



**ADDIS ABABA UNIVERSITY**

**ADDIS ABABA INSTITUTE OF TECHNOLOGY  
SCHOOL OF CHEMICAL AND BIO- ENGINEERING**

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**Activated Carbon Preparation and Characterization from Flax Straw for  
Removal of Pb (II) Ions from Aqueous Solution and Industrial Wastewater**

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A Thesis Submitted to the School of Chemical and Bio Engineering, Addis Ababa Institute of  
Technology in Partial Fulfillment of the Requirements for the Degree of Master of Science in  
Chemical Engineering (Process Engineering Stream)

**Addis Ababa University**

**Addis Ababa, Ethiopia**

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ADDIS ABABA UNIVERSITY  
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This is to certify that the thesis prepared by Tayto Mindahun, entitled: ‘**Activated Carbon Preparation and Characterization from Flax Straw for Removal of Pb (II) Ions from Aqueous Solution and Industrial Wastewater**’, submitted in partial fulfillment of the requirement for the Degree of Master of Science in Chemical Engineering (Process Engineering Stream), complies with the regulation of the University and meets the accepted standards with respect to originality and quality.

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## Declaration

I, the undersigned, declare that this thesis entitled '**Activated Carbon Preparation and Characterization from Flax Straw for Removal of Pb (II) Ions from Aqueous Solution and Industrial Wastewater**' is my original work and has not been presented in any form for another degree, diploma or an award at any university or other institution. I confirm that appropriate credit has been given within this thesis where reference has been made to the work of others and has been duly acknowledged.

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## Abstract

*There are different modern technologies to remove heavy metals from industrial effluent. However, applying these modern technologies is impossible in all situations. Hence, investigating alternative technologies like adsorption by AC for such kinds of problem is very important. Considering this, the aim of this study was to prepare AC from flax straw and investigate its potential for the removal of Pb (II) ions from aqueous solution and paint industry wastewater. AC was prepared by chemical activation method using  $H_3PO_4$  as activating agent. The effects of activation time, activation temperature and activating agent concentration on yield and iodine number were studied by using RSM. The effects of initial Pb (II) ion concentration, adsorbent dose, contact time and pH on removal efficiency were studied by using synthetic wastewater prepared from lead nitrate on a batch mode. The collected wastewater sample was characterized before and after treatment according to APHA methods. AC was characterized and results showed that the flax straw AC had 8.04% of moisture, 6.04% of ash, 18.615% of volatile matter, 79.421% of fixed carbon, 459.807 mg/g of iodine number and surface area of 489.455  $m^2/g$ . Physico-chemical characteristics revealed that raw wastewater had a concentration of 3.95 mg/L Pb (II), 158.52 mg/L BOD<sub>5</sub>, 2482 mg/L COD, and 652.667 mg/L TSS. The highest removal efficiencies of Pb (II) metal ion which was achieved from aqueous solution and paint wastewater were found to be 95.16% and 78.73%, respectively. The experimental data are fitted with pseudo-second order model and adsorption of Pb (II) on flax straw AC fits the model of Langmuir very well. The results suggested that flax straw AC can be used as adsorbent to remove Pb (II) ion from paint industry wastewater.*

**Keywords:** activated carbon; flax straw; optimum conditions; removal of Pb (II) ions

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## List of Abbreviations

AC	Activated Carbon
ANOVA	Analysis of Variance
APHA	American Public Health Association
ASTM	American Society for Testing Materials
BET	Brunauer-Emmet-Teller
DOE	Design of Experiment
FTIR	Fourier Transform Infrared
IR	Impregnation Ratio
pH	Power of Hydrogen
PZC	Point of Zero Charge
RSM	Response Surface Methodology
SEM	Scanning Electron Microscope
UV/vis	Ultraviolet Visible
XRD	X-Ray powder diffraction

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## List of Symbols

$q_e$	Amount of dye adsorbed per gram of the adsorbent at equilibrium (mg/g)
$q_m$	Maximum monolayer coverage capacity (mg/g)
$R_L$	Langmuir equilibrium parameter/ separation factor
$k_L$	Langmuir isotherm constant (L/mg)
$C_o$	Initial adsorbate concentration (mg/l)
$C_e$	Equilibrium concentration of adsorbate (mg/l)
$k_F$	Freundlich isotherm constant
$n$	Constant of freundlich isotherm
$k_1$	Rate constant of pseudo-first-order sorption Rate
$k_2$	Rate constant of pseudo-second-order sorption
$R^2$	Correlation coefficient

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# 1. Introduction

## 1.1. Background

Activated carbon is an adsorbent obtained from carbon containing materials or carbonaceous materials with large specific surface area and well developed pore structure, which is physically and chemically stable [1]. It is also widely known that activated carbon is one of the most important types of adsorbents utilized in a variety of industrial and residential applications to safeguard the environment [2].

The history of activated carbon (AC) can be traced back to ancient Egypt (1500 B.C). Egyptians used its adsorbent properties for water purification and medicinal adsorbent during this time. Activated carbon was first manufactured industrially in the first half of the twentieth century. Activated carbon was made from vegetable matter at the time and utilized in the sugar refining process. In the US, the first production of activated carbon used black ash as the source, after it was unintentionally discovered that the ash was very effective in decolorizing liquids. Starting from this time of production, activated carbon has been used extensively for this purpose in many industries [3].

Activated carbon is widely used in purification and separation in many industrial processes, including medicinal use, pollutant and odor removal, gas separation, and catalysis. Furthermore, activated carbon is a common adsorbent for removing pollutants from wastewater, particularly heavy metals and dyes discharged from different industrial wastes [1].

Heavy metals like arsenic, chromium, copper, mercury, nickel, and silver are among the most widely known pollutants found in modern effluents. However, lead is a substantial heavy metal found in wastewater from the paint industry which is toxic to life, even at low concentrations, and can affect the nervous and reproductive system [4–7]. To remove heavy metals from industrial effluent, precipitation, ion exchange, coagulation and electro dialysis, are the most commonly used technologies [7]. These technologies have numerous drawbacks like incomplete metal ion removal, high energy and reagent costs, and toxic sludge. But, adsorption technique using activated carbon look to be more attractive due to its simplicity, ease of use, high efficiency, and being economical in the removal of heavy metals from wastewater [4, 6, 7]. AC can be regenerated and reused at the end of the process as compared to other existing methods

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due to this they are economical. Despite the fact that activated carbon has numerous applications in various industries, the main challenge is obtaining the best and excess raw materials with the potential to produce effective activated carbon since commercial activated carbon is primarily coal-based activated carbon, which is more expensive. As a result, it is critical to look for alternative adsorbents made from lignocellulose agricultural waste. Conversion of lignocellulose content to activated carbon is a possible and feasible approach. Conversion of lignocellulosic raw materials in to activated carbon can be done via physical activation or chemical activation method [7].

Chemical activation methods are often carried out at lower activation temperatures and shorter activation time than physical activation. Chemical activation produces higher yields of activated carbons than physical activation because the chemical agents utilized have dehydrogenation capabilities, which prevent the creation of tar and limit the development of other volatile compounds. Now a day, several researchers have adopted various low-cost adsorbents, but there is still a need to develop activated carbon from cheaper and readily available materials, which can be effective and economical for the removal of heavy metals from wastewater [8].

It is known that flax straw is one of the low cost agricultural waste by-products which is left in the fields after harvest, on the other hand, have found a very limited application. In most Ethiopian region, flax straw is cultivated, this indicated that there is enough resource of flax straw from agricultural waste for the preparation of activated carbon. Therefore, utilizing this material as raw material for AC preparation is very important. and also, in Ethiopia, flax straw is highly cultivated. Flax straw is widely cultivated in the high elevations area of Arsi, Bale, Shewa, Gojam, Gonder, Wollo, and Wellega [9]. Therefore, the availability of these large quantities of fibrous residues has triggered the interest used as input for production of AC. Flax straw contains high carbon and volatile matter content and low ash percentage [10]. So, it is possible to produce activated carbon from flax straw. Thus, this research explores the potential of this raw material to prepare AC and its performance for the removal of lead metals ions from synthetic solution and paint industry wastewater.

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## 1.2. Statement of the Problem

Currently there is a rapid population growth and there is also a growing trend of industrial sector development. Continued population growth and rapid industrialization are found to be the cause of wastewater discharge into the environment, affecting the environment, human health and the life of future generations. The liquid waste discharged from industries contains heavy metals like Pb, Cd, Cr, Cu, Ni and Zn toxic to living organisms [11]. Among these toxic metals; Lead is a potent poison and is harmful in even very small amount to humans and other living organisms because of its known toxicity [12]. Therefore, industries have to use treatment technologies in order to remove this toxic metal from wastewater. A number of advanced technologies have been developed over the years to remove this toxic heavy metal from waste water. But; it is difficult to use some of these advanced technologies in all level of developing countries for industries to deal with heavy toxic industrial wastewater.

Thus; it is important to investigate alternative technologies for this bottleneck problem for industries. Since lead is the most easily adsorbed cation by activated carbon [10], we can use adsorption technology by activated carbon prepared from agricultural wastes. In the preparation of activated carbon from agricultural wastes such as straw biomass, we have to identify the varieties from which the straw biomass is sourced. However, most of the research findings did not give information on varieties of the biomass. This may pose a difficulty on the reproducibility of data and may hinder future research that may want to compare the effect of varieties on the biomass properties. Hence, it is crucial task to properly describe the varieties of biomass. In addition to this, even if activated carbon preparations from different straw biomass have been studied so far, but little attention has been paid to flax straw for preparation of activated carbon.

And also, flax straw is availability most regions of Ethiopia since it is preferable at low soil fertility. However, flax straw is not used for animal feed rather it is directly discharge to the environment and pollutes the environment. Thus, using this biomass for production AC is very important. Since phosphoric acid activation causes less environmental and toxicological contamination, this study was used  $H_3PO_4$  as activating agent for the preparation of AC from flax straw of known varieties for lead metal ions removal from aqueous solution and paint industry wastewater.

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## **1.3. Objectives**

### **1.3.1. General Objective**

The general objective of this research was to prepare and characterize activated carbon from flax straw as an adsorbent for the removal of lead (II) ions from aqueous solutions and real wastewater.

### **1.3.2. Specific Objectives**

- To investigate the effects of activation temperature, concentration of  $\text{H}_3\text{PO}_4$  and activation time on yield and iodine number of AC and to find the optimum conditions.
- To characterize the physico-chemical properties of flax straw activated carbon.
- To determine the effect of pH, initial concentration of lead (II) ions, contact time and adsorbent dose on removal efficiency by using batch adsorption method.
- To determine the kinetic and isotherm models for lead (II) ions adsorption process.

## **1.4. Research Questions**

In this study the following research questions were designed to enable the objectives of the study to be met. These include;

1. What is the effect of activation temperature, concentration of  $\text{H}_3\text{PO}_4$  and activation time on yield and iodine number of AC?
2. What is the effect of pH, initial concentration of lead (II) ions, contact time and adsorbent dose on removal efficiency of lead (II) ions removal?
3. Which isotherm model best describes the adsorption of lead (II) ions from wastewater on to flax straw activated carbon?
4. Which kinetic model best describes the dynamic mechanism of lead (II) ions adsorption process?

## **1.5. Significance of the Study**

This research was carried out in order to produce a low-cost adsorbent from agricultural wastes or byproducts for the adsorption of lead metal ions from industrial wastewater in order to reduce pollution loads of lead metal ions on the environment.

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This research will help to increase environmental awareness at all levels of government, reduces the cost of industrial wastewater treatment, and decrease locally available agricultural wastes or byproducts. It also provides a mechanism for exploitation and management of local resources and fundamental information for policy makers and activated carbon manufacturers on the potential of flax straw as a raw material. In addition to this, the findings of this research will help both agricultural waste management and wastewater purification process.

### **1.6. Scope of the Study**

This study was focuses on the preparation of activated carbon from flax straw at different experimental conditions (activation time, activation temperature and concentration of  $H_3PO_4$ ) and characterization of the prepared activated carbon (proximate, ultimate and surface properties). Finally, the study was completed by performing adsorption experiment for lead metal ions removal from aqueous solution in order to determine the optimum adsorption parameters (initial Pb (II) ion concentration, adsorbent dosage, contact time and pH).

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## **2. Literature Review**

### **2.1. Overview of Activated Carbon**

Activated carbon can be viewed as a porous carbonaceous material with continually expanding applications in water treatment and desalination, wastewater treatment and air purification due to its unique characteristics [14]. The elimination of all non-carbon impurities and the oxidation of the carbon surface distinguish activated carbon from elemental carbon [15]. All activated carbon has similar structural features, such as graphite-like planes with varied degrees of disorientation and the spaces between these planes that constitute porosity [15, 16].

In general, activated carbon is a black, amorphous solid that contains a large percentage of fixed carbon and a lower fraction of other components such as ash, water vapor, and volatile substances. Aside from that, activated carbon has physical properties like internal surface area and pore volume [17, 18]. Because of its high surface area, it has a high capacity for absorbing heavy metals and other contaminants from wastewater. The adsorptive ability of activated carbon is due to the vast internal pore structure that develops during the activation process as it was studied by [19].

To summarize, activated Carbon is proven to be the most essential material for eliminating numerous contaminants from both potable water and wastewater in both industry and households [3, 20, 21]. Thus, the adsorption features of activated carbon inspire most researchers to utilize it in practically every field of study, owing to its ease of construction and operation, selectivity for specific chemicals, and entire elimination of contaminants even from dilute solutions. As a result, there is a growing interest in developing long-lasting, dependable, and selective alternative raw materials for the production of activated carbon in order to save and protect the environment by treating and disposing of clean wastes [22].

### **2.2. Raw Materials for Activated Carbon**

Practically any carbonaceous material can be used to produce activated carbons if correctly managed during the production processes. Coal, lignite, peat, petroleum residue, and wood were the most common sources of activated carbons on the market [23, 24].

However, for the production of activated carbon, these basic ingredients are very expensive and limited. As a result of these situations, researchers are looking for alternative raw materials for

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production activated carbon which use agricultural bio-waste (lignocelluloses materials) [25]. Agricultural wastes have been exploited as precursors for the manufacture of activated carbon by numerous researchers in recent years due to four key reasons: renewable supply, low cost, easy availability, and environmental friendliness [23]. In general, when selecting raw materials for the production of activated carbon, we must keep the following characteristics in mind [24]:

- High carbon content
- Low inorganic matter content for low ash result
- High density and volatile matter content.
- Abundant; so that the raw material is always in very low cost
- Potential extent of activation
- Low degradation rate upon storage
- Possibility of producing an activated carbon with high percent yield.

However, because biomass wastes have lower carbon content than fossil fuel sources, meeting the last condition for raw material selection from biomass wastes is extremely challenging. As a result, the percent yield of activated carbon made from biomass waste could be reduced [23].

Different agricultural by-products, such as straw biomass or other lignocellulosic by-products, can potentially replace conventional and expensive sorbents for the manufacture of activated carbon. Among the different straw biomasses, flax straw is one of the lignocellulosic agricultural by-products which can be activated to produce activated carbon since it is carbonaceous straw agricultural waste biomass [26, 27].

Due to this reasons flax straw could be an excellent precursor for the commercial production of activated carbon. There are different varieties of flax (linseed) released in different years. Table 2.1 show different varieties of flax (linseed) found in Ethiopia.

Table 2.1: Different varieties of flax (linseed) found in Ethiopia

Variety	Year of release	Days to maturity	Seed yield (qt/ha)		Oil content (%)
			Research field	Farmers' field	
Kulumsa-1	2006	131	11	17.85	36.1
Tolle	2004	143	16.9	10	36.0
Berene	2001	140	16.17	9.10	37.0
Belay-96	1996	141	16.8	9	36.3
Chilalo	1992	140	16.70	9.0	35.2
CI-1525	1984	146	14.3	8.10	38.5
CI - 1652	1984	146	13.6	8.80	38.6
Kassa-2	2012	143	12.59	---	37.45
Jiituu	2012	152	19-20	16-18	37.1
Jeldu	2010	180	15.14	11.23	37.0

Source: (Ministry of Agriculture and Natural Resources, 2016)

Table 2.2 below shows the carbon content of the flax straw investigated by different researchers. As stated in table 2.2, flax straw has high carbon content like other crop straws. As a result of this, flax straw biomass can be used as raw material for the production of activated carbon. To create high-quality AC, we have to choose straw bio mass with a high carbon content, high volatile matter and low ash content [28].

