observation [9].

B. pH:

Before the determination of pH, the pH meter was calibrated. 1g of prepared alum was dissolved in 100 ml of distilled water in a beaker. pH was measured using the pH meter.

C. Identification test [10, 11]:

1. Flame test (Test for potassium)

The small amount of alum was taken in a spatula and ignited in the direct flame.

Observation: Lavender color flame

Inference: the presence of potassium

2. Flame test (Sodium test): Take a small amount of alum in a spatula and ignite it under a direct flame.

Observation: Yellow color flame

Inference: the presence of sodium

3. Test for ammonium: 10 mg of ammonium alum was heated with sodium hydroxide solution; ammonia is evolved, which is recognizable by its odor and by its action on moist red litmus paper, which turns blue.

Observation: Red litmus turns to blue

Inference: Presence of ammonium

4. Test for aluminum: 20 mg of the potash alum was dissolved in 2 ml of water, 0.5 ml of 2M hydrochloric acid was added, and about 0.5 ml of thioacetamide reagent; no precipitate was produced. 2M sodium hydroxide was added dropwise; a gelatinous white precipitate was produced which re-dissolves on the addition of a further 2M sodium hydroxide. Gradually ammonium chloride solution was added.

Observation: A gelatinous white precipitate was obtained

Inference: Presence of aluminum

5. Sulfate test: 50 mg of soda alum was dissolved in 5 ml of water and 1 ml of dil. Hydrochloric acid was added and 1 ml of barium chloride solution.

Observation: A white precipitate was obtained.

Inference: Presence of sulfate.

Selection of water sample

The water sample was collected from various parts of Chennai and the sample name was given as follows Perambur (WS1), Sriperumbudur (WS2), Retteri (WS3), Ernavoor(WS4) Maduravoyal (WS5).

Semi-quantitative evaluation for water samples

A limit test for chlorides, sulfates, iron, and lead was performed for all different areas of water as mentioned in Indian pharmacopeia [12, 13].

Determination of sodium content by flame photometry method

Preparation of a standard solution

0.548 g of sodium chloride (equivalent to 0.23 g of sodium) was dissolved in 100 ml of distilled water. 1, 2, 3, 4, and 5 ml of solutions were pipetted out in five different volumetric flasks and the volumes were made up to 100 ml with distilled water to get a solution of 1, 2, 3, 4, and 5 millimoles of sodium or 2.3, 4.6, 6.9, 9.2, and 11.5 mg of sodium per 100 ml respectively [14].

Preparation of sample solution

2 ml of water sample was dissolved in 50 ml of distilled water. The standard solution was introduced into the flame and emission intensities were measured at 589 nm and the sample solutions were also measured as the same. A

calibration graph with a concentration on the x-axis and intensity on the y-axis was drawn [15].

Purification of groundwater by prepared alum

The method adopted is coagulation. 100 ml of groundwater was taken in a beaker 0.1 g of different types of alum was added to a beaker containing groundwater and kept aside overnight. The solution was filtered [16].

IN-VITRO antibacterial activity

Method: Agar well diffusion

Organisms: Gram-negative bacteria: *Escherichia coli*, Gram-positive bacteria: *Staphylococcus aureus*

Medium: Mueller Hinton Agar (MHA)

Alum in the form of crystals pounded using mortar and pestle. It was weighed using analytical balance as 0.5g, 1.5g, and 2.5g. It was dissolved in 10ml distilled water. So that the concentration obtained in percent is 5%, 15%, and 25%. The prepared sample solutions were used for antibacterial activity by the Agar well diffusion method [17, 18].

Results and Discussion

Preparation of alum derivatives

1. Potash alum:
   - Theoretical yield – 40.83 g
   - Practical yield- 33.91 g
   - Percentage yield- 83.00%
2. Ammonium alum:
   - Theoretical yield – 79.24 g
   - Practical yield-94.03 g
   - Percentage yield-118.66%
3. Soda alum:
   - Theoretical yield -42.58 g
   - Practical yield-45.61 g
   - Percentage yield- 107.11%

Physical properties

The physical properties of Alum Derivatives are provided in **Table 1**.

Table 1. Physical Properties of Alum Derivatives

Name	Potash Alum	Ammonium Alum	Soda Alum
Chemical formula	$KAL(SO_4)_2.12H_2O$	$NH_3Al(SO_4)_2{\cdot}12H_2O$	$NaAl(SO_4)_2.12H_2O$
Molecular weight	474.39 g	453.33 g	458.28 g
Color	Colourless	Colourless white crystals	White
Odour	Odourless	Odourless	Odourless
Melting point	98 ^{0}C	102 ^{0}C	105 ^{0}C
Solubility	Soluble in water and sparingly soluble in ethanol	Soluble in water and sparingly soluble in ethanol	Soluble in water and partially soluble in ethanol
Solubility	3.29	3.62	3.13
Taste	Astringent	Astringent	Astringent

Chemical test

Depicting chemical test results for various types of alum are provided in **Table 2**.

