

# The Optimization of $\gamma$ -Al<sub>2</sub>O<sub>3</sub> Production from Aluminium Foil Waste by Precipitation

Nessren M. Farrag\*, Yousef M. Ibrahim

The British University in Egypt, Faculty of Engineering, Chemical Engineering Department, Egypt  
 Nessren.farrag@bue.edu.eg

The objective of this work encompasses the application of aluminium foil waste for preparing alumina (Al<sub>2</sub>O<sub>3</sub>), by precipitation. The response surface approach is developed for the produced  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase. Experiments were performed according to a 32 factorial design to evaluate the effects of hydrochloric acid (HCl) concentration, sodium hydroxide (NaOH) that was used as precipitation agent, as well as the calcination temperatures on the purity and yield. The effect of the two independent variables on the response variables was studied by response surface plots and contour plots generated by the Design-Expert software. The desirability function was used to optimize the response variables. The compatibility of the resulted purity and yield of the prepared alumina was further investigated and analysed by EDX test that results more than 97% for the three samples which calcined at three different temperatures. The study of the evolution of crystalline phases of obtained powders was accomplished through different studies. The morphology of the obtained alumina was tested by using SEM test. The observed responses taken agreed with the experimental values, and  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> phase was produced with less experimental trials, and a high yield was achieved with the concept of formulation by design.

## 1. Introduction

The tremendous amounts of solid wastes from domestic uses or industrial activity have been exponentially increased. Aluminium foil is involved in many applications inside homes, auto-mobile parts, and isolation purposes. Despite all its applications, it has severe harsh effects on the environment after being used and disposed. Aluminium foil as a solid waste is mainly disposed in landfills, its accumulation over time produces harmful leachate and gases. So, the principles of solid waste management should be carefully considered to avoid harming the environment. This research mainly addresses the idea of recycling aluminium foil scrap instead of being disposed to produce valuable products (Hafez et al., 2018).

### 1.1 Aluminum foil recycling (Secondary smelting)

Recycling of aluminium foil scarp is known also as secondary smelting or production, and it is cheaper than the production of the material from start. Additionally, it uses less amount of energy, machinery, and relatively milder operating conditions. Firstly, large batch is collected, then washed and sorted to be melted together and finally purified and separated to be ready for practical applications (Padamata et al., 2021).

### 1.2 $\gamma$ -Alumina as a catalyst

Alumina is one of the most valuable products that can be generated from aluminium foil recycling. It has different complex polymeric phases, each has its own distinguish property or specific application i.e., Alpha ( $\alpha$ ), beta ( $\beta$ ) and gamma ( $\gamma$ ) Alumina catalyst. Alumina phases are produced by thermal treatment of alumina, the most important phase for industrial application is the gamma alumina phase (Gholizadeh et al., 2022).

Gamma alumina shows has high melting point over 1500 °C, high chemical resistance for oxidation and corrosion and large surface area compared to other phases. Gamma phase is mainly employed as a catalyst in many reactions and industrial processes Operating conditions such as pressure and temperature can occur at

lower sets in the presence of gamma alumina catalyst to increase the productivity at reduced costs (Niero et al., 2022).

## **2. Experimental set-up**

### **2.1 Pretreatment of aluminum foil waste**

Aluminium foil waste was collected from different restaurants in the area for the aim of performing the experimental work. The waste contained organic and inorganic substances that should be segregated and treated prior any experiment. So, the waste was passed over manganate several times to insure of removal of all metal traces like iron from the samples. The samples were washed well using distilled water to remove all organic and inorganic wastes attached. The washing process was repeated three times to ensure the removal of all contaminants. Then after washing, each sample was dried either by vacuum or by using clean cotton cloth to remove water droplets to avoid its oxidation. The clean samples were sent to a local blacksmith store that has Electric Mill Grinder (End Mill Grinder 3000 W); the grinder was used to reduce the particles size of samples into fine powder form to increase the speed of digestion reaction. The methodology is used to reuse aluminium foil to produce aluminium that in particular may be used for catalyst production (Srivastava and Meshram, 2023).

### **2.2 Aluminum foil waste digestion**

The standard weight of each sample was fixed at 5 g while different acid concentrations were employed for the optimization purpose. The acid used in digestion was the concentrated hydrochloric acid. The samples reacted with the acid in 24 runs as developed by the factorial design that will be discussed in the next section. The batch volume was fixed at 125 ml. The sample with the acid were left on a magnetic stirrer (fixed at 30 rpm) to digest the mixture of pulverized solid and acid faster. The reaction was spontaneous and exothermic where the sample react rapidly with concentrated hydrochloric acid at room temperature to produce aluminium trichloride and emit large amount of hydrogen gas. Hydrogen gas was vacuumed to avoid its severe effects. The reaction temperature retained to room temperature after the reaction (Niero et al., 2022).