Table 2.2: Elemental analysis of flax straw

Straw biomass	C (%)	H (%)	N (%)	S (%)	Ash (%)	Moisture content (%)	Volatile matter (%)	Fixed carbon (%)	References
Flax straw	49.1	6.1	1.3	0.12	2.9	8.4	81.2	18.8	[27]
	46.76	6.34	1.1	1.14	1.12	4.75	74.80	19.32	[29]
	43.1	6.2	0.68	0.09	3.0	7.9	80.3	8.8	[30]

### 2.3. Classification of Activated Carbon

Because activated carbons are such complicated compounds, categorizing them based on their production methods, physical qualities, and surface features is difficult. However, AC can be

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classified based on particle size as powdered activated carbon (PAC), granular activated carbon (GAC), and activated carbon fibers [31]. Nonetheless, the most frequently used are granular and powdered AC [22].

### **2.3.1. Powdered Activated Carbon**

Powdered activated carbon (PAC) has a typical particle size of less than 0.1 mm and the common size of the particle ranges from 0.015 to 0.025 mm. PAC is used in the treatment of industrial and municipal waste water, sugar decolorization in the food industry, pharmaceuticals, and the removal of mercury and dioxin from flue gas streams. Because of its low initial cost and short contact time, PAC is also employed for wastewater treatment [22].

### **2.3.2. Granular Activated Carbon**

When we look at granular activated carbon (GAC), we can see that the average particle size ranges from 0.6 to 4 mm. It is applicable in continuous liquid and gas phase operations. It also has an advantage over PAC in that it has a lower pressure drop and can be regenerated, allowing it to be reused multiple times [22].

### **2.3.3. Activated Carbon Fibers**

Carbonized carbon fibers that have been heat treated in an oxidizing atmosphere are known as activated carbon fibers (ACFs). Despite the fact that a good ACF precursor must be non-graphitic and non-graphitizable carbon fiber that is isotropic in nature, research continues to focus on the creation of ACFs from a variety of low-cost precursors.

## **2.4. Preparation of Activated Carbon**

Generally, there are two methods for preparation of activated carbon. These includes physical activation and chemical activation [33–37].

### **2.4.1. Physical Activation**

Physical activation is a two-step process that begins with the carbonization of dried samples at (400-700)<sup>o</sup>C to produce biochar, followed by activation with oxidizing gases such as steam, air, CO<sub>2</sub>, or their mixtures at a high temperature of about (800 -1100) <sup>o</sup>C for some burn off [38, 39].

The main aim of the carbonization process is to reduce volatile content of the raw material in order to convert it to a suitable form for activation. Apart from widening the small pores created on the surface of biochar during physical activation, additional pores are produced in the

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meantime, increasing the porosity and surface area of the carbonaceous porous structure. Physical activation, on the other hand, tends to produce a lower carbon yield and quality of AC than chemical activation due to the greater activation temperature and longer activation time [38].

#### **2.4.2. Chemical Activation**

In this process of producing activated carbon, the raw material will be impregnated with the activating agent before being introduced to the carbonization. Finally, the activated carbon is washed to eliminate any remaining activator residues in the material and to recover the chemical agent for subsequent activated carbon synthesis cycles [8].

Common chemical activating agent includes: potassium hydroxide (KOH), sodium hydroxide (NaOH), calcium chloride (CaCl<sub>2</sub>), potassium carbonate (K<sub>2</sub>CO<sub>3</sub>), phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) and sulfuric acid [38]. Chemical activation uses phosphoric acid and zinc chloride to activate lignocellulosic materials that haven't been carbonized yet, and metal compounds like potassium hydroxide to activate charcoal and char precursors[39].

When phosphoric acid is compared with zinc chloride as chemical activation agent, phosphoric acid has less environmental and toxicological limits and requires a lower activation temperature [39]. Furthermore, chemical activation is almost primarily employed for carbons made from recently formed materials, such as lignocellulosic materials [32–34], [41]. The pyrolytic process is influenced by the activation agent. The following are some of the advantages of chemical activation over physical activation for the production of activated carbons [32, 36], [41–44]:

- Lower activation temperature (< 800<sup>o</sup>C) compared to the physical activation temperature (800 – 1100)<sup>o</sup>C
- Single activation step
- Higher yields
- Better porous characteristics and
- Shorter activation times.

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## **2.5. Factors Affecting AC Preparation via Chemical activation**

### **2.5.1. Activation Temperature**

The temperature at which the activated carbon is prepared has a significant impact on the characteristics of the activated carbon that is produced. Carbon will be lost if the temperature is too high during the production process. Temperature influences not just straw activation, but also the yield and surface area of activated carbon produced from straw biomass [1]. Temperature like (450–600)<sup>o</sup>C, (400–900)<sup>o</sup>C, (700–1000)<sup>o</sup>C, (550–850)<sup>o</sup>C, and (450–850)<sup>o</sup>C were the most common activation temperatures in the process of producing activated carbon with phosphoric acid, zinc chloride, potassium carbonate, sodium hydroxide, and potassium hydroxide, respectively [22].

### **2.5.2. Activation Time**

If we apply excessive activation time during preparation of activated carbon; it causes the reduction in product yield. Increasing activation time gradually, will cause the volatilization of organic substances from raw agricultural waste residues [22]. The pore structure of activated carbon is gradually formed as the activation time increases, but due to the loss of carbon for an extended period of time, the micropore of activated carbon will tend to become mesoporous and macropore, and the specific surface area of activated carbon will be reduced [1]. For activated carbon production utilizing phosphoric acid as an activating agent, activation times ranging from (30 – 210) minutes are acceptable [23, 45, 46].

### **2.5.3. Activating Agent Types**

The most often used activating chemicals in chemical activation processes are ZnCl<sub>2</sub>, H<sub>3</sub>PO<sub>4</sub>, NaOH, and KOH. Each of these agents has a unique effect on the raw materials [24]. Phosphoric acid activation causes less environmental and toxicological contamination than zinc chloride activation, and it also requires a lower activation temperature than potassium hydroxide activation [39]. Activators have a significant influence on the chemical activation process. Furthermore, H<sub>3</sub>PO<sub>4</sub> is commonly used in chemical activation to enhance bond cleavage processes while also maintaining the interior pore structure [35].

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#### **2.5.4. Impregnation Ratio**

Impregnation ratio is the weight of the activating agent to the impregnated raw material. The study found that a larger impregnation ratio leads to the production of mesopores, while a lower impregnation ratio leads to the formation of micropores. This is because a larger impregnation ratio causes more swelling and a stronger release of volatile matter contents, which causes pores to enlarge. The most typical impregnation ratios discovered in most of the literature are between 0.5 and 5 [47, 48].

### **2.6. Characterization of Activated Carbon**

The primary goal of characterization for activated carbon (AC) is to classify activated carbon for specific applications and provide qualitative and quantitative data. Modeling the behavior and performance of activated carbons can also benefit from such data and information. Characterization also gives information that can be used in the design and preparation of activated carbons [48].

#### **2.6.1. Moisture Content**

The total quantity of moisture in activated carbon in all forms except water of crystallization of the mineral particles residing within the activated carbon is known as moisture content. For activated carbon, a moisture content of up to 10% is required. This moisture content has no effect on the adsorptive activity for many uses, but it does dilute the carbon. As a result, an additional weight of moist carbon is required to meet the dry weight requirement [49].

#### **2.6.2. Ash Content**

Ash content is the residue that remains when the carbonaceous fraction of an activated carbon is burned off. Excessive ash concentration in activated carbon is undesirable since it diminishes the carbon's mechanical strength and affects its adsorptive activity. In the end, a high ash concentration lowers the fixed carbon proportion [50].

#### **2.6.3. Volatile Matter**

Volatile matter is the thermal decomposition products like methane, hydrocarbons, hydrogen, carbon monoxide and incombustible gases like carbon dioxide and nitrogen which are liberated or given off when coal is heated at high temperature in the absence of air. Naturally occurring

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plants have more volatile organic compounds and high percentage of volatile matter. High value of volatile matter reduces the quantity of fixed carbon [50].

#### **2.6.4. Fixed Carbon**

Fixed carbon is the carbon that remains after the volatile matter have been determined, which does not occur naturally in coal but occurs as a result of thermal decomposition [49]. According to the study performed by [51], fixed carbon content is also affected by cellulose and lignin content that can be converted to carbon atoms.

#### **2.6.5. pH of Activated Carbon**

The pH of activated carbon is the pH of a suspension of carbon in distilled water. The presence of acid functional groups on the surface of carbon, such as carboxyl, phenolic, and others, may cause activated carbon to have an acidic characteristic [50]. Activated carbon carrying inorganics and chemically active groups on its surface may alter the pH of its liquids to which it is added[51] .

#### **2.6.6. Bulk Density**

Bulk density is the weight of material that can be contained in a given volume under specific conditions. More density suggests higher volume activity and, in most cases, higher quality activated carbon. The shape, size, and density of individual particles determine the bulk density of activated carbon. Data on bulk density can be used to estimate tank or packing volume [52].

#### **2.6.7. Water Solubility of Activated Carbon**

The main goal of performing activated carbon solubility test in water is to determine the quantity of contaminants contained in activated carbon prepared using various carbonization processes. This is because the presence of contaminants in activated carbon might have an impact on the quality of the treated water [50]. Since all activated carbon exhibits moderate level of impurities, this test method covers the determination of these impurities and are expressed as a percentage of dry carbon [53].

#### **2.6.8. Acid Solubility of Activated Carbon**

This test method is performed in order to determine the acid-extractable content of a sample of activated carbon. It is also vital to be aware of the presence of substances other than carbon in activated carbon, but it makes no sense to address or identify such substances. Acid-extractable

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content can be measured quantitatively to evaluate activated carbon samples that contain acid-soluble contaminants. These acid-soluble contaminants can affect how activated carbon is used [53].

### **2.6.9. Surface Area Analysis**

Activated carbons are widely known for the high specific surface area, which provides a well-developed porosity. The typical values for agricultural waste biomass activated carbon ranged from  $200\text{m}^2/\text{g}$  to  $2000\text{m}^2/\text{g}$  and can be explained by the micropore structure [54]. One could believe that a larger surface area will improve adsorption properties as an adsorbent because there will be more sites where adsorbates can be held. However, not all surfaces will be available for those molecules if the adsorbate molecules are larger than some of the micropore sizes. At the same time, the adsorbent's geometry and the pore's geometry must be taken into account.

### **2.6.10. Iodine Value of the Activated Carbon**

Iodine number is a measure of the activated carbon's adsorptive ability. The iodine adsorption capacity of activated carbon indicates its ability to adsorb tiny molecular compounds. Iodine adsorption from the liquid phase was used to understand more about the porous structure of activated carbon. The iodine value is calculated using the American Society for Testing and Materials' technique, which is defined as the quantity of iodine adsorbed per gram of activated carbon at an equilibrium concentration of 0.02 N [50].

### **2.6.11. Scanning Electron Microscope (SEM) Analysis**

The primary goal of SEM analysis is to visualize the surface texture and porosity of activated carbon products [51]. Evaporation and breakdown of non-carbon chemicals in the feedstock can also affect the shape of the pores that will develop. Activated carbon also has a rougher surface and more irregularity than ordinary carbon. The activator can cause the pores of activated carbon to grow and the surface to expand.

### **2.6.12. Determination of Point of Zero Charge (PZC)**

The ease with which activated carbon adsorbs potentially harmful ions such as heavy metals, pesticides, dyes, and other pollutants is predicted by its point of zero charge (PZC) values [55]. The pH at pzc ( $\text{pH}_{\text{pzc}}$ ) is the value of pH at which the total external and internal surface of the ion is fixed at zero [56].

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If the surface is positively charged and the pH is raised, anions will be less adsorbed as the hydroxide ion concentration rises. If the pH of the adsorbent is less than the pzc value, the surface charge of the adsorbent will be positive, allowing the anions to be adsorbed. Conversely, if the pH is above the pzc value, the surface charge would be negative so that the cations can be adsorbed [57]. Therefore, determination of the PZC is an important element of the characterization of commercial adsorbents [58].

#### **2.6.13. Fourier Transform Infra-Red (FTIR) Analysis**

The main aim of doing FTIR analysis is used to characterize the functional group presented on the surface of activated carbon [51]. FTIR is most useful for identifying chemicals that are either organic or inorganic. The term Fourier Transform Infrared Spectroscopy (FTIR) refers to a fairly recent development in the manner in which the data is collected and converted from an interference pattern to a spectrum [59].

#### **2.6.14. X-ray Diffraction (XRD)**

The primary goal of X-ray diffraction is to gather structural information on activated carbon crystalline solids. According to XRD studies, activated carbons are categorized into two categories depending on their graphitizing capacity [31]. Due to the production of strong cross-linking between adjacent randomly oriented elementary crystallites, non-graphitizing carbons are hard and have a well-developed micro porosity structure. Graphitizing carbons, on the other hand, showed poor cross-linking and a less developed porous structure. The XRD pattern of amorphous materials does not contain unambiguous peaks; instead, it has noise signals, smeared peaks, or short order bumps [60].

### **2.7. Application of Activated Carbon**

Activated Carbon is used in a variety of industries, including food and beverage, sugar, petroleum, fertilizer plants, nuclear, paints, medicines, cosmetics, textiles, automobiles, and vacuum manufacturing [22]. Because activated carbon has been discovered to have good porous materials in its structure, it is particularly effective in adsorbing solutes from aqueous solutions or wastewater.

Activated carbon is also utilized as a filter medium for removing hazardous particles from exhaust gases, water purification, and waste water treatment. This concept is used to meet the

ever-increasing demand for activated carbon at a low cost [23]. One of the most applications of activated carbon is the removal of lead metal ions from the industrial wastewater.

### 2.7.1. Lead Removal from Wastewater Using Activated Carbon

High amounts of lead in the environment may pose a long-term health concern to individuals and ecosystems alike. Processing industries, such as paint, acid battery production, metal plating and finishing, tannery, fertilizer, mining, petrochemical, ceramic, and glass sectors, are important contributors of lead contamination [67–69].

Table 2.3 show that the permissible limit of some selected heavy metals in water. Compared to the other heavy metals, the allowable limit for lead metal is extremely low. As a result, removing lead metals to the acceptable amount is required. The most critical issue is its removal from wastewater before to discharge into the environment.

Table 2.3: Concentration and specific properties of some heavy metals in waters [64]

Heavy metals	Permissible limits (mg/L)			
	WHO	US EPA	EU	EEPA (2003)
Ni <sup>2+</sup>	0.07	–	0.02	0.1
Zn <sup>2+</sup>	0.005	0.005	–	5
Pb <sup>2+</sup>	0.001	0.0015	0.001	0.05
Cd <sup>2+</sup>	0.003	0.005	0.005	1

Where:

- ◆ WHO is World Health Organization.
- ◆ US EPA United State Environmental Protection Authority.
- ◆ EU is European Union.
- ◆ EEPA is Ethiopian Environmental Protection agency.

The most common methods for removal of pollutants or heavy metals like lead from industrial effluents including: ion exchange, chemical precipitation, electrochemical, membrane filtration process, flocculation and flocculation and adsorption etc [69]. Adsorption is the most widely used method for the treatment of industrial wastewater having color, heavy metals and other

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inorganic and organic impurities due to its simplicity in operation, inexpensive (compared to other separation processes and without sludge formation [8]).

### **2.7.2. Batch Adsorption Experiment for Lead Removal from Wastewater**

Batch adsorption experiments are crucial for determining its effectiveness for removing metal ions. The batch adsorption experiment can be used to explore the parametric effect of pH, adsorbent dose, initial concentration, and contact time on the removal efficiency and adsorption capacity of activated carbon for lead metal from wastewater [19, 71–74]. Furthermore, the majority of the studies on metal adsorption using waste materials utilized batch experiments to see how the adsorbate and adsorbent performed under different settings. Temperature and agitation speed, as well as other parameters, are included in these conditions [71,72].

