

## Lead Ions Absorption Kinetic Study by In-house Prepared Activated Alumina in Aqueous Solution

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### Abstract

River and marine lead (Pb) concentrations are increasing. Lead and other heavy metals in drinking water have been associated with health problems worldwide. It is imperative that levels be lowered. Hence, in order to remove lead (Pb) ions from water, this study investigated the use of activated alumina as an adsorbent. The prepared precursor was then processed to create powdered activated alumina from the activated alumina. Response surface analysis was used to examine the effects of pH, NaOH, H<sub>2</sub>SO<sub>4</sub>, DI (H<sub>2</sub>O), and Al (powder) on the quality and yield of activated aluminium. According to the pattern, more aluminium Micropore volumes for the activated alumina were 0.000821 cm<sup>3</sup>/g, 0.00221 cm<sup>3</sup>/g, and 0.002203 cm<sup>3</sup>/g. Alumina that has been activated was used to gauge the heavy metal content's decline over time. The results of the kinetic analysis showed and the experimental design were able to capture the behavior of the study parameters.

**Keywords**—*activated alumina, kinetic study, absorption capacity*

### INTRODUCTION

Fast population growth, vast industrial expansion, rapid urbanisation, rising energy consumption, and waste generation from residential and industrial activities have all affected many water sources, poisoning them with heavy metals and other harmful toxins [1]. Water contamination has captured the world's attention since it affects both humans and the environment [2]. Due to their closeness to the sea and rivers, as well as the rising industrialisation of South Asian nations such as Malaysia [3], Asia's regions are especially heavily impacted. Chemical, physical, and biological classifications are used to classify wastewater [4]. Water contamination has increased significantly in recent years as a result of rapid industrialization, urbanisation, and



agricultural expansion, all of which has resulted in increased usage of hazardous chemicals [5]. This rapid expansion has resulted in widespread degradation of soil, plants, and, most importantly, increasing fouling of aquatic bodies [6]. Water pollution is a widespread problem. This problem has become progressively worse over time [7]. The majority of underdeveloped nations just recently recognised the problem, since such recognition frequently takes a long time. Implementing the necessary preventive measures takes much longer. Diverse reports and worries about industrial waste dumping and odorous rivers inside densely populated cities were early expressions of water pollution [8]. Today, the world is dealing with serious water contamination issues that have been linked to industrial activities such as mining and waste management practises such as discharging toxic metallic waste into freshwater, contaminating ground and surface water and resulting in soil, air, and ground pollution [9]. Other sources of harmful metal ions have been found, including agricultural-based industrial activities such as rubber and oil palm mills, household and animal farms, and sewage treatment facilities [10]. Some admirable efforts have been made in Malaysia, and to emphasise further, the department of environment has made major efforts to assure implementation, culminating in the 1974 enactment of the Environmental Quality Act, which is a viable choice. Since then, a total of 111 instances of water pollution from point sources have been documented, with 48 percent of pollutants coming from sewage treatment facilities, 4 percent from industrial businesses, 5 percent from animal farms, and 9 percent from agricultural industries, specifically rubber and oil palm mills. According to some accounts, the principal drivers of heavy metal enrichment in water reservoirs are man-made causes such as poorly planned urban expansion and agricultural runoff, as well as human sources of rock weathering and volcanic activity [12]. Today, surface water contamination has been seen in various locations around Malaysia, and water quality is degrading as a consequence of waste water discharged containing extremely harmful pesticides and herbicides, mostly from rice farms. The bulk of these pesticides and herbicides include heavy metal ions, which are hazardous to both humans and animals, especially aquatic organisms such as fish that are eaten directly by humans, resulting in health concerns [13]. The quality of fresh water in Malaysia's

major rivers and dams has deteriorated further as a result of heavy metal discharge from different industries, notably in industrial zones such as Perak. According to several experts, 340 rivers in the country have already exceeded the 0.01 mg/l lead (Pb) guideline. According to a recent study, the Tasoh dam in Malaysia's Perlis region is heavily contaminated by metal-rich wastes. The exposure of lead-related compounds during the initial mining operations in 1975 resulted in this acute pollution. This mine's runoff has high amounts of many metals, most notably lead, which is a consequence of the flotation process used to create iron concentrates [57]. As a consequence of this study, a low-cost approach for producing activated alumina for the removal of lead ions from aqueous solution was given. This study was done based on the experimental design aided by RMS software.

