

**Crystallization of a compound from different solvents &
comparison of the size and purity of the crystals obtained.**

Submitted for the partial fulfilment of the degree of

Bachelor of Technology

In

Chemical Engineering

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UNDER THE SUPERVISION AND GUIDANCE OF

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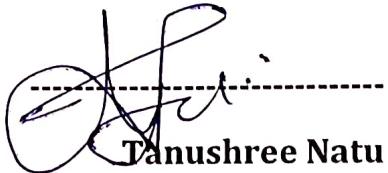


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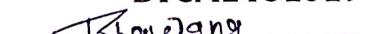
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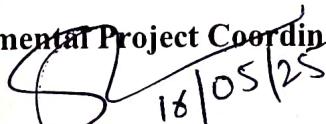
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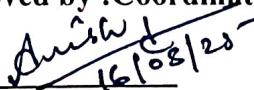
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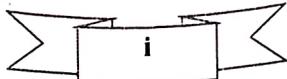


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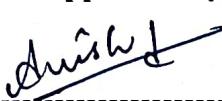


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ABSTRACT

This study explores the recycling of waste aluminium cans through the synthesis of alum (potassium aluminium sulphate dodecahydrate, $KAl(SO_4)_2 \cdot 12H_2O$), a valuable industrial compound. Discarded aluminium cans were collected from MITS canteen, cleaned, and reacted with potassium hydroxide (KOH) to form soluble aluminate ions. The resulting solution was then acidified with sulfuric acid to precipitate alum crystals. The product was filtered, washed, and dried before being characterized using physical and chemical methods. The dried alum crystals are weighed on an electronic balance. Theoretical yield is calculated based on the initial mass of aluminium used and the percentage yield determined as:

$$\% \text{ Yield} = (\text{Actual Yield} / \text{Theoretical Yield}) \times 100$$

The experiment demonstrated an efficient and environmentally beneficial method of recycling aluminium waste while producing a useful compound, highlighting the potential of waste-to-resource conversion in sustainable chemistry.

KEYWORDS: - Aluminium cans , Potassium hydroxide (KOH), Alum.

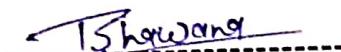
ACKNOWLEDGEMENT

The have taken effort in this project. However, it would not have been possible without the kind support and help of many individuals and organizations. We would like to extent our sincere thanks to all of them. We would like to take the opportunity to express our humble to our faculty Prof. Shivangi Sharma under whom we have done this project. Her constant guidance and willingness to share her vast knowledge made us to understand this project and manifestations in great depth and helped us to conquer the assigned takes. We would like to thank all faculty members of Chemical Engineering MITS; Gwalior for their generous help in various ways for completion of this project.

We would like to express our heartfelt thanks to teachers for their help and wishes for successful completion of this project.



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CHAPTER 1: INTRODUCTION

Recycling and green management of resources have emerged as important in confronting the growing international issues of environmental degradation and resource depletion. Of many recycling methods, converting waste materials into utilizable chemical products is an imaginative way of practicing sustainable development. Synthesis of alum (potassium aluminium sulphate dodecahydrate, $KAl(SO_4)_2 \cdot 12H_2O$) from rejected aluminum cans is one such useful and educationally valuable experiment. This is not only a great example of chemical recycling but also illustrates basic inorganic chemistry concepts such as redox reactions, crystallization, and characterization of compounds.

Alum and Its Importance

Alum refers to a family of double salts that are formed by the combination of a trivalent metal ion (such as Al^{3+} , Fe^{3+} , or Cr^{3+}) and a univalent cation (like K^+ , Na^+ , or NH_4^+) with sulfate ions. The most common form is potassium aluminum sulfate dodecahydrate. It has the chemical formula $KAl(SO_4)_2 \cdot 12H_2O$ and appears as a colorless, crystalline solid that is soluble in water. Alum is widely used in various industries for water purification, dyeing textiles, tanning leather, and as a component in baking powders and antiseptics.

Recycling Aluminium Cans

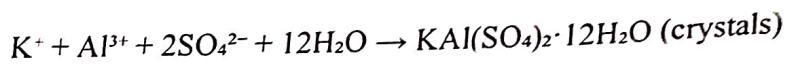
Aluminium cans are the most numerous and readily collectible waste resource across the globe. Cans are mostly composed of elemental aluminium that is sometimes coated with a thin layer to avoid corrosion. Aluminium recycling is of monumental environmental advantage, e.g., by eliminating the necessity for bauxite mining and conserving energy. Recycling waste aluminium to produce a value-added product such as alum presents a reflective example of green chemistry application.