### **2.7.3. Factors Affecting Heavy Metal Removal by Adsorption Experiment**

#### **2.7.3.1.The Effect of pH**

The pH of a solution has an enormous influence on heavy metal adsorption since it influences adsorbent properties including surface charge, as well as adsorbate speciation and degree of ionization in aqueous solutions. Adsorbents' adsorption capability is typically poor under acidic environments, but increases as pH rises [73, 74]. The decrease in competition between metal cations and hydrogen ions for the same sorption sites can be attributable for this situation. It could also be explained by a decrease in electrostatic repulsion due to a decrease in positive charge on the adsorbent's surface [73].

#### **2.7.3.2.Effect of Contact Time**

When the adsorption process begins, adsorption is usually higher at lesser contact time since the adsorbent has a lot of active sites. The active sites of the adsorbent become exhausted as time passes, and the rate at which the adsorbate is transferred from the external to the internal sites of the adsorbent regulates the sorption process [74].

#### **2.7.3.3.Effect of Initial Metal ion Concentration**

The initial metal ion concentration rises, the amount of metal ions adsorbed decreases [72]. The fraction of ions adsorbed, on the other hand, falls. This increase is due to the many active binding sites on the adsorbent that are initially vacant. As the metal ion uptake proceeds, adsorbates

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saturate active sites, resulting in reduced percentage removal. As a result, the binding sites immediately absorbed the available ions at low concentrations, but at greater concentrations, the adsorption species had to migrate to the interior sites via intra-particle diffusion, resulting in a slower adsorption rate [71].

#### **2.7.3.4. Effect of Adsorbent Dosage**

The adsorption capacity ( $q_e$ , mg/g) decreased as the adsorbent dosage was raised, whereas the adsorption percentage increased. This is because all active sites were exposed at low levels, while only a small percentage of active sites were exposed at higher doses. As a result, a larger adsorbent dosage might lead to aggregation, which reduces the overall surface area of the adsorbent and reduces adsorption. Furthermore, as adsorbent dosages grow, adsorption sites may overlap due to overcrowding of adsorbent particles, which explains why an increase in adsorbent dosage may not result in an increase in adsorption [71].

### **2.8. Adsorption Isotherm Model**

An adsorption isotherm is a graphical representation of the amount of adsorbate on the adsorbent as a function of its concentration at constant temperature. The isotherms show the relationship between the amount of adsorbate adsorbed on a specific adsorbent and the concentration of adsorbate in solution [7, 77]. The capacity, surface characteristics, and affinity of an adsorbent are commonly determined using equilibrium adsorption isotherms [76].

The performance of activated carbon is evaluated using adsorption isotherms. The most common isotherms models for adsorption system includes Langmuir and Freundlich isotherm models [7, 62, 77].

#### **2.8.1. Langmuir Model**

This model assumes that all the available adsorption active sites are similar, the adsorbed species does not interact, and a monolayer is formed during adsorption [73, 74]. The Langmuir isotherm can be expressed by [71]:

$$\frac{C_e}{q_e} = \frac{C_e}{q_m} + \frac{1}{K_L q_m} \quad 2.1$$

Where:

- $q_e$  (mg/g) is the equilibrium amount of solute adsorbed in mg per gram of solid
- $C_e$ (mg/L) is the equilibrium concentration of solute in solution
- $q_m$  (mg/g) is the maximum monolayer adsorption capacity of the adsorbent.
- $K_L$  (L/mg) is the Langmuir constant describing adsorption affinity for the adsorbent.

### 2.8.2. Freundlich Isotherm

According to [76], the Freundlich isotherm is the earliest known relationship describing non-ideal and reversible adsorption that is not limited to monolayer formation. This empirical model can be applied to multilayer adsorption, with non-uniform distribution of adsorption heat and affinities over the heterogeneous surface. The linearized form of the Freundlich isotherm is given by the following equation [72]:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad 2.2$$

Where:

- $K_F$  and  $1/n$  are the Freundlich constants related to sorption capacity and sorption intensity, respectively.

The Freundlich isotherm, unlike the Langmuir isotherm, has no thermodynamic basis and does not provide much physical interpretation of adsorption data. One limitation of the Freundlich model is that the amount of adsorbed solute increases indefinitely with the concentration of solute in the solution [66].

## 2.9. Adsorption Kinetic Model

In a batch adsorption process, kinetic studies are utilized to understand about the optimum conditions, the adsorption mechanism, and possible rate control steps. These details are mostly employed in the process modeling and design. Adsorption kinetics also describes reaction pathways as well as the time it takes to reach equilibrium [77]. According to the study that was performed by [5, 68, 69], the adsorption of the lead (II) from wastewater can be tested by using pseudo first-order and pseudo second-order kinetic models.

### 2.9.1. Pseudo-First-Order Model

According to the pseudo-first order model, the rate of adsorption site occupation is proportional to the number of vacant sites [76]. The linear equation for this model is:

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$$\log(q_e - q_t) = \log q_e - \frac{K_1}{2.303} t \quad 2.3$$

Where:

- $q_e$  and  $q_t$  are the amounts of metal adsorbed ( $\text{mg g}^{-1}$ ) at equilibrium and at time  $t$  (min),  
 $k_1$  ( $\text{min}^{-1}$ ) is the rate constant of pseudo-first-order adsorption rate.

### 2.9.2. Pseudo-Second Order Model

According to the study that was previously done by [76], the rate of adsorption site occupation is supposed to be proportional to the square of the number of vacant sites in the pseudo-second order model. The differential equation for this model is:

$$\frac{dq_t}{dt} = K_2(q_e - q_t)^2 \quad 2.4$$

Where:

- $K_2(\text{g mg}^{-1} \text{min}^{-1})$  is the equilibrium rate constant of the pseudo second-order sorption. Integrating the above equation for the boundary conditions  $t = 0$  to  $t$  and  $q_t = 0$  to  $q_t$  gives:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{1}{q_e} t \quad 2.5$$

The rate-limiting stage in the pseudo-Second Order Model is surface adsorption, which involves chemisorption, in which the adsorbate is removed from a solution due to physicochemical interactions between the two phases [77].

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### 3. Materials and Methods

#### 3.1. Raw Materials

The raw material used for this study was flax straw (linseed straw). Two flax straw varieties (Bekoji-14 and CI-1525) were collected from Holeta Agricultural Research Center (HARC), which is located in Holeta, a town in the Oromia region's special zone with a latitude and longitude of 9°3'N 38°30'E / 9.050°N 38.500°E and an elevation of 2391 meters above sea level.

#### 3.2. Chemicals and Reagents

The chemicals and reagents used for this study include: phosphoric acid ( $H_3PO_4$ ) as activating agent, Analytical grade Pb (II)  $NO_3$  for standard metal ion sample preparation, distilled water for solution preparation and washing purpose, Sodium hydroxide and Hydrochloric Acid for pH adjustment, Sodium Thiosulfate, ( $Na_2S_2O_3 \cdot 5H_2O$ ), Iodine (I), Potassium Iodide (KI), Potassium iodate, primary standard ( $KIO_3$ ), potato starch and Sodium Carbonate for iodine number determination.

#### 3.3. Equipment and Instruments

The table below outlines the various equipments and instruments that were employed to conduct this study's experiment.

Table 3.1: Equipment and instruments used for the study

Equipment's Name	Model	Available area
CHNS/O elemental analyzer	EA 1112 Flash CHNS/O- analyzer	AAU, Chemistry department
UV-Vis Spectrophotometer	UVD-3200, LABOMED, INC.	AAiT, Analytical laboratory
Scanning Electron Microscopy	FEI-INSPECT-F50	Addis Ababa science and Technology university (AASTU)
▪ Thermo Scientific FT-IR	SMART iTX, Thermo scientific	
▪ BET Surface area analyzer	Horiba SA-9600 Series, California	
▪ DO meter	HI 9829, HANA instruments	
▪ Vacuum filtration Pump	MZ-2CNT, Germany	
▪ Muffle furnaces	MV 106, Germany	

▪ Hot air oven	PRI/1501A, India	Both AASTU & AAiT laboratories
Electronic Analytical balance	AD300-3, Germany	
pH meter	3505 pH meter, JENWAY	Addis Ababa science and Technology university (AASTU)
Silica crucibles with lid, Desiccator, Spatula, Heat resistance gloves, mortar and Pestle, Buret, 50-mL capacity, Erlenmeyer Flasks, Beakers, Bottles, Funnels, Qualitative Filter Paper, Micro Pipette, Volumetric Flasks, Graduated Cylinders, Flasks, Erlenmeyer 250-ml with ground glass stoppers, Pycnomotor		Addis Ababa science and Technology university (AASTU) And AAU, School of Chemical and Bioengineering research laboratories

### 3.4. Methods

#### 3.4.1. Collecting, Pretreatment and Characterization of Flax Straw

The collected flax straws were first chopped by using knife into small pieces and washed with tap water for the removal of dirt and dust particles that may have been presented on the surface of the straw. Then it was sun dried for the purpose of removing the moisture from the samples. The sun dried samples of the two varieties were milled to 0.5 mm size using cutting mill (FRITSCH Cutting Mills Pulverisette, Germany). The milled samples were then oven dried for 24 h at 105°C for further moisture removal from the samples. Finally, the samples were stored in airtight plastic bags for further experimental work.

Proximate analysis (ash, moisture content, volatile matters and fixed carbon) were analyzed according to the methods described by ASTM (2010). And also, elemental analyses (Carbon, Hydrogen, Nitrogen, Sulfur content) of the two varieties were analyzed by using CHNS/O elemental analyzer (Model- EA 1112 Flash CHNS/O- analyzer) at Addis Ababa University chemistry department under carrier gas flow rate of 120 ml/min, reference flow rate 100 ml/min, oxygen flow rate 250 ml/min, furnace temperature of 900°C and oven temperature of 75°C.

### 3.4.2. Activated Carbon Preparation

In this study, 11g of the pretreated flax straw was first impregnated with 30.3 ml of solution (30, 50 and 70) % phosphoric acid at fixed impregnation ratio of 2.75. The mixture was then left for 24 hours in order to facilitate impregnation process. Then the impregnated sample was placed in oven at 105°C for 24hrs. After drying is completed, the dried samples were transferred into a muffle furnace (MV 106, Germany) and carbonized at different carbonization temperatures (400, 500 and 600) °C at different carbonization times (30, 60 and 90) minutes [23].

After completion of carbonization process, carbonized samples were immediately taken into a desiccator to prevent contact with oxygen (moisture absorption) and allowed to cool to room temperature. Then activated carbons obtained were washed with distilled water, 0.5 M HCl (37%) or 0.5 M NaOH until the effluent water shows the neutral pH to remove the free phosphoric acid. Lastly, activated carbons were dried in an oven at 105°C for 24hrs, grinded with mortar and pestle, sieved using 125 µm sieve and stored in plastic bottles for further analysis or characterization.

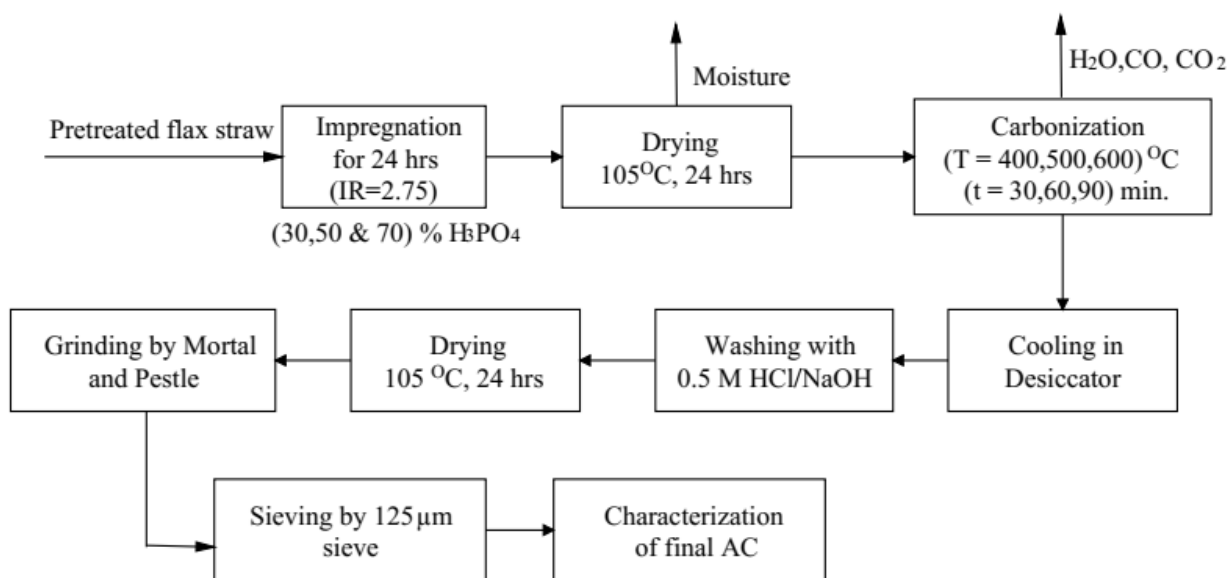


Figure 3.1: Block diagram for preparation of activated carbon

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### 3.5. Characterization of Final Activated Carbon

#### 3.5.1. Ash Content

The ash content of activated carbon was determined by the method developed by ASTM D2866-94(2004). The ash content of the final AC is determined in % according to equation 3.1 given below:

$$\text{Ash content}(\%) = \frac{(M_c + M_a) - M_c}{(M_c + M_s) - M_c} \times 100 \quad 3.1$$

Where:

- $(M_c + M_a)$  is the weight of crucible and ash after complete ashing.
- $(M_c + M_s)$  is the weight of crucible and sample before ashing.
- $(M_c)$  is the weight of empty crucible

#### 3.5.2. Moisture Content

The moisture content of flax straw activated carbon was determined by the method and procedures developed by ASTM D2867-94(2004). Finally, the moisture content was determined in weight % according to equation 3.2 given below:

$$\text{Moisture content}(\%) = \frac{(M_1 - M_2)}{M_1} \times 100 \quad 3.2$$

Where:

- $M_1$  is mass of empty dry crucible (g) and  $M_2$  is mass of crucible plus sample after drying (g)

#### 3.5.3. Volatile Matter

The percentage of volatile matter of the activated carbon was determined by the procedures set by ASTM D5832-98(2014). At the end, the volatile matter content ( $V_m$ ) was determined in % according to the equation 3.3 given herein.

$$V_m(\%) = \frac{100(B - C) - M_c(B - A)}{(B - A)(100 - M_c)} \times 100 \quad 3.3$$

Where:

- A, B & C= weight of crucible, weight of crucible plus sample & weight of crucible plus sample after heating respectively.

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### 3.5.4. Fixed Carbon Content

Fixed carbon content of the prepared activated carbon was determined according to the method of [78] which states that fixed carbon is a calculated value and it is the resultant of summation of percentage moisture, ash, and volatile matter subtracted from 100.

$$\text{Fixed carbon (\%)} = 100 - (\text{moisture, \%} + \text{ash, \%} + \text{volatile mater}) \quad 3.4$$

### 3.5.5. Carbon Yield

The yield of flax straw activated carbon samples were calculated by the method developed by [79] on a chemical-free basis. The yield of AC is calculated as the percentage weight of the resultant activated carbon divided by weight of dried flax straw.

$$\text{Yield(\%)} = \frac{\text{Weight of activated carbon}}{\text{Weight of dried flax straw}} \times 100 \quad 3.5$$

$$\text{Or, } Y(\%) = \frac{m}{m_o} \times 100 \quad 3.6$$

Where:

- ◆  $m$  and  $m_o$  are the dry weight of final activated carbon (g) and dry weight of precursor (g), respectively.

