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Utilization of Ketapang Seed Shells in Reducing $Pb(NO_3)_2$ Metal Levels by Adsorption Process

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Abstract. Lead is a heavy metal that is dangerous if it enters the environment through industrial waste, mining, waste from the electroplating process, burning fuel oil and so on which is carried by streams. Therefore, it is necessary to limit the lead metal content. Efforts to reduce the impact of lead metal pollution can be done in several ways, one of which is by adsorption using adsorbents. Adsorption has been proven to be a fairly effective method for treating liquid waste and is the most commonly used method because it has a simpler concept and can be regenerated and is economical. One of the natural resources around us that contains cellulose that is not utilized is the outer shell of ketapang seeds. The outer shell of ketapang seeds which contains cellulose is a good alternative for utilizing this waste so as not to pollute the environment. To reduce lead metal levels, you can use a natural adsorbent from activated charcoal on the outer shell of ketapang seeds. The adsorption process uses activated charcoal adsorbent, which is one of the most frequently used non-polar adsorbents because it has better adsorption power and surface area than other types of adsorbent. This research aims to determine the variables that influence and the optimum conditions for natural adsorbents from activated charcoal in the outer shell of Ketapang seeds to reduce $Pb(NO_3)_2$ metal levels. The research method used is an experimental method with a two-level factorial design. The research results showed that adsorption temperature was the most influential variable in this research with an optimum yield presentation of 66.77%. The activated charcoal produced has a water content of 8%, an ash content of 5.2% and a methylene blue absorption capacity of 698 mg/g.

Introductions

Indonesia as a developing country is currently filled with various industries. Waste disposal from the industry can pollute the environment, especially waste disposal of heavy metals. Lead is one of the dangerous heavy metals when it enters the environment through industrial waste, mining, waste from electroplating processes, burning fuel oil and so on which is carried away. Therefore it is necessary to limit the content of lead metal [1].

Efforts to reduce the impact of lead metal pollution can be done in several ways, one of which is adsorption using an adsorbent. Adsorption has been proven to be a fairly effective method for treating wastewater and is the most commonly used method because it has a simpler concept and can be regenerated and economical [1]. Along with the development of science, research is currently being developed which aims to find alternative adsorbents based on natural ingredients. One of the natural resources around us that contains cellulose that is not utilized is the outer skin of ketapang seeds. The outer shell of the ketapang seed is produced from the ketapang tree which is found in several regions in Indonesia. The large number of ketapang seeds that fall from the ketapang tree which is used as a shade causes waste in the environment. So far, there are quite a number of ketapang seeds that fall from the ketapang tree, so using the outer shell of the ketapang seeds which contain cellulose is a good alternative for utilizing this waste so it doesn't pollute the environment.

Metal pollution basically does not stand alone, but can be carried by water, soil and air. If all of these components have been contaminated by inorganic compounds, then they may contain various heavy metals such as Cr, Zn, Pb, Cd, Fe and so on. Lead (Pb) has a negative impact on health, especially if the levels exceed the levels required by body. Even at low concentrations, the effects of heavy metal ions can have a direct effect and accumulate in the food chain. like other sources of environmental pollution, these heavy metals can be transferred over very long distances in the environment [2].

Several methods that can be used to reduce the concentration of heavy metal ions in liquid waste include adsorption, precipitation, ion exchange using resin, filtration, and by absorbing pollutant materials by adsorbents in the form of synthetic resins. and activated carbon [3]. Among these methods, adsorption is the most commonly used method because it has a simpler concept and can be regenerated and is economical.

Adsorption techniques for heavy metals have been widely carried out using various kinds of adsorbents, namely [4] conducted research on making active carbon from rice husks with H_3PO_4 activator, [5] also conducted research on guava bark as an adsorbent for lead metal.

Furthermore, [6] researched the manufacture of adsorbents through the use of corn stalks as activated charcoal using sulfuric acid activator with its use in the absorption of copper (II) ions and the use of palm oil shells as an adsorbent through physicochemical activation [7].

So far, there are ketapang fruit that use the seeds as a substitute for almonds in making cakes [8] and there are also those who use them to extract oil from ketapang seeds as a basic ingredient for biodegradable monodiglyceride surfactants [9] so that the outer skin is wasted. To increase the economic value of the outer skin of ketapang seeds, this can be used as activated carbon because organic waste from plants is a source of carbon such as cellulose, hemicellulose and lignin.