### **2.3 Filtration and precipitation**

The obtained solution of digested alumina was filtered using vacuum filter (Model: VP115/American) and DR-filter paper 24 cm to speed the filtration process and to remove any impurities or traces of unreacted metals. The solution obtained must has decent degree of transparency. Sodium hydroxide was used as precipitating agent for the precipitation stage. Different pH values were studied for different runs to obtain the optimum values. The reaction sodium hydroxide and digested alumina continued until aluminium hydroxide was produced and recognized as a viscous white gel. Also maintaining the reaction mixture at low rpm prevent the dissolution of the white gel formed. The white gel obtained was extracted using DR-filter paper 24 cm and 18 cm while washing the gel with enough distilled water until all the traces of sodium chloride is removed. After filtration, the gel was put onto electric oven at 110 °C for about 2 h for drying and to be prepared for the calcination step (Rahman et al., 2023).

### **2.4 Calcination**

Calcination is a thermal treatment method that convert aluminium hydroxide obtained from last step into gamma phase alumina. The gamma phase alumina is the first phase to be obtained from the thermal treatment process and it can be obtained at temperature range between 550 °C – 800 °C. After the white gel was completely dried, it was put in an evaporating dish to withstand the high temperatures. The yielded gel was crushed into fine powder. High temperature Muffle Furnace (laboratory PT-1700M) was used and set for the calcination temperatures (Gholizadeh et al., 2022).

### **2.5 Experimental design**

The preparation of alumina from waste aluminium foil by precipitation is affected by several process variables. Three main variables have significant effect on the purity and yield of the prepared alumina: the concentration of hydrochloric, pH value from sodium hydroxide addition and the calcination temperature in the calcination step. Experimental design software (Design Expert version 11) was used to develop an experimental factorial model to provide an optimization model for research objectives. Experimental methodology was prepared by factorial design to investigate the effect of factors on the response surface which is in the proposed research the yield of alumina. The pH values inserted were 1, 8 and 13, for hydrochloric acid levels inserted were 1, 3 and 5 M and for calcination temperatures were 550, 675 and 800 °C. 24 runs were developed at the different levels as reported in table 1.

Table 1: Experimental runs from design expert

Run	pH	HCl concentration (M)	Temp. (°C)
1	13	1	550
2	13	5	550
3	13	3	800
4	3	3	800
5	8	5	675
6	13	5	675
7	8	1	675
8	3	5	675
9	8	1	800
10	13	3	675
11	8	3	800
12	3	3	550
13	13	5	800
14	8	5	800
15	8	5	550
16	8	3	550
17	3	5	800
18	13	3	550
19	8	1	550
20	3	1	550
21	3	1	800
22	3	1	675
23	3	3	675
24	13	1	675

### 3. Effect of process variables interactions on the yield of alumina

The Design Expert was used to develop graphical illustrations between the process variables to show the significance of each variable on the process. 3D surface plots and contour plots were used to investigate the potential relationship between the three variables (while holding other variables constant if any). Figure (1) shows a 3D plot to study the response of alumina yield in grams with the pH and the concentration at 800 °C. While Figure (2) shows a contour plot to study the response of alumina yield with the pH and the concentration. Table (2) concluded the actual yield obtained versus the predicted values for each run.