### 3.5.6. Bulk Density Determination

The bulk density of flax straw activated carbon was measured in accordance with the method developed by [80] with little modifications. A pycnometer with a capacity of 50 cm<sup>3</sup> was used and it was calculated by equation 3.7.

$$\rho = \frac{M_3 - M_1}{V_1} \quad 3.7$$

Where:

- $\rho$  is bulk density
- $M_3 - M_1$  is the mass of the adsorbent in the pycnometer.
- $V_1$  is the volume of the weighed quantity of activated carbon which is equal to the volume of the displaced water and is given by equation 3.8.

$$V_1 = \frac{[(M_2 - M_1) - (M_4 - M_3)]}{\rho_{\text{water}}} \quad 3.8$$

---

### 3.5.7. Determination of Iodine Number

The iodine number was measured according to the procedures established by the American Society for Testing Materials, ASTM D4607-94(2006). The iodine number was obtained from equation 3.9 as described by [81]:

$$\text{Iodine number} = \frac{[25.4x(30 - V_n)]}{M_{AC}} \quad 3.9$$

Where:

- $M_{AC}$  is the mass of activated carbon (g),  $V_n$  is the Volume of thiosulfate solution at equivalence point (ml).

### 3.5.8. Specific Surface Area

The surface area of flax straw activated carbon was determined by BET surface area analyzer (SA-9600 Series Surface Area Analyzer, Horiba instruments, Inc.) at Addis Ababa Science and Technology University (AASTU) chemical engineering research laboratory. Two samples of activated carbons were degassed at 100<sup>0</sup>C for 45 minutes prior to analysis in order to remove any adsorbed moisture or other impurities bounded to the surface of the activated carbon samples. Surface area (m<sup>2</sup>/g) was obtained from the experimental adsorption isotherm over a relative pressure range of BET surface area measurement experiment under the stated conditions of the experiment.

### 3.5.9. pH Determination

The pH of activated carbon was determined by the procedures established by the American Society for Testing Materials, ASTM D3838-98(2004).

### 3.5.10. Water Solubility

The water solubility (S), of the activated carbon was determined by the methods developed by ASTM D5029-98(2004). The percentage solubility in water was calculated as the weight ratio of unrecovered carbon to the original sample.

$$S(\%) = \frac{\text{Loss in weight on dissolution}}{\text{Weight of orginal carbon}} \times 100 \quad 3.10$$

---

### 3.5.11. Acid solubility

For solubility in HCl, the same method was follow as water solubility but 0.2 M HCl is used instead of distilled water [82].

$$S(\%) = \frac{\text{Loss in weight on dissolution}}{\text{Weight of orginal carbon}} \times 100 \quad 3.11$$

### 3.5.12. Determination of Point of Zero Charge((pHpzc)

Point of zero charge (pHpzc) of an activated carbon samples was investigated by the solid addition method [83] with some modification. A mixture of 0.1 g flax straw activated carbon in 45 ml of 0.1 M NaCl was equilibrated at 25<sup>o</sup>C on an incubator shaker (Model: Excella E24R) for 24 h. The initial pH of the suspension was adjusted in the range 2–10 with 0.1 M NaOH or 0.1 HCl using pH meter (Model: 3505 pH meter, JENWAY). The final pH of the suspension was measured after the 24 hour equilibrium time. The PZC of the samples was determined from the plot of  $\Delta\text{pH}(\text{pH}_{\text{final}}-\text{pH}_{\text{initial}})$  versus  $\text{pH}_{\text{initial}}$  of the sample mixtures.

### 3.5.13. Scanning Electron Microscope (SEM)

The surface morphology of the flax straw activated carbon sample was analyzed by the Scanning Electron Microscope (Model: FEI-INSPECT-F50) at Addis Ababa Science and Technology University (AASTU), central laboratory.

### 3.5.14. FTIR (Fourier Transform Infrared spectrometry)

The analyses of the activated carbon samples were done in Addis Ababa Science and Technology University (AASTU), Food Engineering Department using Thermo Scientific FT-IR instrument (Model: SMART iTX, Thermo scientific). FTIR spectroscopy analysis of the flax straw activated carbon treated with phosphoric acid before and after adsorption experiment were carried out to identify the functional groups that might be involved or responsible for in the binding of heavy metal ions on its surface. The analysis was done in such a way that first; the dried activated sample was mixed with KBr particles to make it suitable to infrared analysis. The mixture was then pressed to a small thickness, slightly below 1 mm, required for FTIR spectroscopy analysis. FTIR spectra of samples were recorded in the wave length of range of (400 – 4000)  $\text{cm}^{-1}$ .

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### 3.5.15. XRD (X-ray diffraction)

X-ray diffraction was performed in order to evaluate the existence of amorphous and crystalline between carbon matrixes. The XRD (X-ray diffraction) analysis was carried out by using X-Ray diffractometer (model: XRD-7000 X-RAY Diffractometre, MAXIma) at Adama Science and Technology University (ASTU) under the condition of voltage 40.0 KV and current 30.0 mA. The scan was obtained from a range of 5.000 to 85.000 (Bragg angle  $2\theta$ ) of sampling pitch  $0.02^\circ$  every 0.40 (sec).

## 3.6. Batch Adsorption Experiments

### 3.6.1. Preparation of Synthetic Wastewater

Synthetic wastewater samples were prepared by using analytical grade lead nitrate (99 % New Delhi, India) by using distilled water. The stock solution of 500 mg/l was obtained by dissolving 0.8 g of lead nitrate in 500 ml distilled water. The lead standard working solutions of (40, 50, 60, 70, 80, 90 and 100) mg/l was prepared by serial dilution using equation 3.12 given below[66].

$$C_1V_1 = C_2V_2 \quad 3.12$$

Where:

- $C_1$  is the initial concentration(mg/l),
- $C_2$  is the final concentration(mg/l),
- $V_1$  is the initial volume(ml), and
- $V_2$  is the final volume(ml)

### 3.6.2. Calibration Curve Plot for Lead Metal Solution

Working solutions were prepared by diluting the stock lead metal solution. The concentrations of lead metal ions in solutions were estimated by measuring absorbance at wavelength of 300 nm by UV-Vis Spectrophotometer (Model-UVD-3200, LABOMED, INC) at Addis Ababa Institute of Technology (AAiT), biochemical engineering laboratory and examined the calibration curve of absorbance versus lead metal concentration. Finally, linear relationship of absorbance versus concentration of lead was plotted and final concentration of lead was found from the equation 3.13 given below.

$$\text{Absorbance} = \text{slope} * C_e + Y - \text{intercept} \quad 3.13$$

---

### 3.6.3. Batch experiment procedures

In this study, for each experimental run, working solutions (40-80)mg/l were obtained from the stock solution by serial dilution and the pH was adjusted from (2-10) by adding 0.1M HCl or 0.1M NaOH. Then, adsorbent dose (1- 4) g was added in each 250ml Erlenmeyer flask containing the solution. The samples were shaken on incubator shake (Model: Excella E24R) at room temperature, at a constant rate (250 rpm) [84].

The contact time for experiment was in the range 10 minutes to 120 minutes. The samples were collected at the end of time required for adsorption. The supernatant liquid was filtered by qualitative filter paper of size 15 cm and collected 100 ml volumetric flask. Finally, the collected samples were analyzed for lead metal ions removal percentage using UV-Vis Spectrophotometer (UVD-3200, LABOMED, INC.) at 300nm. The final concentration of lead metal ions solution at the equilibrium was measured from the calibration curve. The percent removal of metal ions and adsorption capacity were calculated by using equation 3.14 and 3.15 respectively [84]:

$$\text{Percentge removal} = \left( \frac{C_o - C_e}{C_o} \right) \times 100 \quad 3.14$$

$$\text{Adsorption capacity} = \left( \frac{C_o - C_e}{W} \right) \times V \quad 3.15$$

Where:

- $C_o$  (mg/l) is the initial concentration of lead metal ions and,  $C_e$  is the concentration of lead metal ions at equilibrium,  $V$  (L) is the volume of lead metal ions solution in the flask and
- $W$  (g) is the mass of activated carbon used in the experiment.

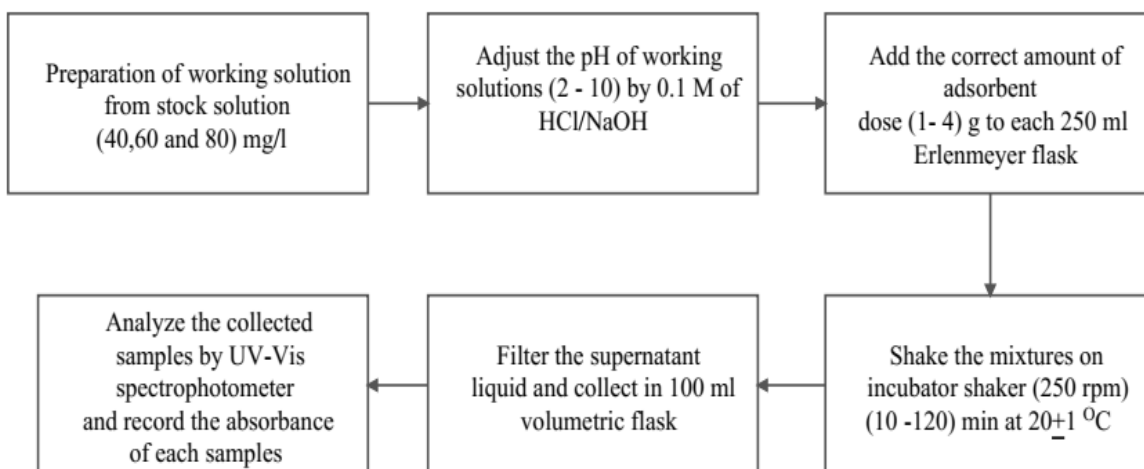


Figure 3.2: Set up for batch adsorption experiment

### 3.6.4. Isotherm Study

Adsorption isotherms were investigated for (20–90) mg/l initial lead metal ion concentrations using 4 g of flax straw activated carbon sample added to 100 ml of the metal ion concentrations and shaken for equilibrium time of 100.6 minutes at 250 rpm. Two isotherm models, namely; Freundlich and Langmuir, were employed [85].

### 3.6.5. Kinetic Study

Kinetics study was conducted by taking 100ml of lead metal ions solution with initial concentration of 46.7 mg/l in 250ml Erlenmeyer flasks and adjusting the pH to 4.9. Then 4 g of flax straw activated carbon was added to each samples and the solution will be agitated at 250 rpm in incubator shaker at different contact time(10-80) minutes at room temperature. The kinetics of lead metal ions adsorption on flax straw activated carbon was analyzed using pseudo first-order and pseudo second-order kinetic models [85].

## 3.7. Experimental Design

Response Surface Methodology, Box-Behnken experimental Design (BBD) with three factors was used for experimental data analysis of activated carbon preparation process using Design-expert -12 software.

Yield and Iodine number were the response variables. The three variables studied for the preparation of activated carbon were activation time, activation temperature and activating agent concentration.

Table 3.2: Independent variables and levels for BBD for AC preparation

Independent variables	Unit	Levels	
		Minimum	Maximum
Activation time	minutes	30	90
Activation temperature	°C	400	600
Activating agent concentration	%	30	70

And also, initial Pb (II) ion concentration, adsorbent dosage, contact time and pH on the removal efficiency of Pb (II) ion using flax straw activated carbon over three levels were analyzed by Box–Behnken method in order to understand their effect on adsorption of lead metal on AC. Removal efficiency was considered as response variable during the experiment.

Table 3.3: Independent variables and their levels for adsorption experiment

Independent variables	Unit	Levels	
		Minimum	Maximum
Adsorbent dose	g	1	4
pH of the solution	---	2	10
Initial lead metal concentration	Mg/l	40	100
Contact time	minutes	10	120

### 3.8. Collection and Characterization of Paint Industry Wastewater

In order to collect wastewater samples from paint factory, plastic bottles of 2000 ml was used. The wastewater sample was collected from KADISCO paint factory located in Addis Ababa, the sub city of Akaki Kality. The sample was then transported to Addis Ababa Sciences and Technology University, department of environmental engineering laboratory.

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Since it is difficult to analyze of (BOD<sub>5</sub>), COD, TSS and TDS of the waste water sample immediately, sample was preserved by adding 1.5 ml of concentrated nitric acid per liter of sample as stated by [86].

### **3.8.1. Temperature, pH and Dissolved Oxygen**

The temperature of wastewater was measured using thermometer. pH and DO were directly measured with DO meter (Model: HI 9829, HANA instruments).

### **3.8.2. Turbidity**

Turbidity of the water samples was conducted according to the methods of APHA (2017) section 2130B using turbidimeter (Model: 2100N).

### **3.8.3. Biochemical Oxygen Demand (BOD<sub>5</sub>)**

Biochemical Oxygen Demand (BOD<sub>5</sub>) was determined by the procedure described by APHA (2017) section 5210 B using BOD incubator (model: KBOD.10.5).

### **3.8.4. Chemical Oxygen Demand (COD)**

The Chemical oxygen demand (COD) is also determined by the method of APHA (2017) section 5220 B.

### **3.8.5. Lead metal Concentration**

The level of lead metal ions in the wastewater sample was determined by Microwave Plasma Atomic Emission spectrometry (Model: MP-AES Agilent Technologies, Melbourne, Australia) at Addis Ababa University in departments of chemistry laboratory.

### **3.8.6. Total Dissolved Solids and Total Suspended Solids**

Total dissolved solids and total suspended solids of the waste water sample were conducted according to the methods of APHA (2017) section 2540 C and 2540 D respectively.

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## 4. Results and Discussion

### 4.1. Characterizations of Flax Straw

The result of ultimate analysis of the flax straw of the two varieties (Bekoji-14 and CI-1525) is indicated in the table 4.1 given below.

Table 4.1: Ultimate analysis of flax straw varieties (Bekoji-14 and CI-1525)

Sample Code	Nitrogen (%)	Carbon (%)	Hydrogen (%)	Sulfur (%)
CI-1525	(n.m)*	54.9625	7.5465	(n.m)*
CI-1525a	(n.m)*	52.7348	7.2406	(n.m)*
Bekoji14	(n.m)*	50.8819	6.9862	(n.m)*
Bekoji14a	(n.m)*	47.3501	6.5013	(n.m)*

*Note:(n.m)\* - not measured.*

From the table 4.1 given above, it can be seen that CI-1525 is composed of average carbon content of 53.8487%, and Hydrogen 7.3936% whereas, Bekoju-14 had 49.1159% of Carbon and 6.7438% of Hydrogen which is expected in most cellulosic materials. Any materials with high carbon content can be employed as AC raw materials [14]. As a result, due to its high carbon content, as shown in table 4.1, flax straw (CI-1525) was utilized as a raw material for the preparation of activated carbon. Table 4.2 given below indicates the carbon content of different straw biomasses for comparison with flax straw.

Table 4.2: Ultimate analysis of straw biomasses

Straw biomass	Carbon (%)	Hydrogen (%)	Nitrogen (%)	Sulfur (%)	Reference
Wheat straw	43.64	5.82	0.68	1.08	[29]
Barley straw	44.83	6.25	0.93	1.18	[29]
Flax shive	50.3	6.1	0.6	0.07	[27]
Oat straw	43.26	6.12	0.88	1.14	[29]
Rice straw	40.67	5.73	0.80	0.01	[28]
Corn straw	41.67	5.91	0.58	0.15	[28]
Soybean straw	41.5	5.52	2.90	0.28	[28]

Compared with other straw biomasses, both flax straw varieties have high carbon content as it can be seen in the table 4.1 and 4.2. The proximate analysis for the selected flax straw variety (CI-1525) is indicated in table 4.3 given below.

Table 4.3: Proximate analysis of the flax straw (CI-1525)

Parameters	Result (%)
Ash content	5.62
Moisture content	7.18
Volatile matter	69.95
Fixed carbon	17.24

As shown in the table 4.3 given above, it can be seen that flax straw had lower ash content and higher volatile matter content. Biomass resources with a higher volatile matter content and lower ash content can be used to make activated carbon [14, 22]. Furthermore, the low ash content would mean that inorganic impurities would have a little impact on pore development during the activation process [100, 101].

#### 4.2. Characterization of Flax Straw Activated Carbon

The flax straw activated carbon should be characterized in order to create a consistent quality activated carbon that can be used in wastewater treatment or other industrial applications [82]. The proximate composition of the prepared activated carbon (using conditions listed in table 3.2) are listed in table 4.4 together with the quality standards for activated carbon [51].