Several studies on the manufacture of ketapang adsorbents have been carried out, including by examining ketapang leaves as an adsorbent [10]. The way of working in the research was carried out with 3 procedures. The first is the preparation of bioarang by cleaning the ketapang leaves and drying them, then pulverizing them using a blender and sifting them with a 100-200 mesh sieve, after that the carbonization process is carried out in the furnace at a temperature of 300ºC with time variations of 30, 60 and 120 minutes.

The second way of working is to determine the moisture content and ash content, the water content is determined by weighing the charcoal weighing 1 gram after that it is placed in the oven at 105ºC for 4 hours, then the charcoal is cooled in a desiccator and weighed until a constant weight. The ash content is determined by weighing 1 gram of charcoal and then placing it in the furnace at 750ºC for 6 hours, when all the charcoal has turned to ash, then cool it in a desiccator and then weigh it.

The third way of working is adsorption of methylene blue by bioarang from ketapang leaf waste, at the time of contact absorption as much as 0.1 gram of bioarang is put into Erlenmeyer and added 20 mL of 10 ppm methylene blue solution and stirred using a water bath shaker at 30ºC at 120 rpm speed with varying times of 5, 7, 10, 15, and 20 minutes, then the stirring solution was centrifuged for 10 minutes at a speed of 3000 rpm after that the filtrate was allowed to stand for 30 minutes and after the charcoal had settled, a number of the filtrate was taken and the adsorption was measured using a spectrophotometer.

The results of this study indicate that the adsorption capacity is not affected by the water content but the ash content because the lower the ash content the greater the adsorption capacity [10].

In this research, the outer shell of ketapang seeds will be examined as an adsorbent. In contrast to previous studies, namely using ketapang leaf adsorbents to remove pollution from dyes using methylene blue. In this study, the outer shell of ketapang seeds was used as an adsorbent to reduce levels of $Pb(NO_3)_2$ metal. The ketapang seed outer shell adsorbent can be obtained by chemical activation with HCl activator, and physically obtained by carbonization.

This substance will activate the carbon atoms which will enter the pores and open the closed surface of the charcoal, so that the impurity compounds in the pores become more easily absorbed and the surface area of activated carbon increases to increase its absorption power. The advantage of this process is that it can reduce environmental pollution because the residues in the form of oxides that are not soluble in water when washing are neutralized with NaOH to bind back chemical residues attached to the surface of activated charcoal and the ash content contained in activated charcoal.

The aims of this study were: (i) to find out the variables that most influenced the reduction of metal $Pb(NO_3)_2$ levels, (ii) to determine the optimum conditions of the variables that affected the process of implementing the natural adsorbent of the outer shell of ketapang seeds on decreasing levels of metal Pb($NO_3)_2$, (iii) To determine the activated charcoal produced according to the Indonesian National Standard (SNI) with the parameters of water content, ash content, and absorption capacity of methylene blue.

Ketapang. Ketapang (Terminalia cattapa) is a tropical almond tree that is widely distributed in tropical climates and is often used as a shade tree in parks, on roadsides and on beaches. Ketapang trees can grow to a maximum height of 25 meters and a leaf canopy width of 9 meters. The leaves of the ketapang tree are oval-shaped, green in color, turn yellow and brown when they fall [11].

Fig. 1. Ketapang Seed Skin [12]

Ketapang seed coat is divided into 2, namely the outer skin layer (testa) and the inner skin layer (tegmen). The outer skin layer on the ketapang seeds is hard like wood. This layer is the main protector for the part of the seed inside. The outer shell of the ketapang seed or the shell of the ketapang seed can be used to make briquettes or activated charcoal because it has a carbon element.

Table 1. Composition of Ketapang Seed Outer Skin [13]

Komposisi	**Presentase**
Selulosa	16,60%
Hemiselulosa	24,70%
Lignin	43,46%

Lead (II) Nitrate. Lead (II) Nitrate is an inorganic compound with the chemical formula $Pb(NO_3)_2$. This compound is generally found as colorless crystals or white powder [14] and is toxic to the human body because lead at a temperature of 550-600°C can evaporate and react with oxygen [15]. Lead in the elemental arrangement is a heavy metal that occurs naturally in the earth's crust [16]. Lead metal is a type of heavy metal that is usually contained in industrial wastewater and can cause serious problems because of its toxic nature [17], so it is categorized as a heavy metal that has the highest affinity [18]. Lead metal is more widespread than most other toxic metals [14], so it is very dangerous if it enters the human body.