### Methods

Materials were prepared and samples of aluminium oxide (AL<sub>2</sub>O<sub>3</sub>) and all other chemicals were obtained from Meridian World Sdn. Bhd., which is one of the environmental service providers in Sungai Petani, Kedah Darul Aman, Malaysia, and which has a high quality standard, as shown in TABLE I **MATERIAL AND PROPERTIES**

NaOH	<b>Name: Sodium Hydroxide Solution</b> <b>Purity: 50% w/w/Certified</b> <b>Brand: CAS: 1310-73-2</b>
DI Water	<b>Name: Deionized Water</b> <b>Laboratory grand</b> <b>Purity: 99%</b>
H <sub>2</sub> SO <sub>4</sub>	<b>Name: Sulfuric acid</b> <b>Brand: CASRN 7664-93-9</b> <b>Purity: 98%</b>
AL <sub>2</sub> O <sub>3</sub>	<b>Name: Aluminum oxide</b> <b>Purity: Purity : 99.9%</b>

The response surface approach was used in order to develop the experiment, and design expert 10 was used to accomplish so. The design gave exactly 27 runs of the optimised experiment, and the parameters were determined as a result of the design. Prior to the preparation of the tests, pure aluminium with a purity of 99.9 percent was procured for use in the research. Aluminum (99.9 percent pure) was first combined



and reacted with aqueous sodium hydroxide (NaOH) at room temperature in a conical flask to generate a solution of sodium aluminate, which was then dried at room temperature (NaAlO<sub>2</sub>). The solution includes some dissolved contaminants that must be removed by filtration. Al(OH)<sub>3</sub>·xH<sub>2</sub>O was precipitated after the solution was thoroughly filtered using a 2 m filter paper, and the clear filtrated solution was neutralised with H<sub>2</sub>SO<sub>4</sub> to pH 4, 6, and 8 to achieve the desired pH. Sulfate ions are formed in the gel as a result of the interaction between the acid and the aluminium. The ions were removed from the gel when it was rinsed. It was rinsed five times in a row until no more sulphate ions could be detected in the solution. Because sulphate ions are often found in colourless solutions, the finding was carried out by physical observation. Finally, they were dried at 80 degrees Celsius for 6 hours before being crushed and put through a sieve with a 60 mesh opening. The samples were then calcined (burned) for 3 hours in an air-cooled muffle furnace at a heating rate of 20 degrees Celsius per minute for 1200 degrees Celsius. The characterisation of the synthesised AA was carried out using BET in order to determine the pore volume, pore diameters, and surface area of the AA. In order to better understand the effects of contact duration, pH, and temperature on the rate of lead removal, kinetic experiments for activated alumina were carried out. The following studies were carried out using activated alumina and three different sample quantities were used. We employed three different volumes of activated alumina and maintained them in contact with the reference solution for varying lengths of time to conduct kinetic experiments on the material (10 mins, 20 mins, 30 mins, 40 mins, 50 mins, and 60 mins). A 100 mL volume of 0.1 ppm lead solution was poured in a round-bottom flask and allowed to stand for 24 hours. Each flask included ten grammes of activated carbon, which was allowed to come into contact with the lead solution for the duration of the experiment. Preliminary and post-kinetic studies were carried out to determine the pH of the lead solution. The impact of temperature was determined by putting the flask on the hotplate at various temperatures ranging from 0-30 degrees Celsius to 60 degrees Celsius, 90 degrees Celsius, 120 degrees Celsius, and 1500 degrees Celsius.