Table 4.4: Proximate composition of flax straw activated carbon

Parameters	Result	Quality standards
Ash content (%)	6.04	Max.10
Moisture content (%)	8.004	Max.15
Volatile matter (%)	18.615	Max.25
Fixed carbon (%)	79.421	Min.60

The proximate composition values of this study were in reasonable agreement with the standard [51] as indicated in table 4.4 above.

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The amount of ash in the activated carbon has an influence on its quality. The presence of too much ash can cause the pores on activated carbon to get clogged, which reduce the surface area of the carbon. In addition to this, fungi and other microorganisms degrade activated carbon when the moisture content is high, and they use it in their metabolic processes. Microbes can also multiply within the AC macro and micropores, obstructing the pore structure and lowering the carbon's adsorptive ability [82]. The pH value for the prepared activated carbon was found to be 6.7, which is almost in neutral region. The pH Value of activated carbon is a measure of whether it is acidic or basic. A pH that is too high suggests that there is a lot of contamination, while a pH that is too low shows that the acid wash was not completed. The value obtained in this research falls approximately into the neutral region.

Water solubility, acid solubility (HCl solubility) and the density of the flax straw activated carbon were found to be 4.6%, 5.2% and 1.089g/cm<sup>3</sup> respectively. The result of water and HCl solubility of this study were smaller when we compare with the water and HCl solubility of phosphoric acid activated sawdust carbon which is reported as 8.40% and 9.3% respectively [82]. Due to the lack of electron donating or receiving species in the structure, such as lone pair electrons, pure carbon does not dissolve in either water or acid. In addition, the density was found to be lower than with the values of coconut shell activated carbon which is reported as between 1.91 and 2.04 g/cm<sup>3</sup> [89].

### **4.3. Point of Zero Charge**

In order to understand the adsorption mechanism, it is crucial to determine Pzc (pH pzc) of an activated carbon. Based on the value of pH<sub>pzc</sub>, adsorption of cations is favored at pH > pH<sub>pzc</sub>, while, the adsorption of anions is favored at pH < pH<sub>pzc</sub> [56]. As can be seen from the figure 4.1, pH<sub>pzc</sub> of flax straw AC is 4.5 and the surface was positively charged when the solution pH was below the pH of 4.5 (pH < pH<sub>pzc</sub>).

Table 4.5: Point zero charge determination result of flax straw activated carbon

P <sub>Hi</sub>	P <sub>Hf</sub>	Change (p <sub>Hf</sub> - p <sub>Hi</sub> )
2	3.13	1.13
3	4	1
4	4.33	0.33
5	4.69	-0.31
6	4.78	-1.22
7	5.01	-1.99
8	4.92	-3.08
9	4.77	-4.23
10	4.94	-5.06

At low pH value, positive charge densities on the sites of the AC surface is high which prevent the metal ions from approaching the binding sites on the surface of AC. On the contrary, when the pH value increased (pH > p<sub>Hpzc</sub>), the surface of activated carbon becomes negatively charged and the electrostatic repulsion between lead (II) ions was decreased. Due to this, the adsorption capacity of AC increased since lead is cationic metal.

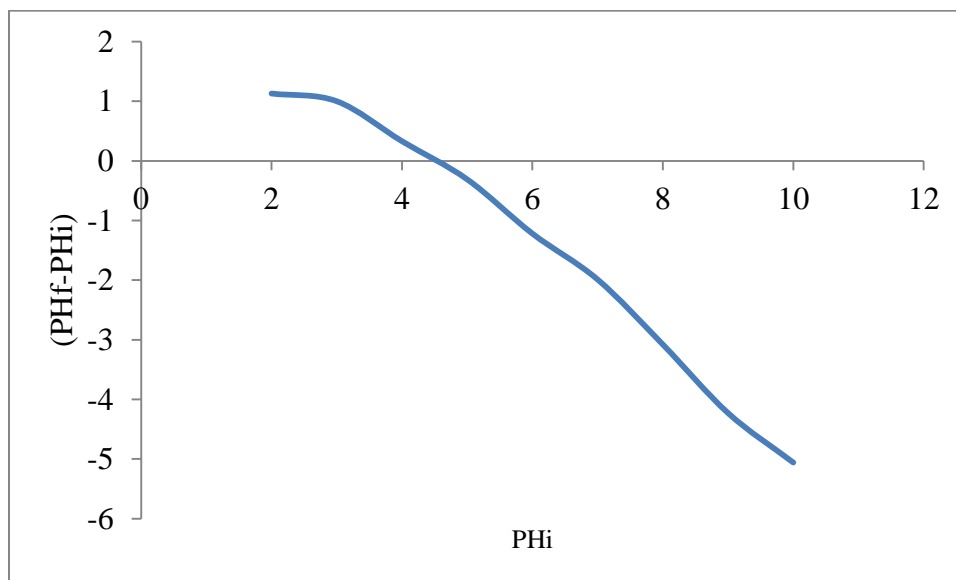


Figure 4.1: Determination of pH at zero point charge (p<sub>Hpzc</sub>) on surface of flax straw AC  
 And also, at this pH value, a strong electrostatic attraction phenomenon occurs between charged cationic metal and the negative charged surface of the activated carbon.

#### 4.4. Fourier Transformer Infrared (FTIR) Spectroscopy Analysis

In this study, FTIR spectroscopy was used to conduct a quantitative investigation of the key functional groups involved in the adsorption of lead metal ions onto activated flax straw. In the range of  $4000\text{cm}^{-1}$  to  $400\text{cm}^{-1}$ , the FTIR spectra of lead metal ions loaded and unloaded activated carbon treated with phosphoric acid were observed. The recorded adsorption spectra before and after adsorption are shown in figure 4.2.

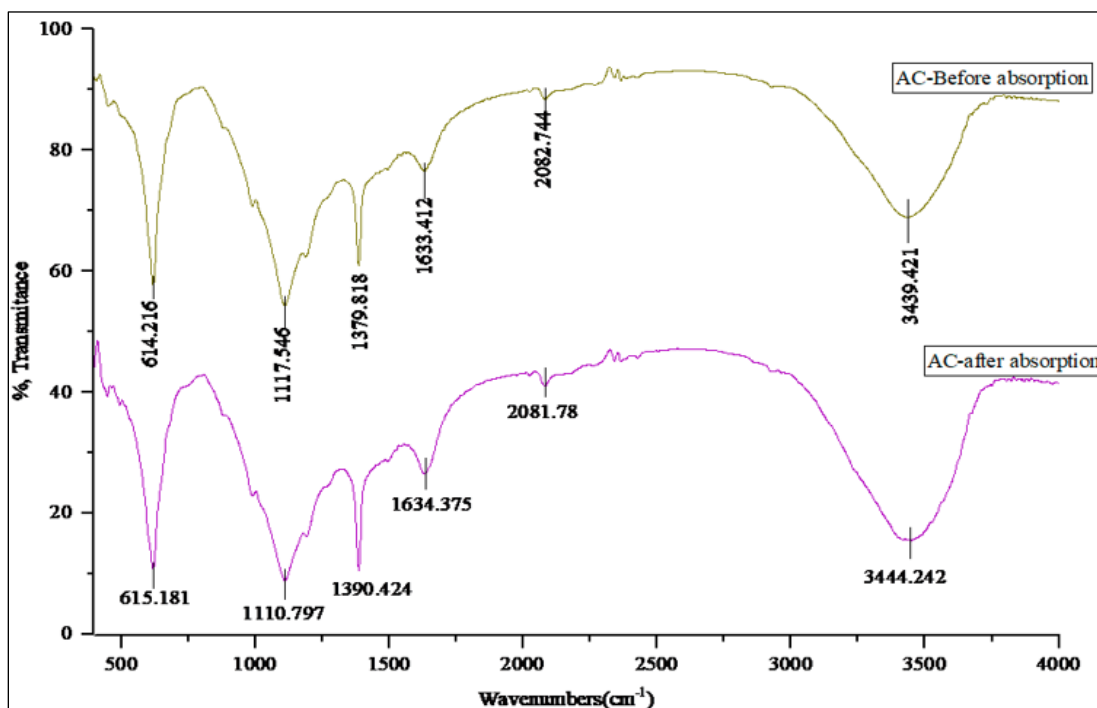


Figure 4.2: FTIR spectra of flax straw Activated carbon

The carbon matrix is known to contain additional heteroatoms such as hydrogen, oxygen, nitrogen, halogen, sulfur and others in addition to carbon atoms. These heteroatoms are attached to the margins of the carbon layers, governing the surface chemistry of the activated carbon and potentially influencing adsorption chemistry [36].

Because the Pb (II) ion binds to the activated carbon's active sites, the FTIR spectrum of the activated carbon exhibits variances in peak frequencies, showing the presence of ionizable functional groups on the activated carbon that might interact with additional cations [78].

Table 4.6: FTIR spectral characteristics of flax straw AC before and after Pb (II) ion adsorption

FTIR peaks	Band wave number (cm <sup>-1</sup> )			Assigned functional groups	Compound Class
	Before adsorption	After adsorption	Shift difference		
1	614.26	615.181	0.921	Strong C-Br stretching	Halo compound
2	1117.546	1110.797	6.749	Strong C- O stretching	Secondary alcohol
3	1379.818	1390.424	10.606	Medium C-H bending	Aldehyde
4	1633.412	1634.375	0.963	Medium C= C stretching	Alkene
5	2082.744	2081.780	0.964	Strong N= C= S stretching	Isothiocyanate
6	3439.421	3444.242	4.821	Strong O-H stretching bond	Alcohol

#### 4.5. X-Ray Diffraction Analysis

The key objective of X-ray diffraction is to gather structural information on activated carbon crystalline solids [60]. Activated carbons can be categorized into two categories depending on their graphitizing capacity, on the basis of XRD studies [31]. Due to the formation of strong cross-linking between adjoining randomly oriented elementary crystallites, non-graphitizing carbons are hard and have a well-developed micro porosity structure. Graphitizing carbons, on the other hand, showed poor cross-linking and a less developed porous structure. The X-ray diffraction patterns of flax straw activated carbon prepared at optimal conditions are shown in figure 4.3 below. The broad peak in the  $2\theta$  range (20.67–30.47) ° suggested that the main structures of prepared activated carbons are amorphous as expected which is a desirable feature for porous adsorbents with well-defined pores. Additionally, the lack of a distinct peak indicates that the majority of the activated carbon is amorphous, which is a favorable adsorbent characteristic [60]. This result was similar with previous studies on activated carbon produced from agricultural wastes [103, 104].

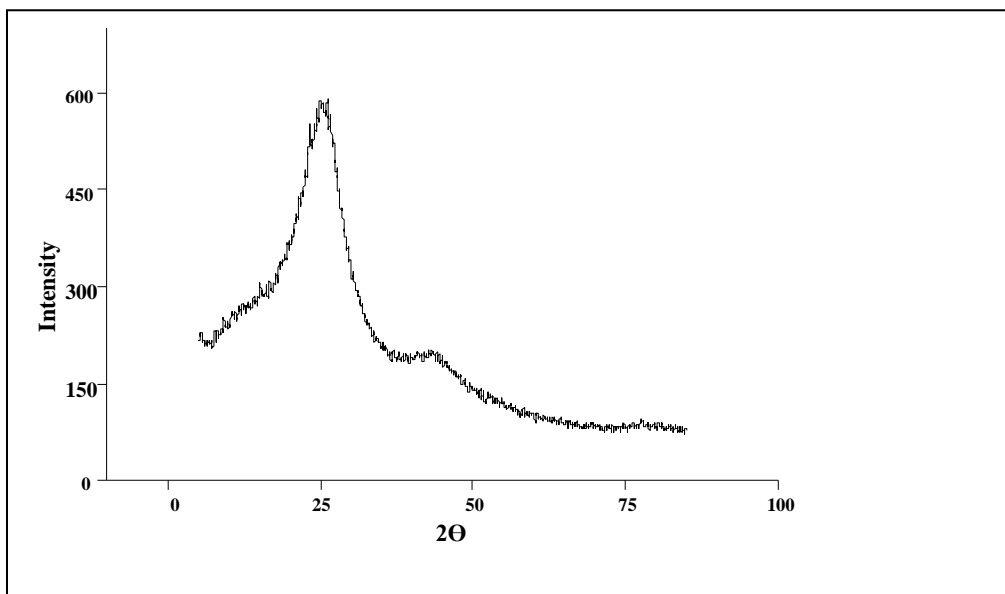


Figure 4.3: XRD patterns of the flax straw activated carbon

#### 4.6. Scanning Electron Microscopy

The main purpose of performing Scanning Electron Microscopy (SEM) technique was in order to observe the surface physical morphology of the flax straw activated carbon prepared by phosphoric acid as activating agent. Figure 4.4 show the SEM image with magnification of  $50\mu\text{m}$ . Pores of different size and different shape were observed from the result. It can be seen from the SEM image that the external surface of the chemically activated carbon is full of cavities or pores.

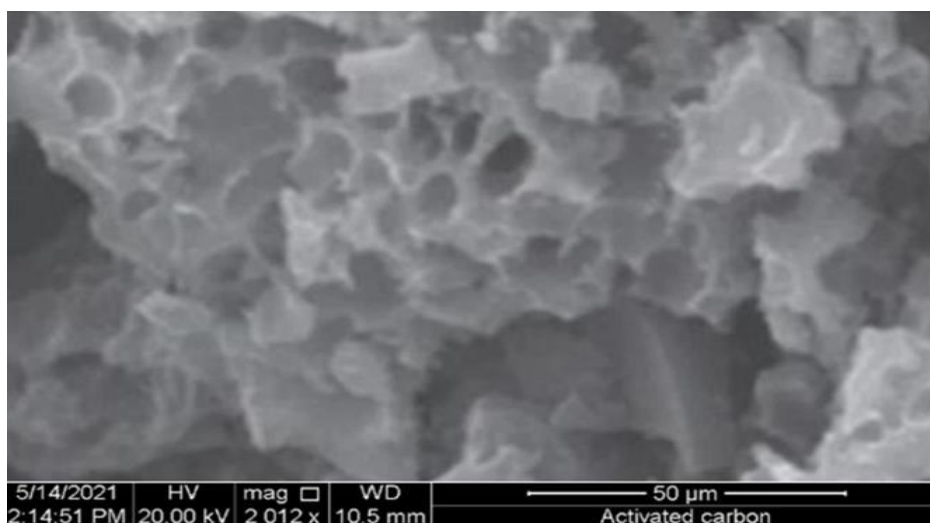


Figure 4.4: SEM images of the flax straw AC treated with phosphoric acid as activating agent

The pores on the surface of activated carbon are caused by the activating agent (phosphoric acid) evaporating during the carbonization of the sample in a muffle furnace under the optimum conditions, leaving empty spaces. Porosity is a great adsorbent texture because it gives a lot of surface area for the adsorption process [87].

#### 4.7. Specific Surface Area Analysis

In this study, the activated carbon prepared at optimum holding time, constant impregnation ratio and activation temperature was conducted using BET surface area analyzer. From the result of BET surface area analyzer, the average surface area of phosphoric acid treated flax straw activated carbon was found to be 489.4545 m<sup>2</sup>/g. Activated carbon derived from chemically activated agricultural wastes has a surface area ranging from (200 – 2000) m<sup>2</sup>/g [54]. The specific surface area of AC found in this study was higher than the value obtained for Indonesian Mangrove Charcoal which is 354.977 m<sup>2</sup>/g [92]. To sum up, activated carbon having high surface area would have better adsorption potential in heavy metal removing process.

Table 4.7: BET result for flax straw activated carbon

Sample Name	Sample weight(g)	Degas temperature	Degas time	SBET m <sup>2</sup> /g	Vm	BET const
Ac Biomass 1	0.0320	100	45	492.645	0.003	23.121
Ac Biomass 2	0.0330	100	45	486.264	0.003	26.320

#### 4.8. BBD Experimental Design Matrix and Results of Response Variables

The effects of processing parameters such as activation temperature, activation time, and activating agent concentration on the yield and iodine number adsorption of activated carbon were investigated. Quadratic models were used to correlate the effect of these parameters on the preparation of activated carbon using the Box–Behenken Design (BBD).