Adsorbents. Adsorbents are solid substances that can absorb certain components of a fluid phase. Most adsorbents are in the form of highly porous materials so that adsorption takes place on the pore walls or at certain locations within the particle [19]. A good adsorbent has a variety of requirements as follows: has great dissolving power, has a low vapor pressure, as far as possible not corrosive, has a relatively low viscosity, thermally stable, affordable prices. From the requirements above, it can be concluded that the adsorbent is used as an absorbent for unwanted substances. Adsorbents used commercially can be grouped into two groups, namely polar and non-polar groups. Types of adsorbents belonging to the polar adsorbent group are silica gel, activated alumina, and zeolite. While the types of adsorbents included in the non-polar adsorbent group are adsorbent polymers and activated charcoal [19].

Activated charcoal. Activated charcoal is an amorphous compound produced from an adsorbent containing carbon or charcoal which is specially treated to obtain high adsorption power. Activated charcoal is one of the most frequently used adsorbents in the adsorption process because it has better adsorption power and surface area than other types of adsorbents, so it can adsorb certain gases and chemical compounds depending on the size of the pore volume and surface area. The absorption capacity of activated charcoal is very large, which is 25-100% by weight of activated charcoal [20]. The adsorption properties of activated charcoal are influenced by several factors including; size of adsorbate molecules, influence of temperature, characteristic of adsorbent, contact time and agitation speed. The types of activated charcoal are differentiated according to their functions, namely activated charcoal in gas and liquid phases. Gas phase activated charcoal is a carbon adsorbent gas absorbent, this type of charcoal is used to adsorb materials in the form of vapor or gas. This type of carbon can be found in coconut shell carbon. Meanwhile, liquid phase activated charcoal is used to absorb unwanted impurities from liquids or solutions. This type of charcoal usually comes from coal and cellulose.

Research Methodology

Methods this research was carried out experimentally in the laboratory. The experimental design method means a set of experiments designed to obtain real data to prove a hypothesis. In the experimental design, each variable is tested by being determined at several prices. Usually two values are used for the independent variables, then the independent variables are combined for several possibilities. From the combination obtained, it is used for drawing conclusions. So that the experimental design method is very possible to obtain more accurate results and conclusions.

Experimental design is a method of calculation that is often used compared to other methods that are considered conventional, because experimental design only requires fewer runs to find out the side effects on all variables. Optimum conditions obtained can be more precise because it includes interactions. Decision making can be more certain because it is supported by easy and simple statistical calculations. Experimental design consists of several ways, one of which is the factorial design method with two levels, namely low level and high level. This factorial design method is used to prove or strengthen the conclusions from conventional methods.

Experiments carried out on experimental designs with n variables use the 2^n equation formula, for 3 variables a total of 2^3 or 8 experiments are needed. In this study the variables used were absorption temperature (A), absorption time (B) and adsorbent size (C). Next, a two-level experimental design (low level and high level) was created, with 3 variables. Variance analysis was carried out by calculating the main effect, interaction effect and calculating the yield equation. Determining influential variables using normal probability charts. The interaction effect point furthest from the approach line is the most influential variable. An approach line is a line created by passing the line over many interaction effect points.

Determination of variables in the experimental design method includes fixed and changing variables. The fixed variable used was heavy metal adsorbed $Pb(NO_3)_2$, with weight was 2 grams, the concentration of $Pb(NO_3)_2$ was 5 ppm. While the changing variables used are adsorption temperature of 30°C and 60°C, adsorption time of 60 and 90 minutes, size of adsorbent 100-120 mesh and 60-80 mesh.

The research materials used were: ketapang seed shell (ketapang seed shell) obtained in the area around Semarang. $Pb(NO_3)_2$, 2.5 N HCl, 1 N NaOH, HNO_3, methylene blue, and aquadest purchased from The Indrasari chemical shop, Semarang Indonesia. Meanwhile, the research tools used were: beaker glass, erlenmeyer, measuring flask, furnace, filter paper, oven, blender, pH indicator, statives, clamps, desiccator, analytical balance, magnetic stirrer, Sieve 60, 80, 100 and 120 mesh, hot plate stirrer, thermometer , Spectrophotometry Atomic Absorption (SSA), shakers.

Research procedure

Adsorbent preparation. The outer skin of the ketapang seeds or ketapang shells is cleaned and cut ± 3 cm then dried in the sun for 2 days until the raw material is dry or loses its water content. After the dehydration process, the raw material is carbonized into the furnace for 15 minutes at a temperature of 400°C. Then blended until smooth and sieved using 60-80 mesh and 100-120 mesh size sieve.