## Results and Discussions

It was chosen to employ expert software to design the experiment in order to save time and money. According to the results of the simulation, the model projected by the programme was based on the highest order polynomials where the extra terms were important and that the models were not aliased. The sequential model sum of squares was utilised to establish the model's shape and function. Model parameters parameters stand for the variables. were estimated using the software, the  $x$  pa, which was a very easy and time-saving method. Alumina yield is equal to  $3 \times 1 - 0.49x_2 + 1.94x_3 + 0.61x_4 + 0.91x_2x_3 + 0.93x_2x_3 + 0.99x_3x_4 - 0.07x_{21} + 11.5x_2x_3 - 0.07x_{21} + 11.53x_1 - 0.49x_2 + 1.94x_3 + 0.61x_4 + 0.91$  The numbers  $4.01x_2x_3$ ,  $3.06x_4$ , and  $5.01x_{25}$  are all multiples of four. While the coefficients with  $(x_1)$ ,  $(x_2)$ , and  $(x_3)$  denote the influence of each individual component on the manufacture of activated alumina, those with  $(x_4)$  denote the combined effect of all three factors. The coefficients with  $(x_1)$  denote the influence of each individual component on the manufacture of activated alumina. It is worth noting that in this example, the interaction between the two variables is represented by coefficients with just two components (such as  $x_1x_2$  and  $x_2x_3$ ), while the quadratic influence is represented by coefficients with second order terms (such as  $x_{2,1}$ ,  $x_2^2$  and  $x_{24}$  and  $25$ ). It is symbolised by a positive or negative sign in front of the words to indicate if they have synergistic or antagonistic effects on one another, accordingly. In many cases, a mathematical model is used to depict the chemical interactions that take place between the components. Distilled water,  $H_2SO_4$ , sodium hydroxide, pH, and aluminium are all used in this recipe (powder). In order to assess the dependability of the expected model, the correlation coefficient,  $R^2$ , and standard deviation values may be calculated for the anticipated model. The connection between the sum of squares (SSR) and the total sum of squares (SST) is denoted by  $R^2$ , and it has been shown to be relevant in assessing how well the models mirrored the experimental data. For each of the four equations, as indicated in Equations 1, 2, and 3, the  $R^2$  values were 0,91 (096), 0,99, and 0.94, respectively, ensuring that the models developed are properly matched to the experimental data. As well as this, Table 2 offers a list of other statistical parameters that were used to determine whether or not the models built were adequate.



**TABLE II STATISTICAL PARAMETERS FOR ANOVA ANALYSIS FOR MODEL REGRESSION OF PERCENTAGE YIELD ACTIVATED ALUMINA**

Statistical Parameters	Factors response				
	DI	H <sub>2</sub> O <sub>4</sub>	NaOH	pH	Al <sub>2</sub> O <sub>3</sub>
Standard Deviation, SD%	2.3	1.5	1.4	2.1	1.7
Correlation Coefficient, R <sup>2</sup>	0.91	0.96	0.99	0.94	0.86
Adjusted R <sup>2</sup>	0.90	0.93	0.91	0.92	0.83
Mean	89	86	85	85	83
Coefficient of Variation	3.07	3.47	4.2	3.1	2.47
Adeq. Precision	13	12	14	16	13

The fact that the standard deviations and coefficient of variation were so modest in Table 2 demonstrated that the model was highly reproducible in the field of research. The Adeq Precision, on the other hand, is a measurement of the signal to noise ratio. In order for the complete process to display Adeq Precision more than 10, a ratio larger than 4 is needed. All of the replies had a high correlation coefficient, which indicates that the models may be used to plan the preparation of activated alumina for various applications.