#### 4.9. Statistical Analysis of the Experimental Results

The statistical significance of the model and the results of corresponding response variables were analyzed using ANOVA as shown in table 4.8 and 4.9 below.

Table 4.8: ANOVA for yield of activated carbon preparation

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1544.18	9	171.58	217.69	< 0.0001	significant
A-Activation time	11.76	1	11.76	14.92	0.0062	
B-Activation temperature	111.60	1	111.60	141.60	< 0.0001	
C-Activating agent concentration	49.10	1	49.10	62.30	< 0.0001	
AB	121.00	1	121.00	153.52	< 0.0001	
AC	24.50	1	24.50	31.09	0.0008	
BC	9.36	1	9.36	11.88	0.0107	
A <sup>2</sup>	243.68	1	243.68	309.18	< 0.0001	
B <sup>2</sup>	389.05	1	389.05	493.62	< 0.0001	
C <sup>2</sup>	458.70	1	458.70	581.99	< 0.0001	
Residual	5.52	7	0.7882			
Lack of Fit	4.55	3	1.52	6.27	0.0542	not significant
Pure Error	0.9680	4	0.2420			
Cor Total	1549.70	16				

According to the design experiment data, the model F-value of 217.69 indicates that the model is statistically significant. Model terms with P-values less than 0.05 are significant. A, B, C, AB, AC, BC are significant model terms in this situation. The Predicted R<sup>2</sup> of 0.9521 is in reasonable agreement with the Adjusted R<sup>2</sup> of 0.9919; i.e. the difference is less than 0.2. Adeq. Precision measures the signal to noise ratio. A ratio greater than 4 is desirable.

Fit Statistics

Std. Dev.	0.8878	R <sup>2</sup>	0.9964
Mean	39.40	Adjusted R <sup>2</sup>	0.9919
C.V. %	2.25	Predicted R <sup>2</sup>	0.9521
		Adeq. Precision	37.0724

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In this research, a ratio of 37.0724 indicates an adequate signal. This model can be used to navigate the design space. A lower value of coefficient of variation,  $CV = 2.25\%$  indicates the precision with which the experiments were conducted.

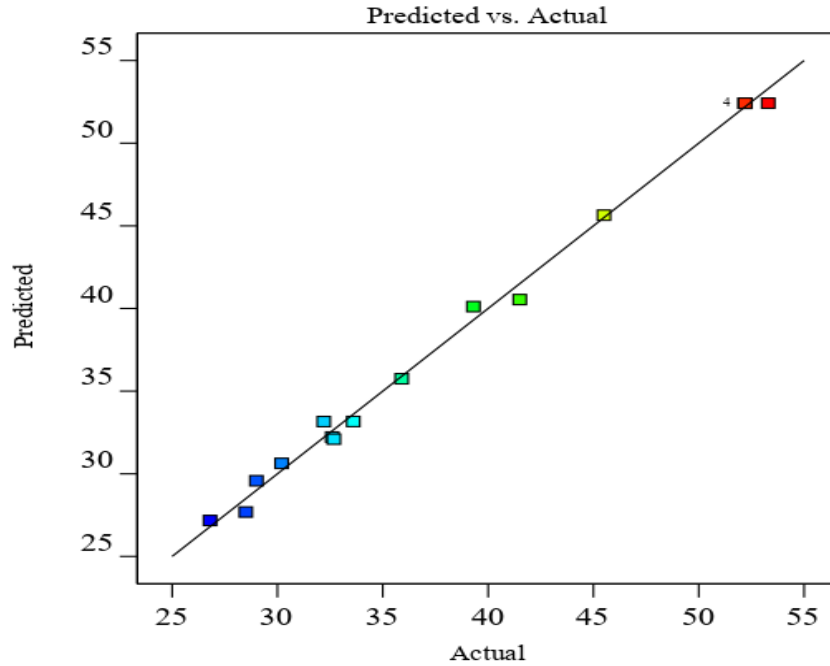


Figure 4.5: Diagnostic plot for the fitted model of yield

Table 4.9 indicates the ANOVA table for iodine number. From the data given in table 4.9 below, the Model F-value of 47.72 suggests the model is significant. P-values less than 0.05 indicate that the model terms are significant. In this case significant model terms are A, B, C, AC and BC

The Predicted  $R^2$  of this research was found to be 0.8048 which is in reasonable agreement with the Adjusted  $R^2$  of 0.9633; i.e. the difference is less than 0.2. Adeq. Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. In this research, ratio of 20.985 indicates an adequate signal. This model can be used to navigate the design space.

Table 4.9: ANOVA for iodine number

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	1.040E+05	9	11559.28	47.72	< 0.0001	significant
A-Activation time	21672.65	1	21672.65	89.46	< 0.0001	
B-Activation temperature	11102.49	1	11102.49	45.83	0.0003	
C-H <sub>3</sub> PO <sub>4</sub> concentration	2175.05	1	2175.05	8.98	0.0200	
AB	6.45	1	6.45	0.0266	0.8750	
AC	1445.17	1	1445.17	5.97	0.0446	
BC	5936.19	1	5936.19	24.50	0.0017	
A <sup>2</sup>	1517.84	1	1517.84	6.27	0.0408	
B <sup>2</sup>	40247.35	1	40247.35	166.14	< 0.0001	
C <sup>2</sup>	15397.91	1	15397.91	63.56	< 0.0001	
Residual	1695.77	7	242.25			
Lack of Fit	1246.17	3	415.39	3.70	0.1195	not significant
Pure Error	449.60	4	112.40			
Cor Total	1.057E+05	16				

**Fit Statistics**

Std. Dev.	15.56	R <sup>2</sup>	0.9840
Mean	372.44	Adjusted R <sup>2</sup>	0.9633
C.V. %	4.18	Predicted R <sup>2</sup>	0.8048
		Adeq Precision	20.9851

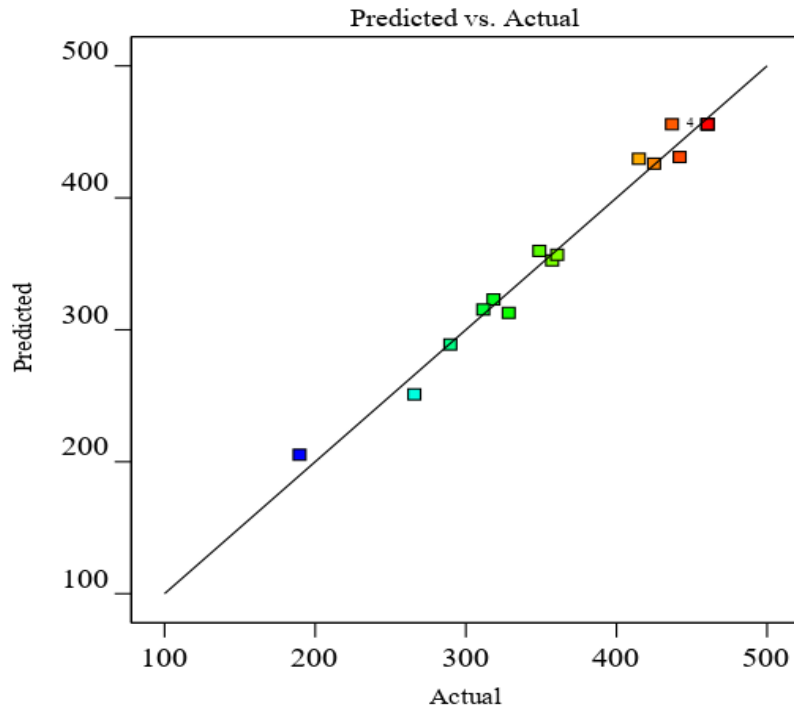


Figure 4.6: Diagnostic plot for the fitted model of iodine number

The final empirical formula models for the yield (Y) and iodine number (IN) in terms of coded factors are represented by Eq. (4.1) and (4.2), respectively.

$$\text{Yield} = 52.42 + 1.21A - 3.73B - 2.48C - 5.50AB - 2.48AC + 1.53BC - 7.61A^2 - 9.61B^2 - 10.44C^2 \quad (4.1)$$

*Iodine number*

$$= 455.85 + 52.05A - 37.25B - 16.49C - 1.27AB + 19.01AC - 38.52BC - 18.99A^2 - 97.77B^2 - 60.47C^2 \quad (4.2)$$

## 4.10. Effect of Activation Process Variables on Yield and Iodine Number

### 4.10.1. The Effect of Activation Time

The effect of activation time on the yield is shown in the figure 4.7 given below. As it indicated in the figure 4.7, the yield of activated carbon increased over the first 30 minutes, reaching its maximum value at 60 minutes. The reason for this is that, as activation time increase, the apparent density of carbon matrix increase which results in increasing of yield for the first 30 minutes of activation time period.

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The yield of flax straw activated carbon decreases from 52.41% to 46.12% when the activation time is increased from 60 to 90 minutes. This is most likely due to the release of volatile substances in combination with the creation of pores, and prolonged activation time may result in pore collapse and an increase in the proportion of meso and macropores.

The result of this research is in agreement with the work of Buah, W. K. and Kuma [89] who reports the effect of activation time on yield of activated carbon from Coconut Shells. In addition to this, the decrease in the yield of the activated carbon when activation time increase is accredited to increasing carbon-activation reagent reaction and consequently increases in the degree of carbon burn-off [31].

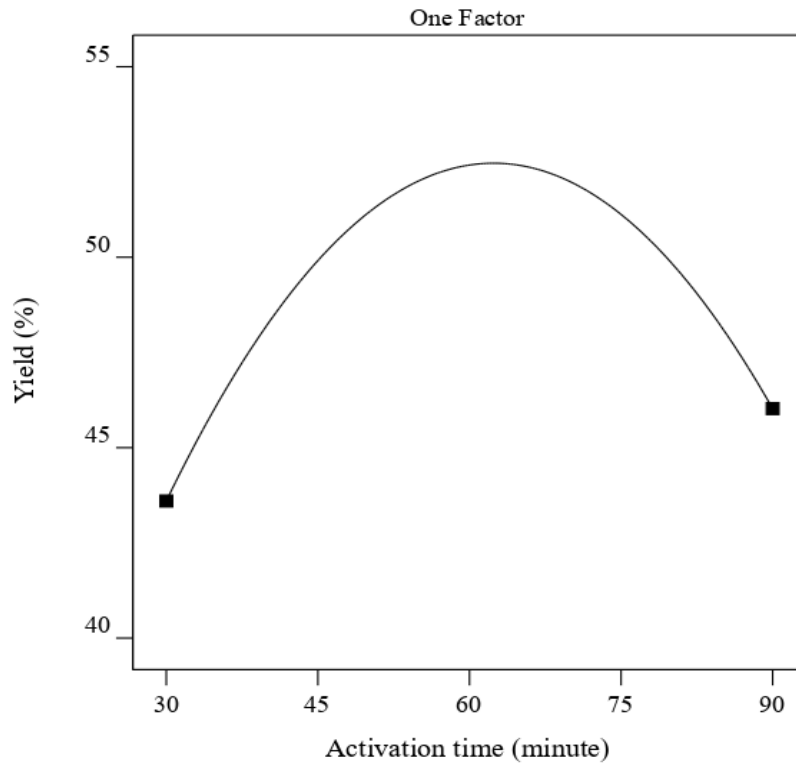


Figure 4.7: The effect of activation time on yield

Activation time has an impact not only on the yield of flax straw activated carbon, but also on the iodine number, as shown in figure 4.8. Iodine number is a crucial criterion for determining the quality of activated carbon. The iodine number demonstrates the carbon ability to adsorb contaminants and heavy metals in the form of solutions. As shown in figure 4.8, increasing the activation time increases iodine number due to the growth of pores that are responsible for iodine adsorption on its surfaces.

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The pore structure of activated carbon is gradually developed as the activation time increases, but due to the loss of carbon for an extended period of time, the micropore of activated carbon will tend to become mesoporous and macropore, and the specific surface area of activated carbon will decrease [1]. In general, the higher the iodine number value, the better the activated carbon for the adsorption process will be [93].

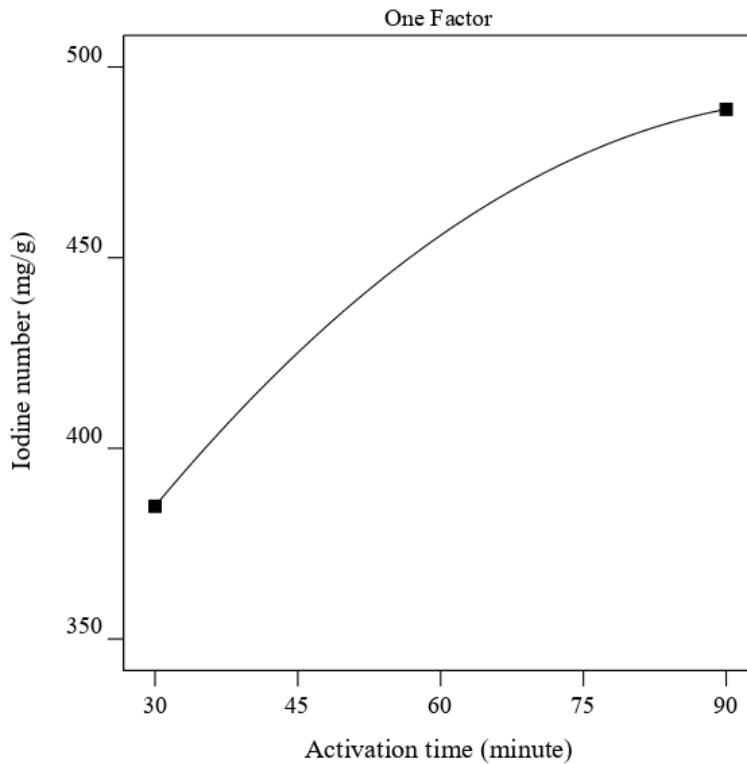


Figure 4.8: The effect of activation time on iodine number

#### 4.10.2. The Effect of Activation Temperature

The effect of activation temperature on the yield of flax straw activated carbon is indicated in the figure 4.9 below. The figure show that the yield of the flax straw activated carbon begin to increase with an increase of the temperature from 400<sup>o</sup>C to around 490<sup>o</sup>C, and then decrease when the temperature exceeds this value of activation temperature. As activation temperature increases, yield of AC also increases due to the increase of densification of carbon matrix during activation process since apparent density increases with activation temperature.

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An increase in temperature would cause more volatiles to be released as a result of increased dehydration and elimination reactions, which would also increase the reaction rate, lowering carbon yield [46]. As a result, the result of this study is consistent with previous research regarding to the relationship between activation temperature and activated carbon yield.