Activation of the adsorbent with HCl. As much as 25 grams of ketapang seed outer shell powder measuring 60-80 mesh and 100-120 mesh were put into 250 ml of 2.5 N HCl and stirred for 1 hour at room temperature. Then allowed to stand for 24 hours and filtered after that neutralized with 1 N NaOH to pH 7 and dried in an oven at 110ºC for 4 hours.

Adsorption Process. 25 ml of 5 ppm $Pb(NO_3)_2$ solution was added to 2 grams of adsorbent size 60-80 mesh 100-120 mesh then stirred at 30ºC and 60ºC for 60 and 90 minutes using a magnetic stirrer. Then filtered to separate the adsorbed $Pb(NO_3)_2$ solution from the adsorbent.

Analysis of $Pb(NO_3)_2$ levels reduction using Atomic Absorption Spectrophotometry. The adsorbed $Pb(NO_3)_2$ solution on the filtering results was measured for its absorption at a wavelength of 283 nm to determine the decrease in its levels.

Analysis of the reduction of metal levels of $Pb(NO_3)_2$ using Scanning Electrone Magnetic. $Pb(NO_3)_2$ solution before and after adsorption with the highest decrease in metal content, was analyzed using Scanning Electrone Magnetic to determine its morphological picture.

Result and Discussion

Determination of the Most Influential Variable. Determination of the most influential variable, using the normal probability chart as shown in Fig. 2. The symbols in the figure, IA are temperature interactions, IB are time interactions, IC are adsorbent size interactions. Meanwhile, IAB states the interaction between temperature and time, IAC is the interaction of temperature with the size of the adsorbent, IBC is the interaction of time with the size of the adsorbent and IABC is the interaction of temperature-time-size of the adsorbent.

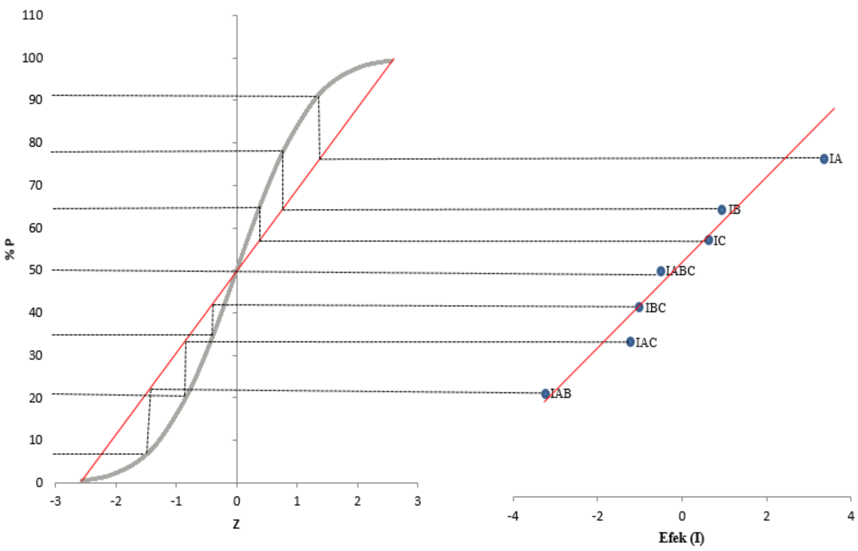


Fig. 2. Normal probability chart

Fig. 2 shows that the temperature variable (IA) is located farthest from the approach line, thus indicating that the temperature variable greatly affects the adsorption power of the active charcoal adsorbent on the outer shell of ketapang seeds on decreasing levels of metal $Pb(NO_3)_2$. This is because when the adsorbate molecules attach to the adsorbent, energy is released so that the adsorption rate will increase with increasing temperature [21].

Optimization of influential variables. Optimization was carried out at a temperature range of 30-60ºC with an adsorbent weight of 2 grams, a concentration of 5 ppm, an adsorption time of 60 minutes, and an adsorbent size of 60-80 mesh. The results of the optimization experiments show that the higher the adsorption temperature, the greater the adsorption power as shown in Fig. 3.