**TABLE III ANOVA ANALYSIS AND LACK OF FIT TEST FOR RESPONSE SURFACE MODEL FOR YIELD PERCENTAGE ACTIVATED**

SOURCE	Sum of Squares	Degree of Freedom	Mean Square	F Value	Prob> F	Comments
MODEL	12208.10	12	12208.10	20.88	<0.0001	Significant
X <sub>1</sub>	972.00	1	972.00	11.32	0.0081	
X <sub>2</sub>	632.00	1	632.00	22.34	0.0051	
X <sub>3</sub>	553.10	1	553.10	22.00	0.0051	
X <sub>4</sub>	352.12	1	352.12	11.50	0.0041	
X <sub>5</sub>	5.233	1	6.22	9.22	0.0003	
X <sub>1</sub> X <sub>2</sub>	8.112	1	8.112	1.530	0.0681	
X <sub>1</sub> X <sub>3</sub>	2.301	1	2.301	4.500	0.0831	
X <sub>2</sub> X <sub>3</sub>	3.500	1	3.500	5.600	0.0891	
X <sub>3</sub> X <sub>4</sub>	32.23	1	32.23	5.711	0.0051	
X <sub>1</sub> <sup>2</sup>	32.00	1	32.00	5.602	0.0051	
X <sub>2</sub> <sup>2</sup>	27.10	1	27.10	5.703	0.0041	
X <sub>3</sub> <sup>2</sup>	38.00	1	38.00	5.618	0.0681	
X <sub>4</sub> <sup>2</sup>	88.20	1	88.20	4.500	0.0831	
Residuals	55.11	11	55.11	3.599	0.0891	
Lack of fit	33.00	3	33.00	7.200	0.0051	
Pure Error	5.5.10	5	5.5.10	6.501	0.0021	Significant



In order to validate the dependability of the constructed models, an analysis of variance (ANOVA) was performed, and the findings obtained are displayed in Tables 3 and 4. It was determined whether or not each model was statistically significant using the F-test value, which is a statistical measure of how well each model predicts variance in the data around the mean. Generally speaking, the bigger the F-value, the more confident one may be in their model's ability to correctly explain data fluctuation, and that their predicted significant terms of the active preparatory variables are closer to the real value. The model F-value for activated alumina is 20.8 at a 90 percent confidence level, which is a good result. This means that these models were notable in terms of their predictions. In addition, the values of Prob > F for the yield were less than 0.05, showing that the model parameters are statistically significant in this case. The parameters for activated alumina preparation include DI (x1), H2O4 (x2), NaOH (3), pH (4), and AL(powder)-x5 are all crucial model parameters, as is the parameter for H2O4 (x2).

**TABLE IV PREPARED AVERAGE ACTIVATED ALUMINA SURFACE AREA**

langmuir surface area	single point surface areas	bet surface area	adsorption cumulative surface area of pores	desorption cumulative surface area of pores
0.3400 m <sup>2</sup> /g	0.3132 m <sup>2</sup> /g	0.3900 m <sup>2</sup> /g	0.4462 m <sup>2</sup> /g	0.4501 m <sup>2</sup> /g

**TABLE V Prepared activated alumina Pore Volume**

Micro pore volume	Adsorption cumulative volume of pores	Desorption cumulative volume of pores
0.000821 cm <sup>3</sup> /g	0.002211 cm <sup>3</sup> /g	0.002203 cm <sup>3</sup> /g