Table 2. Depicting chemical test results for various types of alum

S/N	Alum	Sodium	Potassium	Aluminium	Sulphate	Ammonium
1.	Potash alum	-	+	+	+	-
2.	Ammonium alum	-	-	+	+	+
3.	Soda alum	+	-	+	+	-

(+) represent Positive result, (-) represent Negative results

Limit test

The limit test of chloride, sulfate, iron, and lead in different areas of water was performed. The opalescence, turbidity, or color produced before alum alum-treated water sample is more than after the alum-treated water. Therefore, the alums absorb the ions from groundwater and purify the water. The results are tabulated **(Table 3)**.

Table 3. Depicts limited test results for water samples from different areas and alum-treated water

S/N	Limit tests	Before alum treatment	After alum treatment		
			Potash Alum	Ammonium Alum	Soda Alum

1.	Chloride	Intense opalescence	Less opalescence	Less opalescence	Less opalescence
2.	Sulfate	Intense turbidity	Less turbidity	Less turbidity	Less turbidity
3.	Iron	No purple-pink color is produced	No purple-pink color is produced	No purple-pink color is produced	No purple-pink color is produced
4.	Lead	Intense color complex	Less color complex	Less color complex	Less color complex

Determination of sodium content by flame photometry

The determination of sodium present in the sample was performed by flame photometry. The test concentration of water samples is compared with standard sodium chloride. The values are tabulated **(Tables 4 and 5)**. From the table, it is observed that all derivatives of alum absorbed the sodium ion from the groundwater samples.

Table 4. Depicts the result of Flame photometry for standard sodium chloride.

	Concentration	Flame Intensity
1.	1 mmol	1
2.	2 mmol	2
3.	3 mmol	3
4.	4 mmol	4
5.	5 mmol	5

Table 5. Flame photometry for water from different areas & alum-treated water.

S/N	Sample	Flame Intensity			
		Before water treatment	Potash alum-treated water	Ammonium alum-treated water	Soda alum-treated water
1.	WS1	1	1	0	1
2.	WS2	3	1	0	1
3.	WS3	2	1	0	1
4.	WS4	1	0	0	1
5.	WS5	2	1	0	1

In vitro antimicrobial activity

The *in vitro* antimicrobial activity for three different alums was performed. The results are tabulated. The zone of inhibition was measured. From **Table 6**, it was observed that the three different alums were active against both gram-positive and gram-negative bacteria antibacterial activity. Among the three different alums, soda alum produces the highest antibacterial activity against both bacteria **(Figure 1)**.

Table 6. Zone of Inhibition of Alum

S/N	Organism	Potash alum in (mm)			Ammonium alum in (mm)			Soda alum in (mm)		
		5%	15%	25%	5%	15%	25%	5%	15%	25%
1.	*E. coli*	12	19	18	10	16	14	13	21	23
2.	*S. aureus*	14	21	20	10	11	11	19	23	29

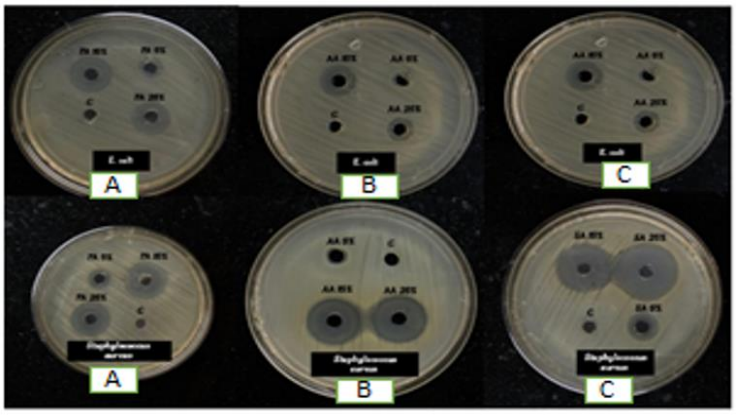

Figure 1. (A) Potash alum zone of inhibition; (B) Ammonium alum zone of inhibition; (C) Soda alum zone of inhibition.

Summary

In this study, the three different types of alum were synthesized and evaluated by physical and chemical characteristics. The groundwater from different areas of Chennai was collected and evaluated by quantitative method via Flame Photometry. All groundwater samples were treated with different alums by coagulation method for purification of water. The alum-treated water was post-evaluated. In the Flame photometry analysis, the alum reduces the sodium ion concentration in all area's groundwater. When compared to all alum Soda alum is more efficient than others. *In vitro*, the antimicrobial activity of alum proved that potash alum produces higher antimicrobial activity than others, but ammonium and soda alum showed significant anti-microbial activity. We summarized that soda alum is highly efficient in the water purification process and produces higher antimicrobial activity than the other two alums.

Conclusion

Different types of alum like potash, ammonium, and soda alum crystals synthesized by simple chemical reactions. The selection of water samples for this study is due to their hard nature. The purification process is achieved by a simple coagulation method. The antimicrobial activity of alum was evaluated for all three alums. Soda alum is more efficient than potash and ammonium alum in purifying water as well as producing better antimicrobial activity. By adopting, another purification method, the removal of all metal ions and elements present in hard water may be achieved.

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