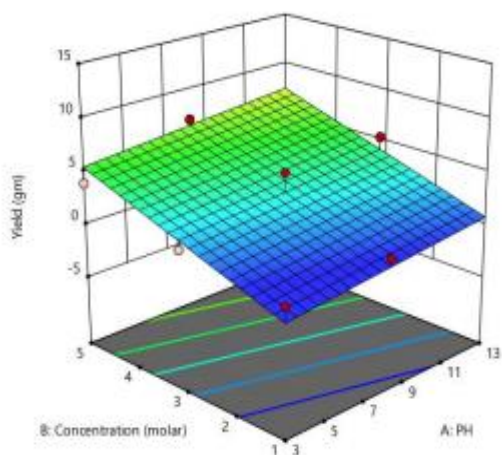


Figure 1: Alumina yield surface plot with concentration and pH

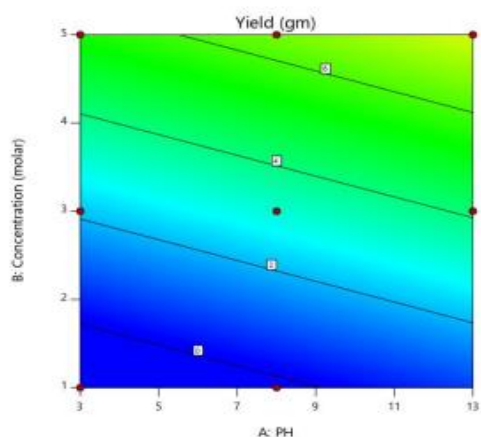


Figure 2: Alumina yield contour plot with concentration and pH

Table 2: Experimental runs from design expert

Run	pH	HCl concentration (M)	Temp. (°C)	Actual yield	Predicted yield
1	13	1	550	0.4480	1.63
2	13	5	550	10.12	8.34
3	13	3	800	5.35	4.12
4	3	3	800	1.28	2.15
5	8	5	675	8.83	6.92
6	13	5	675	6.50	7.91
7	8	1	675	0.3115	0.2095
8	3	5	675	5.48	5.93
9	8	1	800	0.5118	0.2215
10	13	3	675	3.71	4.55
11	8	3	800	4.98	3.13
12	3	3	550	1.09	3.01
13	13	5	800	5.10	7.48
14	8	5	800	7.08	6.49
15	8	5	550	10.43	7.35
16	8	3	550	3.26	4.00
17	3	5	800	3.96	5.50
18	13	3	550	4.79	4.98
19	8	1	550	0.3303	0.6405
20	3	1	550	0.1452	0.3469
21	3	1	800	0.1998	1.21
22	3	1	675	0.1328	0.7779
23	3	3	675	0.9541	2.58
24	13	1	675	0.5993	1.20

### 3.1 Effect of pH value

The value of the pH has significant effect on the preparation process of alumina as it's governed by the addition of sodium hydroxide on alumina trichloride solution with continuous stirring to mix the solution. It was observed that the amount alumina produced is increasing as pH value increases till it reaches 9, the amount produced decreases gradually till reaching 13. If the pH value exceeds 13, aluminium hydroxides will dissolve in the solution and the reaction equilibrium is reversed toward producing the reactant.

### 3.2 Effect of concentration

Hydrochloric acid concentration is an important parameter that effect on the yield of alumina as its used to digest and transform aluminium to aluminium trichloride, and it has a direct effect on increasing the yield of alumina in range of molar concentration between 1-5. Runs performed at lower concentration (below 5 M), takes longer time for the reaction to take place as well as decrease the yield.

### 3.3 Effect of calcination temperature

Temperature is an effective variable on the alumina yield and its purity, however it's not a major factor. It is used during thermal treatment to convert aluminium hydroxide into gamma phase alumina. The calcination temperature ranges by which gamma phase alumina is obtained lies between 550-800 °C and has major effect on the quantity and the purity of the yield of alumina.

## 4. Solid characteristics

The characteristics of the prepared alumina samples was determined by performing EDX and SEM tests. The tests were performed on the optimum run prepared which is run 15 in table 1. The sample was prepared under high precision and accuracy to insure high yield productivity. EDX test was particularly selected to determine qualitatively the chemical composition of the sample (Hellmy and Azida, 2022), as shown in Figure (3).

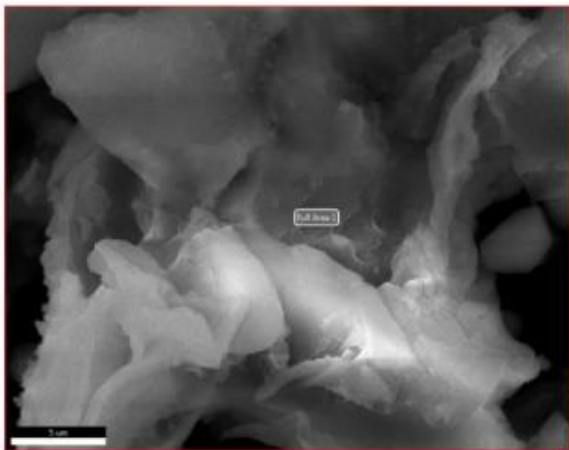


Figure 3: EDX test for alumina particles

Also, SEM analysis was performed on the same sample to identify the morphology of the sample particles. SEM test provided a microscopic image as shown in Figure (4). Particles shown have spherical elongated shapes with quite tendency to form a clot that consist of many round edges and corners, that is correlated to alumina structure morphology (Hellmy and Azida, 2022).