**TABLE VI PREPARED ACTIVATED ALUMINA PORE SIZE**

Adsorption average pore width	Desorption average pore width
198.215 Å	195.746 Å

The surface integrity of synthesis activated alumina is shown in Tables 5, 6, and 7. We were able to get a surface area of 0.3900m<sup>2</sup>/g, which corresponded to a pore volume of 0.002211 cm<sup>3</sup>/g and an average pore width of 198.215 microns for adsorption. In this study, the preparation parameters used for the preparation process

were responsible for the high BET surface areas and favourable pore size distribution of the activated alumina adsorbents that were produced. When it comes to the manufacture of activated alumina adsorbent, the size distribution of the pores is critical. It has been observed that tiny pore size fails to catch bigger adsorbents, while large pore size may collect smaller adsorbents instantaneously but is unable to keep them permanently, resulting in a reduction in the total removal effectiveness of the system. Numbers 5 and 6. It is possible to manufacture macro-porous activated alumina by including a higher proportion of lignin into the precursors, whereas micro-porous activated alumina is produced by incorporating a higher percentage of cellulose into the precursors. According to published research, while microspores have significant surface areas, they account for a much less proportion of the total pore volume in comparison to macropores, which is extensively established in the literature. The semi-step is critical in the preparation of activated alumina because it disrupts the cellulose backbone. The BET surface areas and pore volumes may be increased by boosting the diffusion of NaOH molecules into the pores, which in turn increases the area available to serve as an efficient activation channel for steam or any other sort of activating gas.

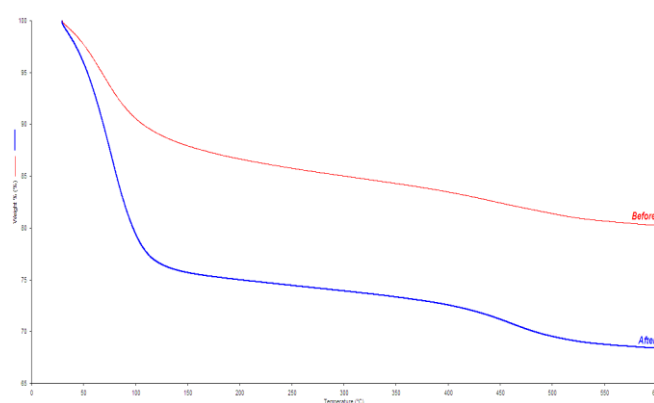


Fig. 1 The weight loss with temperature for activated alumina before and after washed during the process



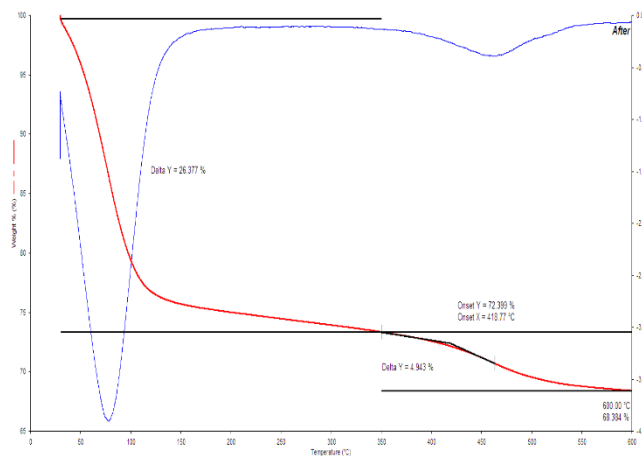


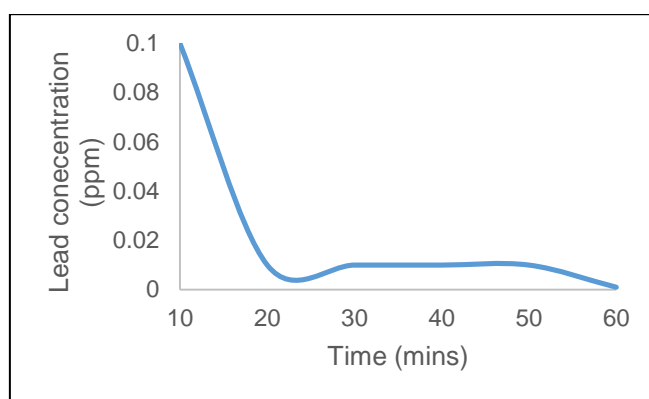
Fig.2 The weight loss with transition temperature for activated alumina