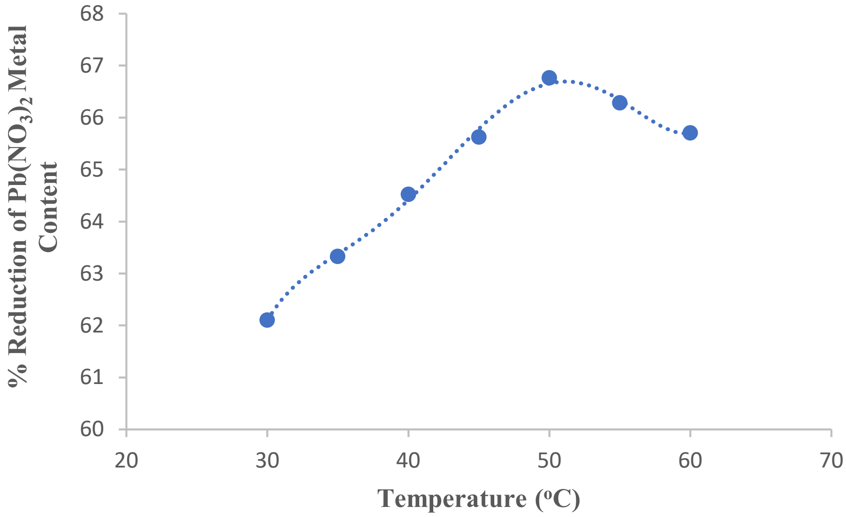


Fig. 3. Optimization of temperature

This happens because temperatures that are too high can cause desorption or re-release of ions or molecules that have bound to the active group on the adsorbent [15], so that it only reaches the optimum point, namely at 50°C and after reaching the optimum point, the % decrease in metal $Pb(NO_3)_2$ will decrease with increasing temperature. The optimum condition was reached at 50°C with a reduction level of $Pb(NO_3)_2$ metal by 66.77%.

Testing the water content, ash content and absorption capacity. Testing the water content aims to determine the hygroscopic nature of activated charcoal. The hygroscopic nature causes activated charcoal under certain conditions and humidity to reach an equilibrium moisture content which is a measure of hygroscopicity. The method used is oven drying. The sample was placed in an oven at 100°C for some time, until a constant weight was obtained. then the weight is measured before and after drying. The difference between the initial and final weights indicates the water content in the sample. The result of measuring the water content is 8%.

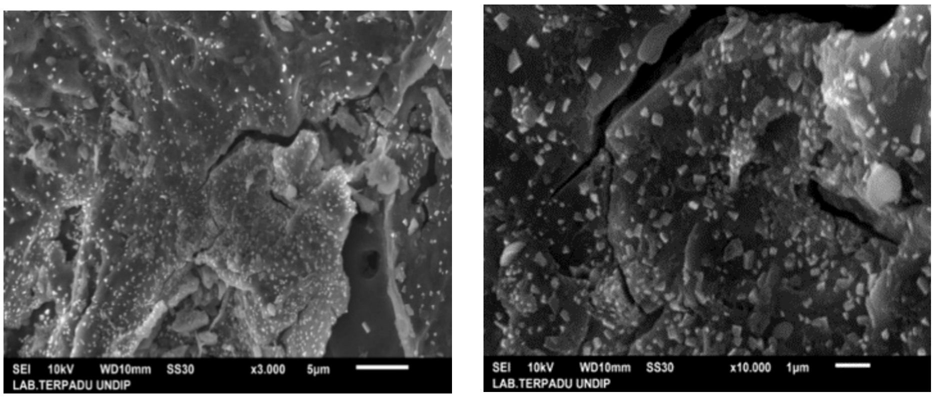
Testing the ash content aims to determine the mineral content of activated charcoal, because activated charcoal made from natural ingredients does not only contain carbon compounds. The lower the ash content in activated charcoal, the greater the ability of the adsorbent to absorb metal ions. This is because the ash content is an impurity that is not expected to exist in the adsorbent. The sample to be tested is cleaned of dirt and other foreign matter. Then, the sample is fired at high temperature, usually in a furnace at 550-600°C, for several hours until ash is formed. After the ash is formed, the ash is taken from the furnace and cooled to room temperature. Then, the ashes were weighed. After that, the ashes were burned again in the furnace at the same temperature as before for 1 hour, then cooled down to room temperature. Then, the ashes were weighed again. The ash content can be calculated by subtracting the weight of the ash after the first burning by the weight of the ash after the second burning, then dividing by the initial weight of the sample and multiplying by 100% to get the ash content in percent. The results of measurement of ash content obtained 5.2%.

