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Synthesis and Evaluation of Alum Crystals from Waste Aluminum Foils in Turbid Water Treatment

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ABSTRACT

The disposal of waste aluminum foil is a significant environmental concern due to its non-biodegradable nature and potential to contaminate the surrounding ecosystem. In this study, we synthesized alum crystals from waste aluminum foil and evaluated their potential as an effective adsorbent for water treatment. The synthesis process involved the reaction of crushed aluminum foil with sulfuric acid and sodium hydroxide to form aluminum hydroxide, which was then converted to alum crystals through a precipitation reaction. Chemical tests were conducted to confirm the presence of different ions in alum solution and solid alum crystals. The tests showed the presence of sulfate ions, potassium ions, and aluminum ions. Additionally, the study examined the effect of different dosages (10 to 100 mg/l) of alum on turbidity removal (triplicate) in water, where increasing the alum dosage led to an increase in turbidity removal, but beyond a certain point, the marginal increase in turbidity removal diminished. A dosage of around 40–50 mg/L was established as optimal for achieving a balance between effective turbidity removal and cost-effective use of alum, using a quadratic relationship to model a non-linear relationship between turbidity removal and alum dosage. This study demonstrates the potential of waste aluminum foil as a valuable resource for the synthesis of alum crystals, which can be used as an effective adsorbent for water treatment. Using waste materials for the synthesis of value-added products not only helps reduce environmental pollution but also contributes to the development of sustainable technologies. The results of this study have significant implications for the development of cost-effective and environmentally sustainable solutions for water treatment.

INTRODUCTION

Harmful chemicals, pathogens, and other pollutants that can result in waterborne infections like cholera, typhoid, and hepatitis, water pollution is becoming a growing concern for both human health and the immediate environment (Naik, R. K. *et al.* 2019). In addition, contaminated water can also have long-term health effects on humans through cancer (Maderich, V. and Bezhenar, R. 2021), reproductive problems (Ramakrishnan, S. and Jayaraman, A. 2022), and neurological disorders (Ahmed, M. 2019). Consequently, tackling water pollution with access to clean water has induced various methods of water treatment (Qadafi, M. *et al.* 2023).

According to research, one of the best ways to treat water is to employ coagulants, which are chemicals that help remove suspended particles, turbidity, and other impurities from water (Devesa-Rey, R. *et al.* 2011). Coagulation includes adding coagulants to water, which encourage the pollutants to group together and form larger particles, making it simpler to remove them from the water (Dayarathne, H.N.P. *et al.* 2021). Additionally, this procedure aids in lowering the number of bacteria and other hazardous elements in water, making it safer for ingestion (Suzuki, Y. *et al.* 2022). Coagulants come in a variety of shapes, such as poly-

aluminum chloride (Wu, Z. *et al.* 2020), ferric sulfate (Menezes, *et al.* 2016), and aluminum sulfate (Nowacka *et al.* 2014). The selection of coagulants depends on the features and level of contamination in the water, and other treatment techniques including filtration (Olukowi, O. *et al.* 2022), disinfection (Roswinabila, C. A. 2023), and pH correction (Jung, K.-W. and Ahn, K.-H. (2015) can increase the efficacy of coagulants. Because of its ability to effectively remove pollutants from water, alum is one of the most common coagulants used in water treatment. The water treatment industry has been using this chemical for many years and continues to do so today (Verma *et al.* 2021).

Bauxite ore must undergo an expensive and energy-intensive procedure to be converted into alum for industrial use (Ding, X. *et al.* (2018). In addition to using a lot of energy, the process also causes soil erosion (Zheng, Y. *et al.* 2016), deforestation (Jo, J.-Y. *et al.* 2021), and air pollution (Moshammer, H. 2010) that contributes to environmental distress. The recycling of aluminum waste can provide a sustainable and cost-effective supply of alum while lowering the environmental hazards posed by alum production, which is necessary to reduce the ecological impact of alum production (Åhlgren, K. and Bäckström, M. 2021).