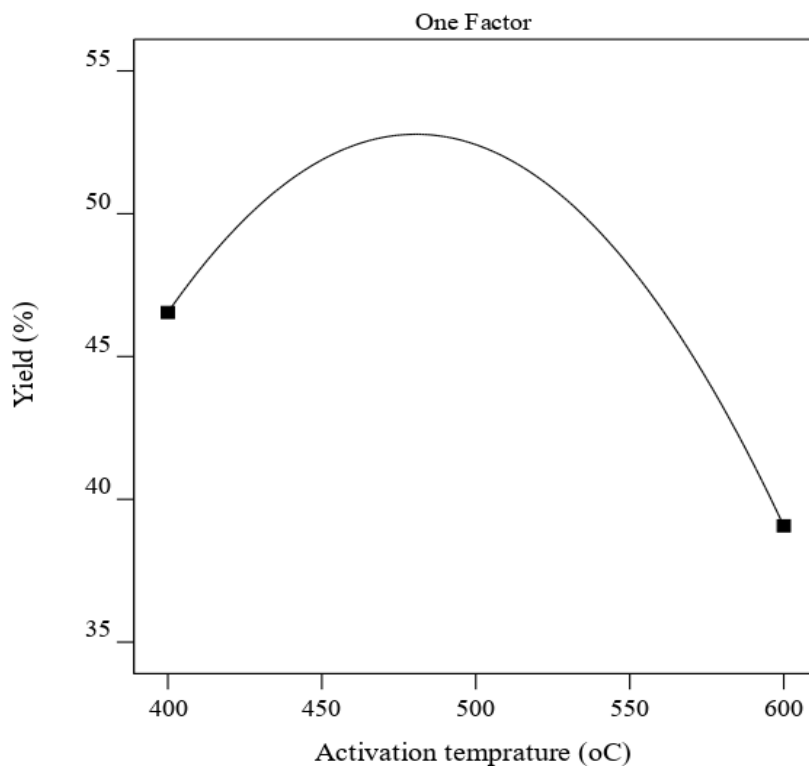


Figure 4.9: The effect of activation temperature on yield of flax straw activated carbon

Activation temperature has a significant influence on the iodine number [93] as it indicated in the figure 4.10. It has been observed from the experiment that the iodine number increase from 395.58 mg/g to 459.807 mg/g as the activation temperature increased from 400<sup>o</sup>C to 489.362<sup>o</sup>C but the iodine number gets decreased from 459.807 mg/ g to 321.685 mg/g as the temperature increase to 600 <sup>o</sup>C. Thus it is inferred that the optimum activation temperature is around 489.362<sup>o</sup>C for flax straw activated carbon using H<sub>3</sub>PO<sub>4</sub> as activating agent activation which is also considerable in terms energy saving. This result regarding to the relation between activation temperature and iodine number is in agreement with the previous work which describes the effect of activation temperature on the formation of active carbon pore structure [93].

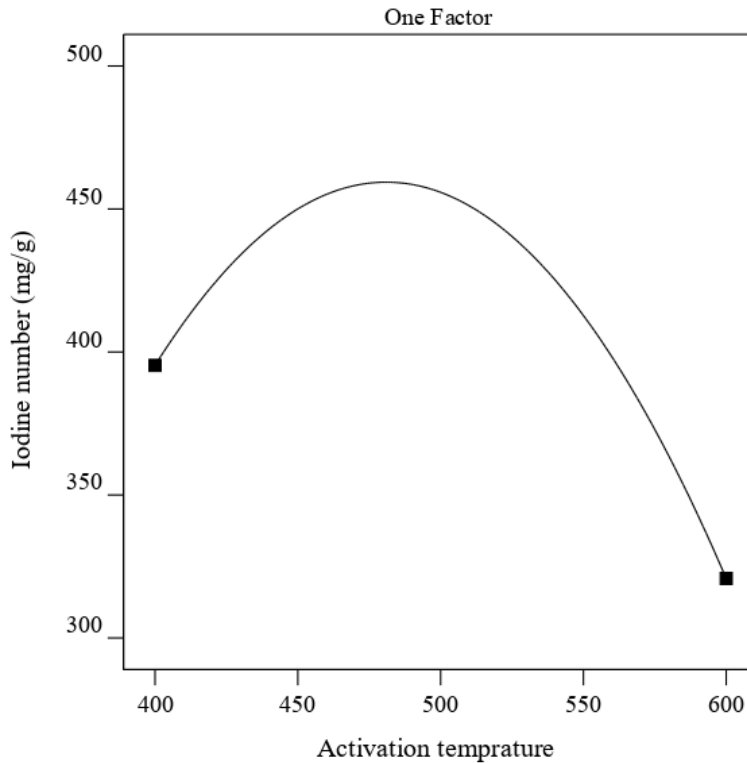


Figure 4.10: The effect of activation temperature on iodine number

#### 4.10.3. The Effect of Activating Agent Concentration

The yield of activated carbon increase as activating agent concentration increased [45]. As it can be seen from figure 4.11 below, the carbon yield of the flax straw activated carbon increases first rapidly from 45.558 to 52.638% with an increase of phosphoric acid concentration range (30 – 50)%  $H_3PO_4$ . Low concentrations of phosphoric acid resulted in a low yield of flax straw activated carbon. This is due to the fact that the interaction of phosphoric acid with the chemical constituents of flax straw activated carbon takes time to create a stable complex at a specific temperature.

Furthermore, when the concentration of  $H_3PO_4$  increased, the yield of flax straw increased until it reached a 50% concentration value of  $H_3PO_4$ . The yield of activated carbon produced by phosphoric activation (about 50%) is also worth considering because it is larger than the yield achieved by pyrolyzing the starting material alone [46].

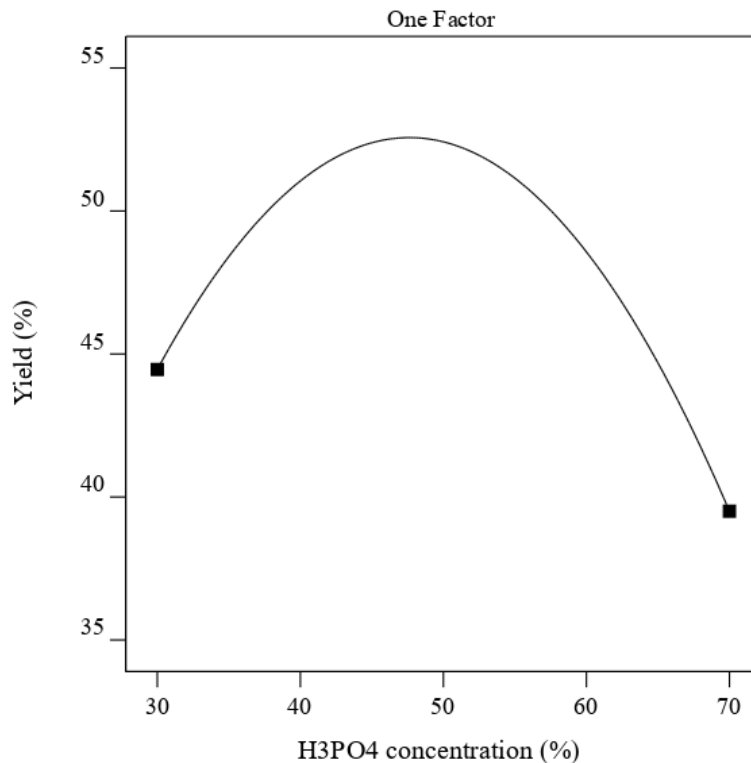


Figure 4.11: The effect of activating agent concentration on yield of flax straw activated carbon. The effect of phosphoric acid concentration on iodine number is also indicated in the figure 4.12 below. As shown in the figure 4.12, raising the concentration from 30% to 50% resulted in a higher iodine number due to increased total pore volume and surface area. However, as the phosphoric acid concentration was increased further, the iodine number of flax straw activated carbon gradually decreased due to action of phosphoric acid in improving mesoporous and broadening micro porous structures [94].

Low activating agent concentrations resulted in the lowest surface areas and adsorption volume, as well as a predominance of macropores generated by the diluted activating agent concentration. High activating agent concentration solutions, on the other hand, produce the best results, leading to increased porosity and iodine adsorption. However, if the activating agent concentration is too high, the pore structure of the activated carbon will be disrupted, and iodine absorption will be reduced [94].

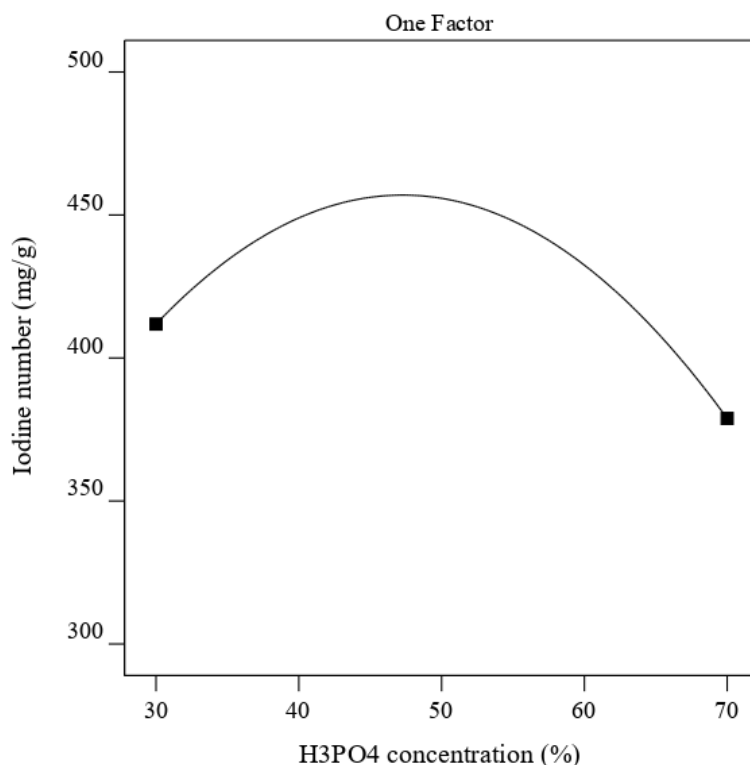


Figure 4.12: The effect of activating agent concentration on iodine number

#### 4.10.4. Interaction Effect of Activation Process Parameters on Yield and Iodine number

The preparation of activated carbons from the flax straw was mainly based on three process variables such as activation time, activation temperature, and H<sub>3</sub>PO<sub>4</sub> concentration. These independent variables have their own effects on the physical, textural characteristics and yield of the prepared activated carbons. The following sections explain the combined effects of variables on the production of flax straw activated carbons.

##### 4.10.4.1. Interaction Effect of Activation Time and Activation Temperature

The combined effect of activation temperature and activation time on the prepared activated carbon yield at impregnation ratio (2.75) is indicated in figure 4.13 below.

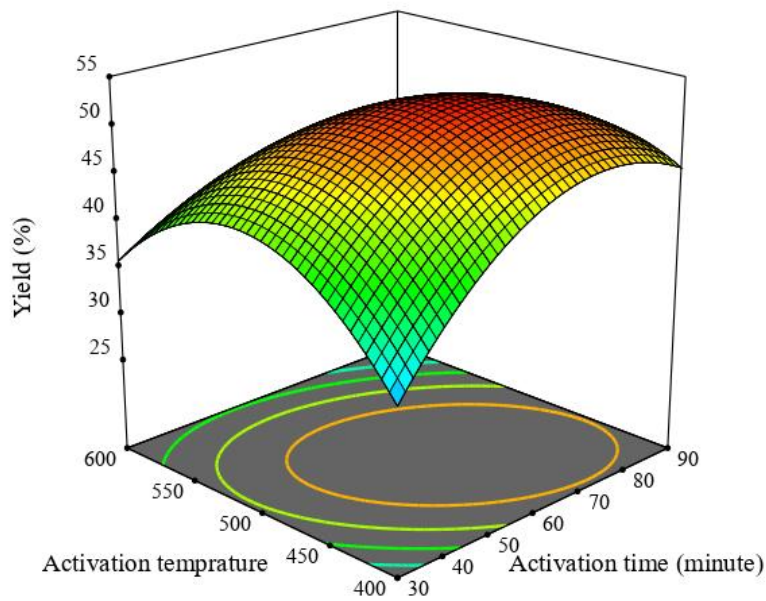


Figure 4.13: Interaction effect of activation time and temperature on the yield of flax straw AC

It can be observed from the figure 4.13 that the yield of flax straw activated carbon marginally changed when move on both sides of the activation time axis from low (30 min) to high (90 min) and activation temperature axis of 400<sup>o</sup>C to 600<sup>o</sup>C. These results show that activation time and activation temperature has its own effect on the yield of the flax straw activated carbon. As the activation temperature rises during the process, the yield of flax straw activated carbon falls steadily. The yield was low when the activation time and temperature were both high. The maximum yield of flax straw activated carbon was obtained at around 60 minutes of activation time and activation temperature of 489<sup>o</sup>C.

#### 4.10.4.2. Interaction Effect of Activation Time and H<sub>3</sub>PO<sub>4</sub> Concentration

Figure 4.14 illustrates the combined effect of activation time and H<sub>3</sub>PO<sub>4</sub> concentration on the yield of activated carbon. When we analyzed the plot by evaluating the combined influence of concentration and time on the yield of flax straw activated carbon, it was found that the yield of flax straw activated carbon was low at low time and concentration. This is because the interaction of phosphoric acid with the chemical elements of flax straw activated carbon took some time to produce a stable compound at a specific activation temperature.

Furthermore, as the activation time and H<sub>3</sub>PO<sub>4</sub> concentration were raised, the yield increased until the mid-concentration value of H<sub>3</sub>PO<sub>4</sub>, and as the phosphoric acid concentration was

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increased, the yield of the flax straw activated carbon declined due to an excess of chemical agent. The greatest yield of flax straw activated carbon was found to be around the midpoint of activation time and  $H_3PO_4$  concentration.

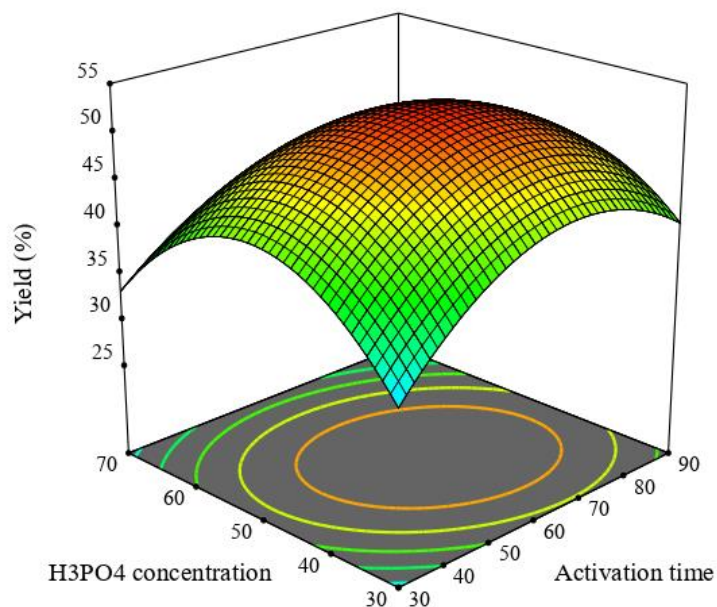


Figure 4.14: Interaction effect of activation time and  $H_3PO_4$  concentration on yield

The combined effect of activation time and  $H_3PO_4$  concentration on the iodine number of activated carbon is also shown in the figure 4.15 below.

Iodine number of flax straw activated carbon decreases as we move from low to high in activating agent concentration axis and increases along activation time axis as shown in figure 4.15. At around 60 minutes of activation time and a 50% activating agent concentration, the highest iodine number (460.587 mg/g) by flax straw activated was obtained.

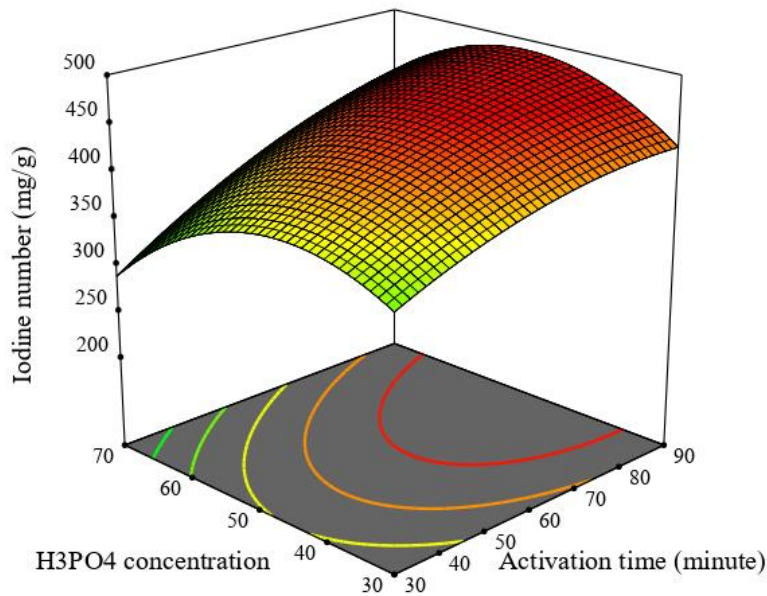


Figure 4.15: Combined effect activation time and  $\text{H}_3\text{PO}_4$  concentration on the iodine number

#### 4.10.4.3. Combined Effect of Activation Temperature and Activating Agent Concentration

Based on Figure 4.16 given below, the yield of flax straw activated carbon was increased as activation temperature increased starting from  $400^\circ\text{C}$  to around  $489^\circ\text{C}$  and  $\text{H}_3\text{PO}_4$  concentration of the first 50% amount.