Testing the absorption capacity of methylene blue aims to determine the surface area of activated charcoal and its ability to absorb colored solutions. This test was carried out by adsorption using a shaker with a speed of 100 rpm without immersion time and the absorbance was measured using atomic absorption spectrophotometry with a wavelength of 664 nm. The measurement results obtained adsorbent absorption of methylene blue 698 mg/g with a surface area of 2583654 m^2/g. Calculation of surface area using the following equation [22]:

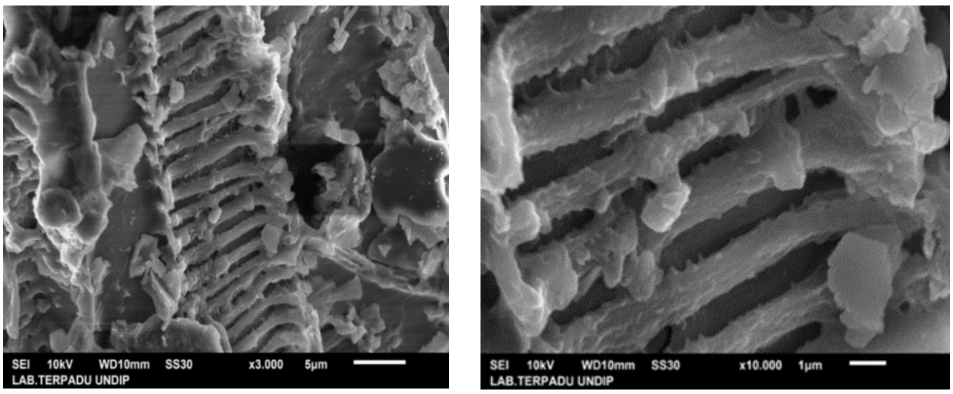
$$S = \frac{X_m . N . a}{Mr}$$

S = Surface area of activated charcoal
Xm = Adsorption power
N = Avogadro's number
a = Cross-sectional area of methylene blue
Mr = Formula Mass

Scanning Electrone Microscope (SEM) Analysis. SEM analysis aims to determine the microstructure (including porosity and crack shape) of solid objects. The results of the SEM analysis can be seen in Fig. 4.



(a) magnification 3,000 times (b) magnification 10,000 times



(c) magnification 3,000 times (d) magnification 10,000 times

Fig. 4. SEM Analysis Results: (a), (c) before adsorption; (b), (d) after adsorption

The results of SEM analysis before adsorption, Fig. 4(a) and 4(b), show pores after activation using HCl. The pores formed are mesoporous, the mesoporous material is activated carbon which consists of carbon skeletons with mesoporosity and microporosity due to a diameter of 2 to 50 nm. The small pores which are the structure of the cellulose of the ketapang seed coat indicate that there are bonds in the form of cellulose OH ions which can cause the adsorption process to occur. For the results of SEM analysis after adsorption, Fig. 4(c) and 4(d), it appears that there are deep holes which have absorbed the $Pb(NO_3)_2$ metal attached to the pore surface, this is because the adsorbent has reacted with the adsorbate through adsorption process.

Several factors that influence adsorption kinetics include: nature of the adsorbant and adsorbate, adsorbant concentration, temperature, surface area of the adsorbent, relative humidity, agitation or mixing, pH level. These factors can interact with each other and influence the overall adsorption kinetics.

Several factors that influence adsorption kinetics include: nature of the adsorbant, adsorbant concentration, temperature, surface area of the adsorbent, relative humidity, agitation or mixing, pH level. These factors can interact with each other and influence the overall adsorption kinetics. Several factors that influence adsorption thermodynamics include: adsorption energy, adsorption enthalpy, adsorption entropy, temperature, pressure, adsorbant concentration, adsorbent surface properties. These factors together influence the thermodynamics of adsorption and determine the thermal and energetic properties of the process

Conclusion

Adsorption temperature is the most influential variable in the implementation of natural adsorbents from activated charcoal outer shell of ketapang seeds on decreasing levels of metal $Pb(NO_3)_2$. Optimum conditions achieved at adsorption time of 60 minutes, adsorbent size of 60-80 mesh is at 50ºC with a maximum yield of 66.77%.

Activated charcoal from the outer shell of ketapang seeds can be used to reduce $Pb(NO_3)_2$ levels in the solution. The adsorption process on activated charcoal will capture Pb^{2+} ions in solution, reducing their concentration and thereby reducing $Pb(NO_3)_2$ levels. Activated charcoal has a large pore structure and surface area which allows it to absorb and bind heavy metal ions such as Pb^{2+} effectively. Therefore, the use of activated charcoal from the outer shell of ketapang seeds can be an effective option in reducing $Pb(NO_3)_2$ levels in the solution.

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