Chemical Principles Involved

Preparation of alum from aluminium cans is a process of sequential chemical reactions each denoting a particular concept in chemistry:

- 1. Redox Reaction:** The metal aluminium (Al) reacts with a strong base (preferably potassium hydroxide, KOH) and produces soluble aluminate ions $[Al(OH)_4]^-$ with the evolution of hydrogen gas. This is a redox reaction as aluminium is being oxidised.
$$2Al (s) + 2KOH (aq) + 6H_2O (l) \rightarrow 2K[Al(OH)_4] (aq) + 3H_2 (g)$$
- 2. Acid-Base Reaction:** The aluminate solution is acidified with sulfuric acid (H_2SO_4), causing the precipitation of aluminium hydroxide, which then dissolves in excess acid to give Al^{3+} and sulphate ions.
$$2[Al(OH)_4]^- + H_2SO_4 \rightarrow 2Al(OH)_3 (s) + SO_4^{2-} + 2H_2O$$
- 3. Crystallization:** When potassium sulfate (K_2SO_4) is added to the acidified solution containing Al^{3+} and SO_4^{2-} ions, alum is formed upon cooling and evaporation of the solution.
$$K^+ + Al^{3+} + 2SO_4^{2-} + 12H_2O \rightarrow KAl(SO_4)_2 \cdot 12H_2O$$

(crystals) Crystallization: Upon addition of potassium sulfate (K_2SO_4) to the acidified solution with Al^{3+} and SO_4^{2-} ions, alum precipitates when cooled and evaporated.



CHAPTER 2: LITERATURE SURVEY

Aluminum waste, such as discarded drink cans, can be utilized to make alum. An aluminate solution is created at the start of the procedure by reacting aluminum with potassium hydroxide. Then, to encourage the crystallization of alum ($KAl(SO_4)_2 \cdot 12H_2O$), this is treated with sulfuric acid and then heated and cooled. The beneficial attributes of aluminum, such as its low density, resistance to corrosion, and exceptional electrical conductivity, make it a highly valued metal that finds extensive application in sectors like electronics and aircraft. Waste aluminum is efficiently recycled in this method to create alum, which has numerous uses, especially in water purification. This study aimed to evaluate the performance of commercially available alum with that of lab-prepared alum. The amount of water treated prior to coagulation was found to be almost identical in both samples. Nevertheless, upon coagulation, the conventional commercial alum produced somewhat more clarity, suggesting slightly superior efficacy. The turbidity of water in both groups before coagulation is the same, and after coagulation, the standard's turbidity is largest. In the standard group, solid particles are very dissolved after preparation and coagulation. The prepared and standard groups' pre-coagulation waters appear muddy/brown, clear/colourless, and very unclear/barely muddy. After coagulation, in both, pH decreases. The product formed is of higher conductivity than usual. In comparison, the material produced in the product is much more acidic and also contains higher amounts of calcium, sulphate, potassium, and sodium. In conclusion, there are no comparative differences in the use of already existing alum available in markets for the same reason and the same being made. [1]

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Synthesis of alum from aluminium waste is possible. In addition to removing ~~.....~~ environment, the requirements of the developing water treating industries are also being met. The synthesis therefore accounts as a better waste aluminium management practice. Just like it, it accounts as one with very high atom economy which is today's trend in the green and sustainable chemistry. The financial feasibility of the process is outside the limit of the present study and is therefore recommended for further studies.[2]

Disposal of waste aluminium foil is a critical environmental issue because it is non-biodegradable and has the potential to pollute the surrounding environment. In this paper, we have prepared alum crystals from waste aluminium foil and investigated their potential as a good water treatment adsorbent. Synthesis of alum involves the reaction of shattered aluminium foil with sodium hydroxide and sulfuric acid to produce aluminium hydroxide, which eventually crystallizes in the form of alum crystals after undergoing a precipitation reaction process. Chemical analysis was carried out in an attempt to ascertain if and what various ions were present in alum solution as well as in crystallized alum solids. The findings confirmed the presence of sulphate ions, aluminium ions, and potassium ions. Furthermore, the research experimentally confirmed the impact of various dosages (10 to 100 mg/l) of alum on turbidity removal (triplicate) in water, where an increase in dosage of alum resulted in increasing removal of turbidity, but at a certain point, the marginal rise in removal of turbidity decelerated. The best dose of approximately 40–50 mg/L was best for effective turbidity removal and economic utilization of alum with quadratic fit to explain the non-linear relation between the removal of turbidity and the dose of alum. The present study confirms the efficacy of waste aluminium foil as a potential material for its application in synthesizing alum crystals that can be utilized as an effective adsorbent for water treatment. Use of waste materials for synthesis of value-added products not only reduces environmental pollution but also helps in the development of sustainable technology. The results of this study have significant implications in terms of the development of cost-effective and eco-friendly water treatment methods.[3]