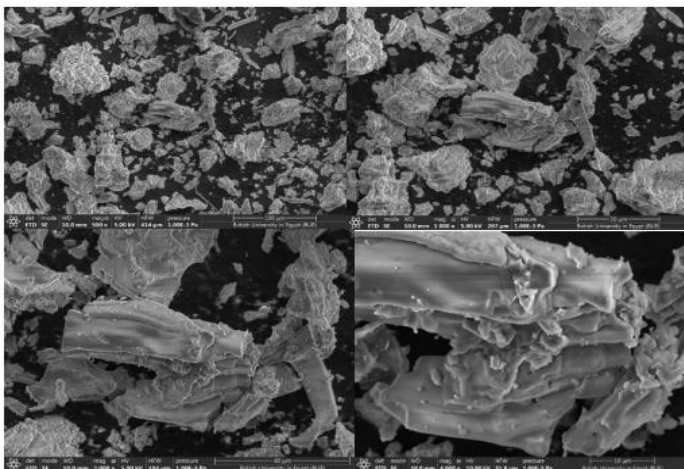


Figure 4: SEM test for alumina particles

## 5. Conclusion

This study has delivered the preparation and production of alumina catalyst as a valuable product obtained from the environmental threatening wastes of aluminium foil. The benefit from this research is not only to produce a valuable product from waste but also to provide a significant environmentally friendly solution against the accumulation of aluminum foil scrap. Alumina catalyst is a valuable product with high stability, inertness and catalytic ability that is chosen for many industrial applications as water treatment, glass indurates and hydrogen generation. Alumina can be prepared by different techniques, however in this study the precipitation method was selected for its higher productivity and simpler operation. The production of alumina catalyst from aluminium foil waste feedstock is mainly controlled and driven by three process variables: the pH value, the HCL concentration and the calcination temperatures. The Design-Expert software has been used to develop 24 experimental runs with different variables' interactions. The model developed fits liner model between the process variables and the yield of alumina produced in grams. The results show that the pH value and the acid concentration have high significant effect on alumina yield, however the calcination temperature is insignificant. 3D surface plot and contour graph have been represented to show the factors' interactions and their effect on the yield. Finally, the solid chemical and physical characteristics of the sample prepared by the optimum conditions have been analysed and verified using SEM and EDX tests.

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## References

- Gholizadeh Z., Ghominejad M., Shariatmadar Tehrani F., Aliannezhadi M., 2022, High Specific Surface Area  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> Nanoparticles Synthesized by Facile and Low-cost Co-precipitation Method High Specific Surface Area  $\gamma$ -Al<sub>2</sub>O<sub>3</sub> Nanoparticles Synthesized by Facile and Low-cost Co-precipitation Method. <https://doi.org/10.21203/rs.3.rs-2085977/v1>
- Hafez H. H. A., El-Salam A., Huseen A. M., 2018, STUDIES ON THE EFFECT OF ALUMINUM, ALUMINUM FOIL AND SILICON BAKED CUPS ON ALUMINUM AND SILICON MIGRATION IN CAKES. In *J. Agric. Res* (Vol. 96, Issue 2).
- Hellmy M., Azida N., 2022, Selective visualization techniques for elemental mapping analysis of granite stones by field emission scanning electron microscopy and energy dispersive spectroscopy (FESEM-EDXS). *Materials Today: Proceedings*. <https://doi.org/10.1016/j.matpr.2022.11.315>
- Niero D. F., Montedo O. R. K., Bernardin A. M., 2022, Synthesis and characterization of nano  $\alpha$ -alumina by an inorganic sol-gel method. *Materials Science and Engineering B: Solid-State Materials for Advanced Technology*, 280. <https://doi.org/10.1016/j.mseb.2022.115690>
- Padamata S. K., Yasinskiy A., Polyakov P., 2021, A Review of Secondary Aluminium Production and Its Byproducts. In *JOM* (Vol. 73, Issue 9, pp. 2603–2614). Springer. <https://doi.org/10.1007/s11837-021-04802-y>
- Rahman M. L., Islam M. S., Ahmed M. F., Biswas B., Sharmin N., Neger A. J. M. T., 2023, Extraction and characterization of highly pure alumina ( $\alpha$ ,  $\gamma$ , and  $\theta$ ) polymorphs from waste beverage cans: A viable waste management approach. *Arabian Journal of Chemistry*, 16(2). <https://doi.org/10.1016/j.arabjc.2022.104518>
- Srivastava A., Meshram A., 2023, On trending technologies of aluminium dross recycling: A review. *Process Safety and Environmental Protection*, 171, 38–54. <https://doi.org/10.1016/j.psep.2023.01.010>