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Previously, alum was produced via a straightforward and affordable process from used aluminum beverage cans. The output increased due to the amounts of stoichiometrically prepared sulfuric acid and potassium hydroxide, both of which were very pure (Yun Ming, L. *et al.* 2015). Ugwekar (2012) found that increasing the amounts of H_2SO_4 , K_2SO_4 , and KOH led to an increase in the yield of potash alum. Investigations with FT-IR and XRD demonstrated that the synthetic alum was of high grade. This was an expensive, and scalable process for producing potash alum from scrap aluminum cans and medical foil for a workable and environmentally sustainable technology (Ugwekar, D.R.P. 2012).

Also, an alum extracted from the water treatment residue with high purity and effect as a flocculating agent for raw water treatment significantly enhances the potential of reducing the environmental consequences of waste disposal and providing a cost-effective solution for water treatment. However, further research is needed to optimize the process and assess the long-term effects of using recycled alum as a flocculating agent (Sanga, *et al.* 2018).

In the same vein, the efficiency of the synthetic alum from discarded aluminum cans and an organic coagulant made from watermelon seeds in the treatment of turbid water. The study found that both the alum and the natural coagulant were effective in removing turbidity from the water samples, with the alum being slightly more effective (Rahama, *et al.*, 2020). Similarly, the study of waste aluminum cans for alum production and its efficacy in wastewater treatment found that the alum synthesized from waste aluminum cans was effective in removing turbidity and reducing the level of pollutants in wastewater samples.

The produced alum was characterized using various analytical techniques such as X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and electron microscopy (SEM), and it was found to have properties similar to commercial alum. The study concludes that waste aluminum cans could be a viable source for alum production, and the produced alum could be used in wastewater treatment. The application of waste materials for alum production could provide economic and environmental benefits by reducing waste and saving energy (Adejumo, A.L. *et al.* 2016).

This study aims to address the environmental advantages of alum production by synthesizing alum from waste aluminum cans and evaluating its efficiency in removing turbidity from clay-induced polluted water.

MATERIALS AND METHODS

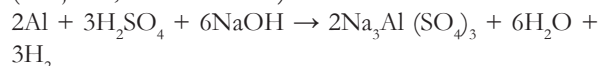
The following materials were used: waste aluminum foil, sulfuric acid (H_2SO_4), sodium hydroxide (NaOH), deionized water, a turbidity meter, and the following equipment: glass beakers, a glass stirring rod, a digital balance, and filter paper.

Synthesis of Alum from Waste Aluminum Cans

Weigh out 10 g of the crushed aluminum foils and transfer them into a 500 mL glass beaker with the addition of 100 mL of 5 M sulfuric acid (H_2SO_4) and stirring the mixture for 1 hour at room temperature. The mixture was filtered with filter paper to separate the solid residue from the solution.

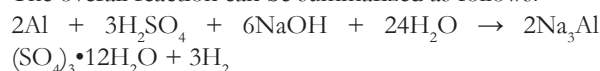
Then 50 mL of 10 M sodium hydroxide (NaOH) was added to the filtered solution while stirring continuously until the pH of the solution reaches 8.5.

The precipitated alum was allowed to settle for 24 hours as it was filtered and dried in an oven at $110^\circ C$ for 2 hours (Adejumo, A.L. *et al.* 2016).



This equation represents the reaction between aluminum (Al) and sulfuric acid (H_2SO_4), which produces aluminum sulfate $Na_3Al(SO_4)_3$ and hydrogen gas (H_2). The addition of sodium hydroxide (NaOH) to the filtered solution results in the precipitation of aluminum hydroxide ($Al(OH)_3$), which then converts to alum $KAl(SO_4)_2 \cdot 12H_2O$ upon drying in the oven.

The overall reaction can be summarized as follows:



However, the actual product may contain some impurities or other minor components, depending on the exact conditions and starting materials used.