The objective of this study was to confirm if waste aluminium beverage cans can be recycled to form potash alum. The experimental process was conducted with concentrated H_2SO_4 and KOH solutions. The pieces of the aluminium can were dissolved in KOH solution. The solution was then subjected to H_2SO_4 solution and afterward crystallized by cold water. The experiment results revealed that alum percentage yield is 80% by utilizing 5 g aluminium cans. XRD, EDX and FTIR spectra of experimentally prepared alum and commercial alum confirmed that both the alums are of same composition and of high purity in essence.[4] Potash Alum was synthesized from scrap cans and its effectiveness for wastewater treatment was ascertained. The fragments of the can were broken down and dissolved in 2.5M Potassium hydroxide and then filtered out the plastic and color coats. The fragments of the can were then chemically and step wisely transformed to alum-a feasible double salt. The prepared alum was qualitatively and quantitatively analyzed later for the ion type and quantity present, coagulation efficiency, and acidity/alkalinity were also ascertained. The alum that is synthesized has coagulation property and possess all other characteristics any alum would have.[5]

CHAPTER 3: METHODOLOGY

Methodology: Synthesis and Characterization of Alum from Aluminium Can Waste

3.1 Materials and Reagents

Materials and reagents required for the preparation and subsequent characterization of alum from aluminium can waste include the following:

3.1.1 Dried and cleaned waste aluminium can

3.1.2 Potassium hydroxide pellets

3.1.3 Concentrated sulfuric acid (H_2SO_4)

3.1.4 Potassium sulphate

3.1.5 Distilled water

3.1.6 250 mL and 500 mL beakers

3.1.7 Conical flask

3.1.8 Measuring cylinder

3.1.9 Filter paper and funnel

3.2 Preparation of Aluminium Feedstock

The can of aluminium is utilized as raw material for alum synthesis. Before initiating the chemical process, the can needs to be cleaned and cut:

The aluminium can is washed thoroughly first to remove any remaining contents.

The layer of paint that is printed on the surface is removed mechanically with sandpaper or chemically with a mild acid. The washed aluminium sheet is then sliced into small squares (about 1 cm^2) using scissors. This makes the surface area bigger and maximizes reaction efficiency with the alkaline solution.



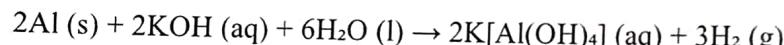
Fig.1. Small cut pieces of cans (10 gms)

3.3 Aluminium to Potassium Aluminate conversion

The chemical alteration of elemental aluminium to soluble aluminate ions via a strong alkali, potassium hydroxide, is the next step:

Approximately 2 g of aluminium chips is added to a beaker of 50 mL of 3 M KOH solution. Solution is stirred very slowly and gently heated on a hot plate or Bunsen burner. The use of a fume cupboard is necessary due to the production of hydrogen gas.

The hydroxide ions react with aluminium to produce potassium aluminate and hydrogen gas:



The reaction goes until all observable pieces of aluminium are dissolved. The solution thus produced is slightly cooled and filtered in order to remove any insoluble impurities or coating residues.

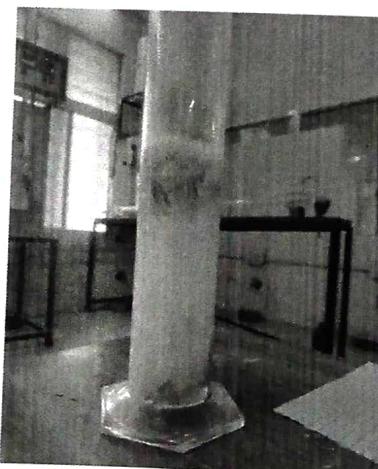


Fig.2 Grey blackish solution(500ml)

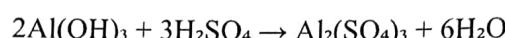
3.4 Acidification and the Precipitation of aluminium Hydroxide

To isolate aluminium from the aluminate solution, acidification by sulfuric acid is done. The process consists of both redissolution and precipitation reactions:

The transparent aluminate solution is poured into a pure beaker. Slow and dropwise addition of concentrated sulphuric acid with continued stirring is carried out to prevent much heating and splashing. White gelatinous aluminium hydroxide precipitate is initially developed:



Upon the addition of additional acid, the precipitate begins to dissolve slowly to give aluminium sulphate:



The amount of acid is then regulated to provide for complete precipitation and the formation of a clear solution of aluminium sulphate.