Metal-ion distribution models based on adsorption kinetics may be described by one or more of a number of adsorption-kinetic models, which are all related to the distribution of metal ions between the liquid and adsorbent. They make a determination on the nature of the adsorption process. The equilibrium kinetic data were interpreted using the Langmuir and Freundlich adsorption kinetic models, respectively. The experimental findings from this investigation were strongly correlated with the Langmuir equation, and the benefit of the Langmuir equation over the Freundlich equation had a role in the decision to choose this model rather than another one. Activated alumina is used in the Langmuir kinetic model, which is predicated on the premise that metal ions will adhere to the surface of the material due to adsorption onto a limited number of adsorption sites with uniform energies of adsorption. The classical adsorption kinetics of Langmuir may be stated mathematically as the following equation: (Langmuir, 1916).

$$C_c / q_c = C_c / Q_0 + 1 / Q_0 b$$

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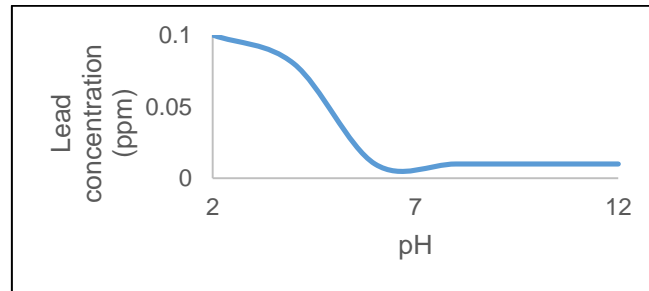
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**Fig. 3 Kinetics adsorption study with 0.1ppm lead solution using 10grams of activated alumina at varying time**

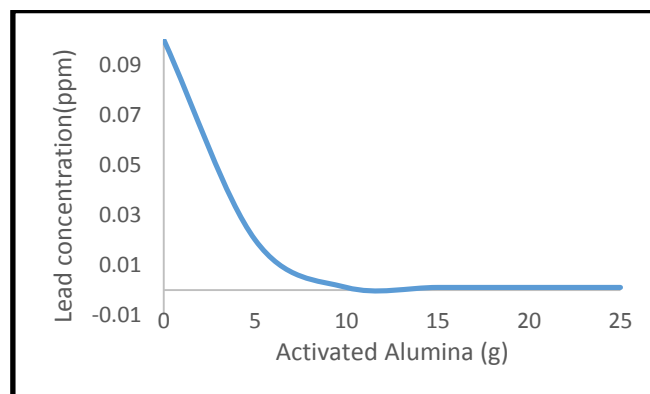
In Fig. 3, the X-axis shows the amount of time in hours, and the Y-axis depicts the amount of lead in the bloodstream. As may be seen in the illustration, the maximum amount of removal is accomplished in twenty minutes (20 min). A little quantity of activated alumina (10 grammes) was sufficient to remove 99.8 percent of the lead from the solution in twenty minutes (20 min). Furthermore, utilising the synthesised activated alumina, it was possible to meet the drinking water standard of 0.05 parts per million (ppm) of lead in twenty minutes. It has already been noted that the elimination of lead ions at various pH levels in solution is represented in figure 3. It is obvious from the figure that the activated carbon was highly successful for the removal of lead ions throughout a pH range of 6.0–7.0 when used in conjunction with other chemicals. Between pH values of one and seven, the concentration of the ion decreases linearly as the pH value rises. This indicates that the adsorption capacity of the adsorbent was pH dependent. The results from kinetic tests for nitrite

ion adsorption onto activated alumina were treated using Langmuir kinetic models, which were developed by the Langmuir group.