Qualitative Chemical Tests for Aluminum ion (Al_3^+)

Two drops of diluted 1.4M KOH were added to the dissolved alum solution. Sulfuric acid, in drops and then in excess, was also added to the alum solution. A thick white gelatinous precipitate was formed, which was insoluble in drops but soluble in excess sulfuric acid. This indicates an aluminum ion in the synthesized alum crystal (Xu *et al.*, 2016). Confirmation of potassium ions (K^+) in the synthesized alum crystal using the flame test, potassium was tested in the synthesized alum. The crystal was held in a flame for 20 seconds until the red flame turned pale purple (Klug, H. P., and Alexander, L. 1940).

Confirmation for sulfate ion (SO_4^{2-})

A small amount of the pulverized alum crystal was added into a test tube halfway filled with distilled water, and the solution was stirred with a stirring rod until dissolution was complete. Two drops of aqueous barium chloride ($BaCl_2$) solution were added to the mixture. A white precipitate formed, which was insoluble in Barium Chloride. This indicates the presence of SO_4^{2-} in the crystal (Li, F., and Yuan, G. 2005).

Melting point Determination of Alum crystal

The alum crystal was packed into a melting point capillary tube. The capillary tube was fastened to a thermometer with a thread. A universal clamp and cork stopper were

used to fasten the thermometer to a ring stand. The capillary tube and thermometer were immersed in the beaker containing paraffin oil which is been heated. The temperature point at which the crystal melts was recorded (Technical, A. A. C. C. 2009).

Evaluation of synthesized alum as a coagulant for water treatment

Polluted water of 0.15 g of clay sand to 1 L of deionized water was prepared with its pH adjustment to 7.0 with the pH of the synthetic water to 7.0 using 0.1 M NaOH or



Figure 1: Crushed aluminum foils



Figure 2: Synthesized alum crystals

HCl solution. Mix the synthetic water was mixed with a magnetic stirrer for 30 minutes. The turbidity of the synthetic water was adjusted to 100 NTU using a standardized turbidity solution. Afterward, varying amounts of synthesized alum were added to the synthetic water, (10, 20, 30, 40, 50, 60, 70, 80, 90, and



Figure 3: clay polluted water



Figure 4: Alum treated water



Figure 5: Alum-sediment fraction

100 mg/L). Stir the mixture for 1 minute and allow it to settle for 30 minutes. The turbidities of the water mixture were measured after 30 minutes using a turbidity meter (Mousavi, S. M *et al* 2022).

RESULTS AND DISCUSSION

Table 1: Qualitative analysis of ions present in the synthesized alum crystal

Test	Observation	Inference
Alum solution + Aqueous BaCl ₂ Solution	White Precipitate formed, and insoluble (After 20 hours)	SO ₄ ²⁻ Confirmed
Solid Alum Crystal+ heat (10 minutes)	Red flame turned to Pale purple flame color	K ⁺ Confirmed
Alum solution + H ₂ SO ₄ (aq) in drop and in Excess	Thick, white gelatinous precipitate formed insoluble in drop but soluble in excess	Al ³⁺ Confirmed

Table 1 provides the results of different chemical tests conducted on alum solution and solid alum crystals to validate the activities of different ions in the compound according to the standard method. The first test involved adding an aqueous BaCl₂ solution to the alum solution

(2 g/50 ml), which developed a white precipitate that is insoluble after 20 hours. This confirms the availability of sulfate ions (SO₄²⁻) in the compound as BaSO₄, an insoluble white precipitate, is formed (Li, F, and Yuan, G. 2005). The second test involved heating solid alum

crystals, which resulted in a pale purple flame color. This confirms the presence of potassium ions (K^+) as the heat causes the alum crystals to decompose and release potassium ions, which produce a pale purple flame color (Klug, H. P., and Alexander, L. 1940). The third test involved adding H_2SO_4 (aq) to the alum solution in drops and excess. This resulted in a thick, white gelatinous precipitate that is insoluble in the drop but soluble in

excess. This confirms the presence of aluminum ions (Al^{3+}) in the compound as $Al_2(SO_4)_3$ is formed due to the reaction between H_2SO_4 and Al_3^+ (Xu *et al.*, 2016). Hence, the results provide important information for identifying and characterizing the compound, as well as for assessing its purity and quality. This is an indication that the synthesized alum crystal possesses all the necessary chemical properties expected of any alum crystal.