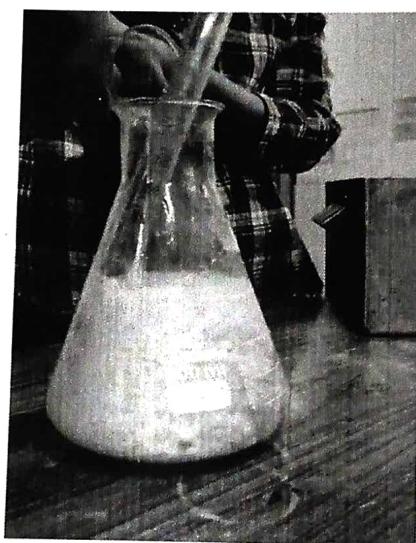
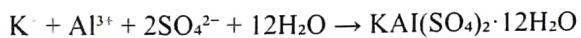


Fig.3 Gelatinous precipitate

3.5 Crystallization and Formation of Alum

Potassium sulphate is added to the acidified solution of aluminium sulphate in order to cause its crystallization:

A stoichiometric amount of K_2SO_4 (around 2 g) is also added to the solution and mixed until it is dissolved. The mixture is gently heated slowly up to almost boiling to dissolve all the ingredients fully. The clear hot solution then is left undisturbed while being cooled to room temperature. For more vigorous crystal growth, the beaker is placed in an ice bath or refrigerator to promote slow and uniform nucleation. Within a few hours, colourless, transparent octahedral crystals of potassium aluminium sulphate dodecahydrate (alum) will have formed:



The crystals are collected by filtration, washed with small amounts of cold distilled water to remove surface impurities, and dried between filter paper or in an oven at low temperature ($\sim 40-50^{\circ}C$).

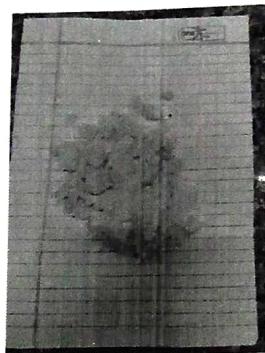


Fig.4 Alum crystals(175gms)

3.6 Yield Determination

The dried alum crystals are weighed on an electronic balance. Theoretical yield is calculated based on the initial mass of aluminium used and the percentage yield determined as:

MATERIAL	QUANTITY	PRICE
Electricity required	1unit	3.34rupees (Source MPEB)
KOH	28gms	2.24rupees(Source Indiamart)
H ₂ SO ₄	78.4gms	1rupee (Source Indiamart)

Our investment--6.58rupees

Price of alum formed(175gms)- 17.5rupees

CHAPTER 4: RESULT & DISCUSSION

A great example of an oxidation-reduction or redox reaction is the dissolution of Al(s) in aqueous KOH. The hydrogen of KOH or water is reduced from an oxidation number of +1 to zero in hydrogen gas, and the metal Al is oxidised to aluminium with an oxidation number of +3. The aluminate complex ion Al(OH)_4^- is referred to as "aluminate." The H^+ ions of sulfuric acid neutralize the base Al(OH)_4^- when there is an acid-base reaction between the aluminate ion and sulfuric acid. When additional acid was added, the white, thick gelatine precipitate of aluminium hydroxide, Al(OH)_3 , was dissolved. The solution then contains Al^{3+} , K^+ , and SO_4^{2-} ions from potassium hydroxide [43]. It is highly likely to recycle waste bins made of aluminium and may be of immense benefit to individuals, towns, organisations, businesses, and industries collectively in the environment, economy, and community. Aluminium cans are already being recycled to make more aluminium products, such as alum, consuming 95% less energy compared to refining and smelting bauxite ore. Alum is a chemical compound employed in a variety of applications including water treatment, deodorant, baking powder, gelling gelatine, plaster cast hardening, and medicinal astringent. The alum crystals were precipitated out of solution by gravity filtration and then rinsed afterwards in a ethanol :water mixture. This acts to filter out impurities from the crystals, and acts to dry out the crystals in a speedy manner.

CHAPTER 5: CONCLUSION

Aluminium is one of the most durable materials used in metal packaging. The largest source of waste aluminium is used beverage cans. These can be recycled to become useful alum through crystallization process. Crystallization is natural or artificial process of solid formation from a solution precipitating. Swenson Walker crystallizer is the most efficient scrapper surface crystallizer which comprises the provision of heating as well as jacketed cooling system. We recycled Around ten cold drink cans which yield 80 gm aluminium using this technique and obtained 76 gm of octahedral alum crystals with size ranging from 1.5 to 3 cm. The percentage of recovery was 95% while the yield of crystal was 35%. A recovery of twenty rupees/76 gm of crystal alum was obtained. Swenson walker crystallization proved to be better than the normal laboratory procedure with regard to percent recovery and yield obtained.

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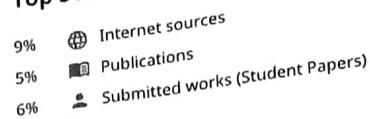
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