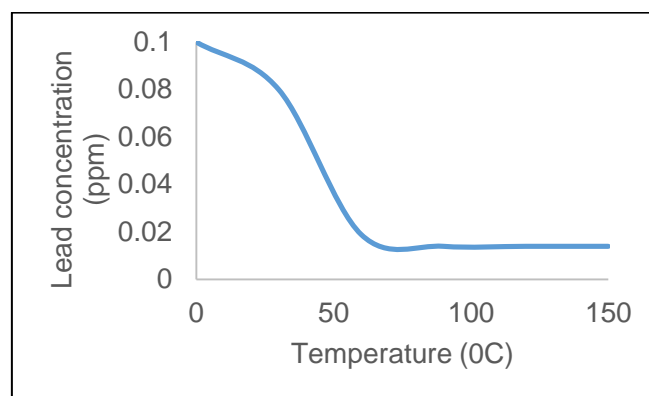
**Fig.4 Kinetics adsorption study with 0.1ppm lead solution using 10grams of activated alumina at varying pH**

Moreover, the influence of pH on the removal rate of lead solution when employing activated alumina (see Fig. 4 for further information). The X-axis indicates the pH of lead solution on a scale ranging from 2 to 12 before treatment with activated alumina, and the Y-axis reflects the concentration of lead before treatment. At pH 7, lead had been removed to a maximum of 99.9 percent of its original concentration. It was discovered that the concentration of lead was 0.01 parts per million (ppm) at a pH value of 7 for an initial concentration of 0.1 parts per million (ppm) lead solution using activated alumina. Beyond pH 7, there was little difference in the removal efficiency.



**Fig. 5 Kinetics adsorption study with 0.1ppm lead solution using varying grams of activated alumina**

Various activated alumina contents were tested for their impact on the adsorption of lead ions, as shown in Fig. 5. As can be observed in the figure, the concentration of lead ions reduced from 0.09 parts per million (ppm) to virtually zero parts per million (ppm) when the activated alumina content was increased from 0 to 25 grammes. The fact that the activated alumina content created in this research has a greater surface area than the adsorbate content may account for the substantially higher adsorption at s adsorbate content seen in this investigation. Also included in the image is the experiment with an initial concentration of 0.1 ppm lead solution on the activated alumina content, which was performed at room temperature. This graph depicts the quantity of active (in grams) and the amount of lead (in milligrams) in a solution. The addition of ten grammes of activated alumina to the solution is sufficient to completely eliminate the lead from the solution. After being treated with 10 grammes of activated carbon, the concentration of lead was below the detection thresholds. Furthermore, with 10 grammes of activated alumina, the drinking standard of 0.0 5 parts per million (ppm) for lead was met.



**Fig. 5 Kinetics adsorption study with 0.1ppm lead solution using 10grams of activated alumina with varying temperature**

To determine effect of the temperature on the removal of lead by the activated alumina, fixed amount of activated alumina (10gram) was kept contact with standard solution of lead with 0.1ppm concentration varying temperature from 0 to 150<sup>0</sup>C. Figure 6 shows kinetics adsorption study with 0.1ppm lead solution using 10grams of activated alumina with varying temperature. Maximum removal is achieved within



50°C. An amount of only 10g of could remove 99 percent of the lead from the solution within 50°C, beyond this temperature there no changes. However, removal mechanism is partially dependent of pH (compared with other researchers).

### CONCLUSION

The performance of activated alumina in the removal of lead ions from aqueous solution was effectively explored in this work. The results were encouraging. For the removal of lead from aqueous solutions, activated alumina is a very efficient absorbent . It was also discovered via this process that 98 percent of the lead was eliminated within 20 minutes of the start of the trial. The removal rate is quite quick during the early stage and continues to be so until the equilibrium is attained after 20 minutes. Temperature, pH, and concentration are all critical elements to be aware of while working with chemicals. When the temperature is raised over 500 degrees Celsius, the proportion of lead ions removed rises. Both the practical and theoretical findings are in excellent agreement with the Langmuir pseudo-second order equation.

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