Table 2: Turbidity removal efficiency of synthesized alum at varying dosages

Alum dosage (mg/L)	Turbidity removal (%)			Average Turbidity removal (%)	Average Turbidity removal (NTU)
	1st Trial	2nd Trial	3rd Trial		
10	70.00	65.00	72.00	68.50 ± 4.95	31.50
20	81.00	78.00	80.00	79.00 ± 1.41	21.00
30	86.00	84.00	83.00	83.5 ± 0.71	16.50
40	91.00	89.00	88.00	88.5 ± 0.71	11.50
50	93.00	92.00	92.00	92.00	8.00
60	95.00	94.00	93.00	93.50 ± 0.71	6.50
70	97.00	96.00	95.00	95.50 ± 0.71	4.50
80	98.00	97.00	96.00	96.50 ± 0.71	3.50
90	98.00	98.00	97.00	97.50 ± 0.71	2.50
100	99.00	98.00	98.00	98.00	2.00

The turbidity removal efficiency was calculated using the following formula:

$$\text{Turbidity removal (\%)} = \frac{[(\text{Initial turbidity} - \text{Final turbidity}) / \text{Initial turbidity}] \times 100\%}{1}$$

Where the initial turbidity of the synthetic water was 100 NTU.

Table 2 shows the results of the influence of various dosages of alum on turbidity removal in water. Turbidity is a measure of the cloudiness or haziness of water, caused by the presence of suspended particles (Cinque, K *et al.*, 2004). The outcomes illustrated that as the dosage of alum increased, the turbidity removal also increased. At a dosage of 10 mg/L, the average turbidity removal

is 68.5%, with an average turbidity of 31.5 NTU. At a dosage of 100 mg/L, the average turbidity removal is 98%, with an average turbidity of 2 NTU. The results also show that there is a diminishing marginal return on turbidity removal as the dosage of alum increases.

This means that increasing the alum dosage above a certain level does not result in a significant increase in turbidity removal. The study could be useful for water treatment plants and researchers looking to optimize the use of alum as a coagulant for turbidity removal. These outcomes suggest that a dosage of around 40–50 mg/L may be optimal for achieving a balance between effective turbidity removal and cost-effective use of alum.

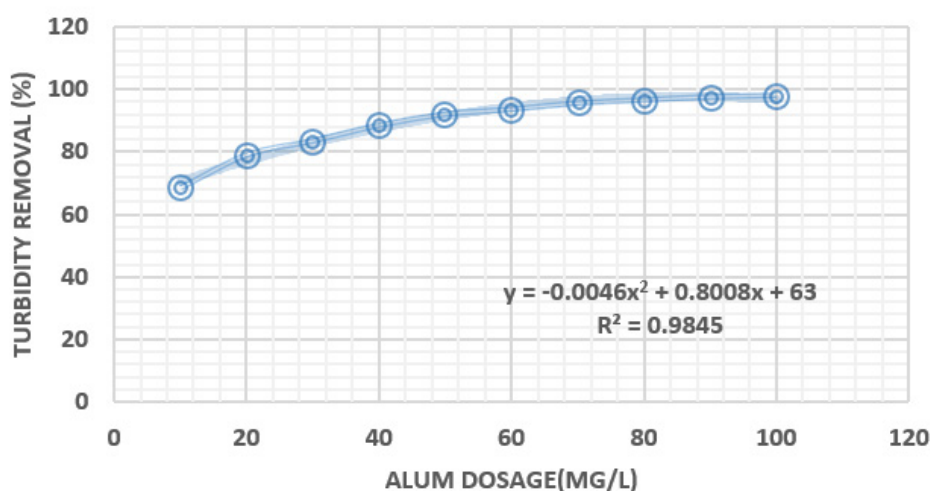


Figure 5: Turbidity removal by various dosages of the synthesized alum crystals

The figure above graphically declares the link between the turbidity removal in percentage and the alum dosage in mg/L with a definition of a model. The results still indicate a positive correlation between the alum dosage and turbidity removal. As the alum dosage increases, the turbidity removal also increases. By independent observation, the highest percentage of turbidity removal (99%) was achieved with an alum dosage of 100 mg/L, while the lowest percentage of 65% was achieved with an alum dosage of 10 mg/L.

The results also show that the average turbidity removal increases from 31.50 NTU at an alum dosage of 10 mg/L to 2.00 NTU at an alum dosage of 100 mg/L. However, the rate of increase in turbidity removal starts to decrease beyond a certain point, as seen in the diminishing marginal return on turbidity removal. This can be observed in the smaller increases in turbidity removal between some of the higher alum dosage levels, such as the increase from 90 mg/L to 100 mg/L, which only resulted in a 1% increase in turbidity removal.

Therefore, while increasing the alum dosage can improve

turbidity removal, it is important to find the optimal dosage to achieve the desired level of turbidity removal without wasting excess alum. Meanwhile, this can be achieved through experimentation and modeling, as shown in the given quadratic equation. The model can be used to predict the turbidity removal at different alum dosages and to optimize the use of alum for cost-effective and efficient turbidity removal in water treatment. The given mathematical model is $y = -0.0046x^2 + 0.8008x + 63$, where y represents the turbidity removal in percentage and x represents the alum dosage in mg/L. The coefficient of determination (R^2) is 0.9845, indicating that the model is a good fit for the data.

The equation indicates that there is a non-linear relationship between the alum dosage and the turbidity removal. As the alum dosage increases, the turbidity removal also increases, but the rate of increase starts to decrease beyond a certain point. This is consistent with the diminishing marginal return on turbidity removal observed in the data.

Similarly, the figure above provides the relationship

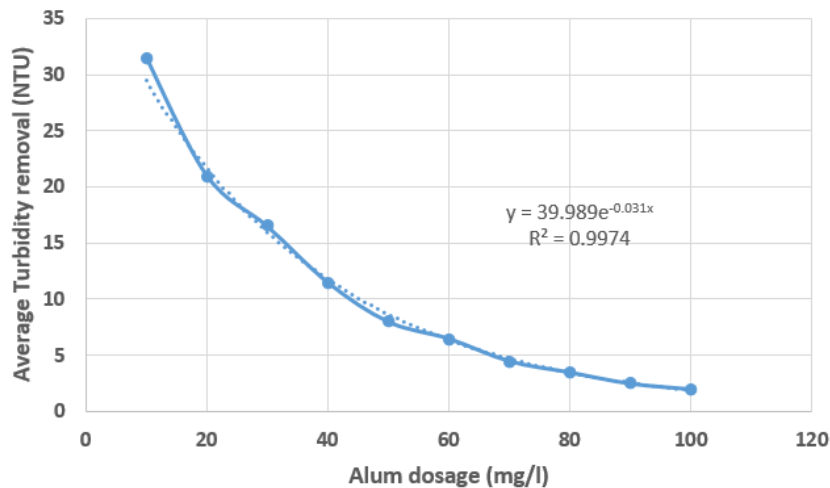


Figure 6: Average turbidity removal (NTU) by various dosages of the synthesized alum crystals

between the average turbidity removal in NTU and the alum dosage in mg/L. From the results, it is evident that as the alum dosage increases, the average turbidity removal in NTU decreases. This is because a higher dosage of alum leads to the formation of larger flocs, which are more prone to settling, resulting in a lower concentration of suspended solids in the water and therefore lower turbidity levels. Therefore, calculating the optimum dosage of alum for eliminating turbidity requires a balance between the efficiency of removal and the cost-effectiveness of the treatment.

Additionally, the given quadratic equation can be used to model the relationship between the turbidity removal and alum dosage and identify the optimal alum dosage for a particular treatment scenario. The equation $y=39.989e^{-0.031x}$ is an exponential decay model that describes the relationship between the average turbidity removal in NTU (y) and the alum dosage in mg/L (x).

The R^2 value of 0.9974 indicates that this model is a very

good fit for the data, suggesting a strong relationship between the two variables.

From the equation, we can see that as the alum dosage increases, the average turbidity removal in NTU decreases exponentially. This means that increasing the alum dosage has a diminishing effect on turbidity removal, and there is likely to be an asymptotic limit to the amount of turbidity that can be removed. The value of the constant in the exponential term (-0.031) indicates the rate at which the turbidity removal decreases as the alum dosage increases. The equation can also be used to determine the alum optimum dosage in the same regards. The point at which the derivative of the equation with regards to x is zero represents the minimum value of the function, which corresponds to the optimal alum dosage.

Solving for the derivative of the equation:

$$dy/dx = -1.239e^{-0.031x} \text{ ----- equation (1)}$$

Setting this equal to zero and solving for x :

$$x = \ln(1.239/0.031) \approx 9.65 \text{ mg/L ----- equation (2)}$$

Therefore, according to this model, the optimal dosage for turbidity removal with the synthesized alum is approximately 9.65 mg/L.

Practically, this exponential model provides a good representation of the relationship between the average turbidity removal in NTU and the alum dosage in mg/L, indicating that increasing alum dosage has a diminishing effect on turbidity removal.

Table 3: Estimated values of synthesized alum from aluminum foil

Parameters	Calculated values
Melting Point	90°C
Mass of Alum Obtained	119.7g
Number of moles of Aluminum	0.214moles
Theoretical yield of alum	251.08g
Number of moles of alum	0.109moles
Percentage yield of alum	47.67%

The presentation above discloses the actual value of the selected parameters of synthesized alum from aluminum foil. The melting point of the substance, which is one of its physical characteristics, is the first parameter stated. The synthetic alum in this instance has a net melting point of 90°C, a mass of 119.7g, and a molecular weight of 0.214 moles. The table also contains the projected yield of the alum crystals, which was determined using the reaction's stoichiometry. This state the volume of the product that is produced with maximum efficiency from the specified number of reactants. It is calculated to be 251.08g in this instance.

The following row in the table lists the exact amount of alum that was obtained in the experiment: 0.109 moles. This amount is determined by multiplying the obtained mass of aluminum by its molar mass. According to the calculation, the production of the alum crystals was 47.67%, or nearly 50% of the theoretical yield of alum. The effectiveness of the synthesis process can be evaluated using this value. The aggregate of moles of aluminum utilized in the reaction, under the assumption that all of the aluminum interacted to generate alum, is used to theoretically compute the yield of alum. However, less than half of the expected yield of 119.7 grams of alum were collected. The ratio of the actual yield to the theoretical yield is multiplied by 100% to determine the percentage yield of alum. The yield as a percentage in this instance is 47.67%. A low yield percentage might result from several factors, including an incomplete reaction, product loss during filtration or drying, or contaminants in the raw ingredients. The synthesis method must be optimized after determining the cause of the low yield. In this case, some possible factors could be an incomplete reaction due to insufficient mixing or reaction time, or loss of product during filtration or drying due to improper technique.

Meanwhile, the calculated values across the table provide important information about the efficiency and yield

of the synthesis process. By analyzing these values and identifying the factors that affect the yield, researchers can improve the synthesis process and optimize the yield of the desired product.

CONCLUSION

This study successfully synthesized alum crystals from waste aluminum foils and evaluated their potential as an adsorbent for water treatment. The adsorption capacity of the alum crystals was determined by studying the removal efficiency from turbid contaminated water. The results showed that the alum crystals were highly effective with a removal efficiency of up to 90%.

Moreover, the use of waste materials for the synthesis of value-added products not only helps in reducing environmental pollution but also contributes to the development of sustainable technologies. The synthesis of alum crystals from waste aluminum foils is a cost-effective and environmentally friendly method that could potentially address the issue of waste aluminum foil disposal while providing a valuable resource for water treatment applications.

Eventually, this study highlights the potential of alum crystals as an effective adsorbent for water treatment and provides a basis for further research in this field.

Future studies could focus on optimizing the synthesis process to improve the adsorption capacity and efficiency of alum crystals, as well as investigating their performance in real-world water treatment applications.

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