At high activation temperature and high  $\text{H}_3\text{PO}_4$  concentration, the yield was found to be small. The maximum yield of flax straw activated carbon was found at around 50%  $\text{H}_3\text{PO}_4$  concentration and low activation temperature around  $489^\circ\text{C}$ . At maximum temperature the decrease of yield is obvious due to removal of some high temperature sensitive molecules.

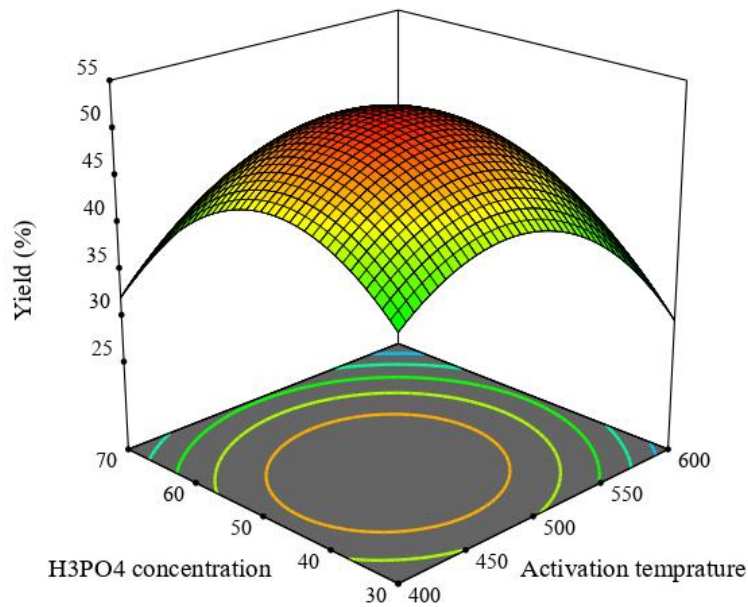


Figure 4.16: Combined effect of activation temperature and activating agent concentration

There is a marginal change in iodine number when we move in both directions of the activation temperature axis and the concentration axis as indicated in the figure 4.17. This demonstrates that both activation temperature and H<sub>3</sub>PO<sub>4</sub> have an impact on the flax straw activated carbon's iodine adsorption effectiveness. However, raising both elements more will likely reduce iodine number of activated carbon.

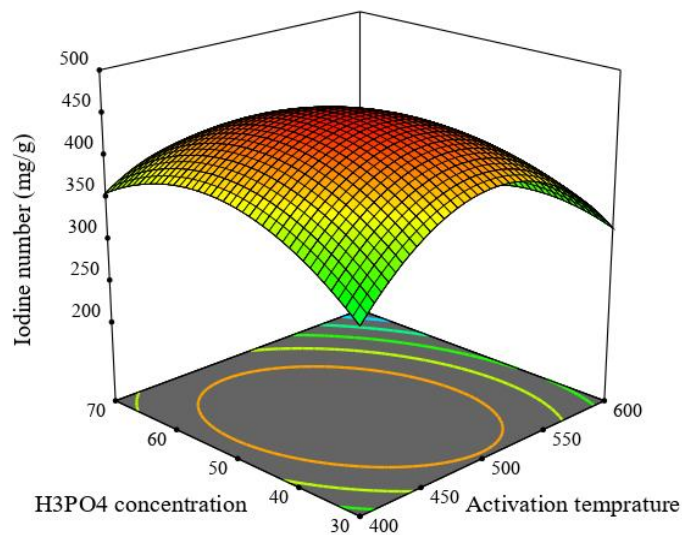


Figure 4.17: The effect of activation temperature and concentration H<sub>3</sub>PO<sub>4</sub> on iodine number

#### 4.11. Process Variables Optimization

In this research, the goal of optimization was to achieve flax straw activated carbon with maximum yield and maximum iodine number within the range of the process parameters. For economic feasibility, activated carbon made from flax straw must have high carbon yield as well as high iodine adsorption efficiency. In order to achieve these two goals, numerical optimization was conducted using Design-Expert software version-12.

Table 4.10: Constraints of the process parameters for the production of Flax straw AC

Name	Goal	Lower Limit	Upper Limit
A: Activation time	is in range	30	90
B: Activation temperature	is in range	400	600
C: Activating agent concentration	is in range	30	70
Yield	maximize	26.8	53.3
Iodine number	maximize	189.653	460.587

Table 4.11: Optimal processing conditions for optimization

Number	Activation time	Activation temperature	Activating agent concentration	Yield	Iodine number	Desirability	
1	66.059	473.515	46.747	53.238	467.458	0.999	Selected

Therefore, the final product of the flax straw activated carbon was prepared by using these optimum conditions given in the table 4.11 above. Figure 4.18 and 4.19 represents the desirability in 3D graphics for optimization of operating parameters, the optimum yield and iodine number of the flax straw activated carbon respectively.

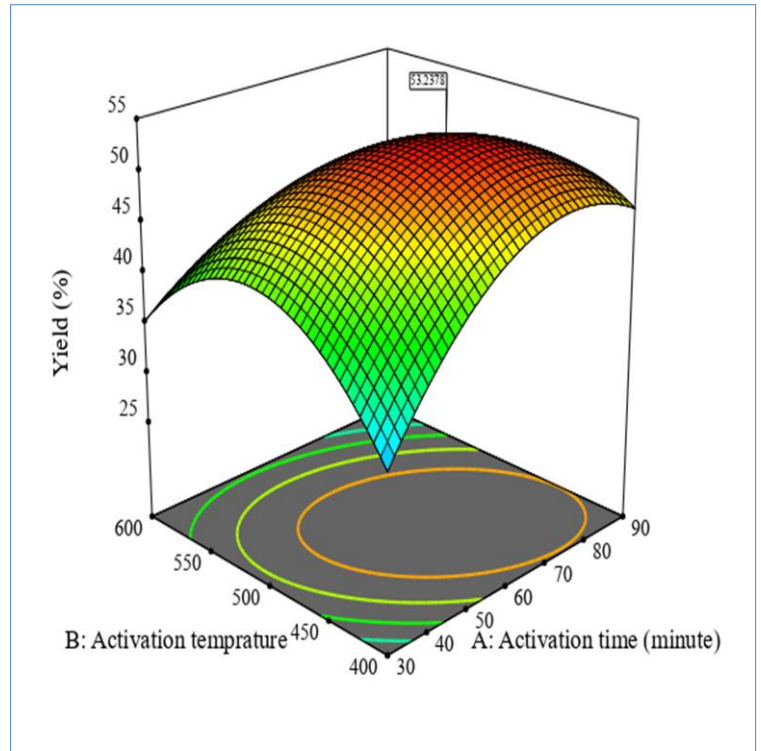
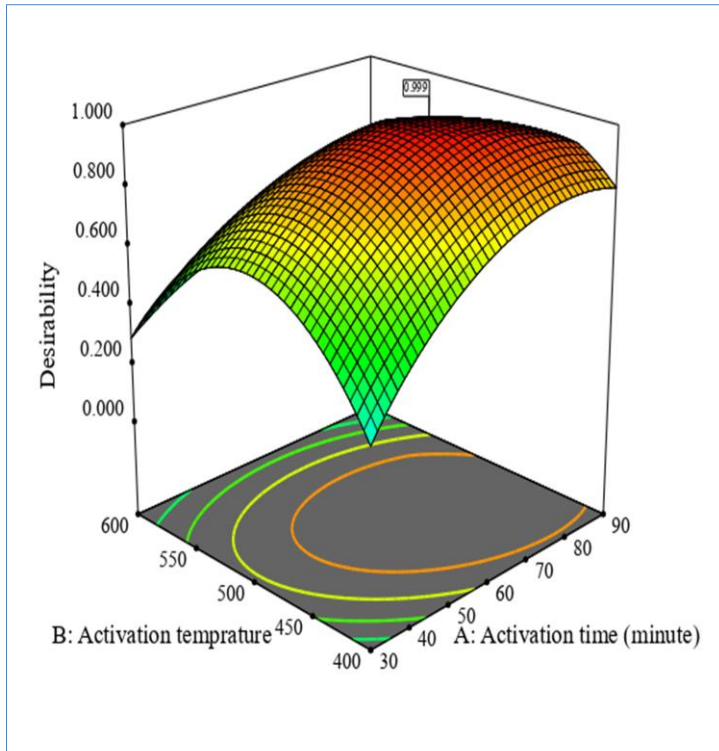


Figure 4.18: 3D plots for desirability and optimum yield of flax straw activated carbon

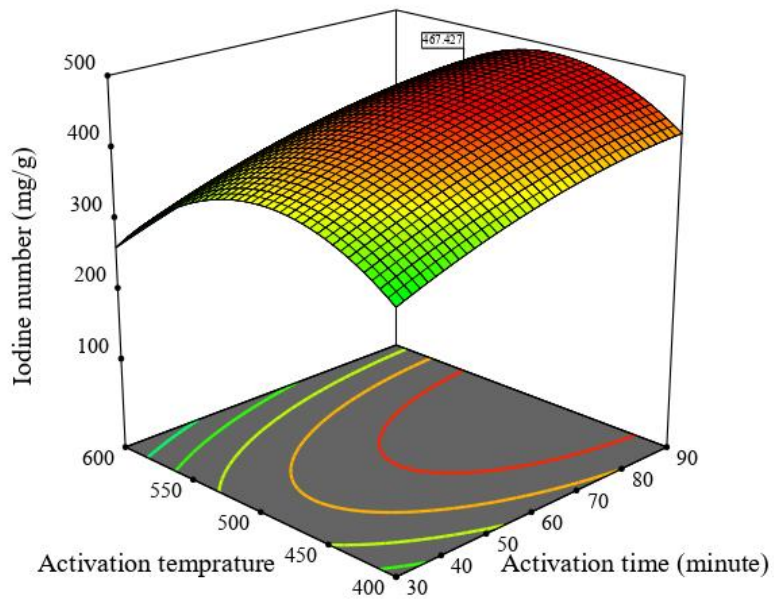


Figure 4.19: 3D plots for Optimum iodine number of flax straw activated carbon

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## 4.12. Adsorption Experiment Analysis

### 4.12.1. Calibration Curve Plot Preparation

The calibration curve plot data obtained from the experiment is presented in the table 4.12 below. And also, the graph of absorbance versus concentration is plotted for the standard solutions and it is presented in the figure 4.20 as it indicated below. The linearity of this plot arises from the Beer-Lambert law (or Beer's law, for short) which states that the absorption of light by a substance is proportional to its concentration in solution [95].

The equation for Beer's law is a straight line with the general form of  $y = mx + b$ .

- *Beer's Law*:  $A = (\epsilon l)C$  4.1
- With the general form  $y = (m)x$

Where:

- A is the absorbance (unit less),  $\epsilon$  is the molar absorptivity coefficient ( $M^{-1}cm^{-1}$ ),
- $l$  is the path length of the light through the cuvette (cm), and C is the concentration (M).
- The slope, m, is equal to  $(\epsilon l)$ ,

Table 4.12: Variation of absorbance with lead metal ions concentration

Concentration(ppm)	Absorbance
0	0
40	0.403
50	0.522
60	0.562
70	0.626
80	0.711
90	0.804
100	0.959

Thus, it is important to determine the value of final concentration knowing the absorbance directly from the UV-spectroscopy.

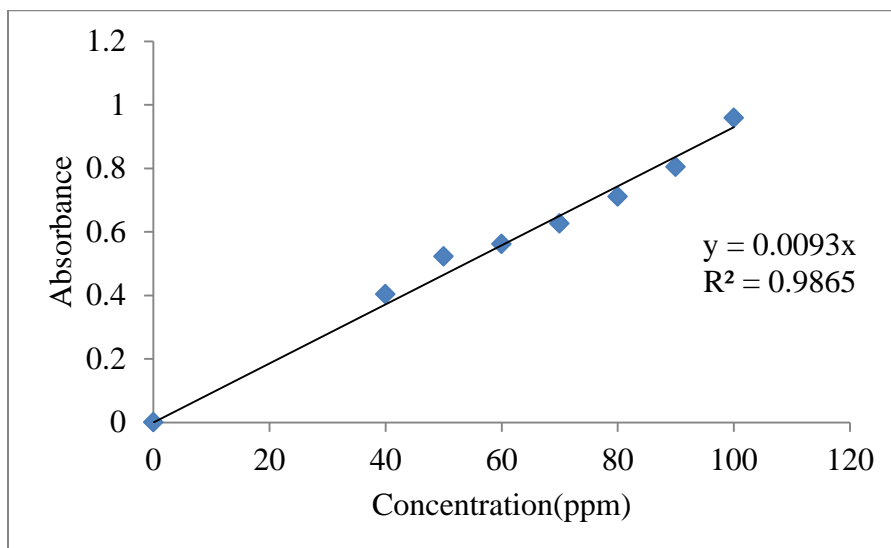


Figure 4.20: Calibration curve for lead metal ions solution

From the graph the slope was found to be 0.0093 so that final equilibrium concentration at any time can be determined by using slope and absorbance.

#### 4.13. Statistical Analysis of Adsorption Process Parameters

In this study statistical analysis and model fitting for adsorption experiment was carried out using Design Expert software version -12. The relationship between dependent response variables and a set of quantitative experimental factors was done by applying RSM. The ranges and levels of the experimental parameters are shown in table 4.13 given below.

Table 4.13: Coded and real values of operational parameters for the BBD statistical design

Factors	Name	Units	Minimum value	Middle value	Maximum value
A	pH of the solution	-	3.00	5.5	8.00
B	Adsorbent dose	g	1.0000	2.5	4.00
C	Initial concentration	mg/L	40.00	60	80.00
D	Contact time	minute	10.00	65	120.00

The analysis of variance (ANOVA) was used to examine the significance of the fitting model along with effects of their parameters.

Table 4.14: ANOVA for removal efficiency of the fitted models (Quadratic model)

Source	Sum of Squares	df	Mean Square	F-value	p-value	
Model	9398.04	14	671.29	56.87	< 0.0001	significant
A-PH of the solution	1258.58	1	1258.58	106.63	< 0.0001	
B-Adsorbent dose	1574.49	1	1574.49	133.39	< 0.0001	
C-Initial concentration	93.34	1	93.34	7.91	0.0138	
D-Contact time	2735.60	1	2735.60	231.76	< 0.0001	
AB	3.54	1	3.54	0.3000	0.5925	
AC	10.84	1	10.84	0.9187	0.3541	
AD	360.86	1	360.86	30.57	< 0.0001	
BC	1691.60	1	1691.60	143.31	< 0.0001	
BD	3.89	1	3.89	0.3292	0.5752	
CD	78.69	1	78.69	6.67	0.0217	
A <sup>2</sup>	560.16	1	560.16	47.46	< 0.0001	
B <sup>2</sup>	200.07	1	200.07	16.95	0.0010	
C <sup>2</sup>	380.36	1	380.36	32.22	< 0.0001	
D <sup>2</sup>	450.65	1	450.65	38.18	< 0.0001	
Residual	165.25	14	11.80			
Lack of Fit	154.72	10	15.47	5.87	0.0513	not significant
Pure Error	10.53	4	2.63			
Cor Total	9563.29	28				

Higher F-value indicates capability of variation about its mean and a p -value (Prob. > F) less than 0.05 indicates the model is significant [45]. Therefore, from the ANOVA for the response surface quadratic model for the removal efficiency of the flax straw activated carbon presented in table 4.14 above, the model F-value of 56.87 and P-values less than 0.05 indicate that model terms are significant. In this case, A, B, C, D, AD, BC, CD, A<sup>2</sup>, B<sup>2</sup>, C<sup>2</sup>, D<sup>2</sup> were found to be significant model terms. Whereas, AB, AC and BD were not significant model terms for this case because of their P- values are greater than 0.05.

## Fit Statistics

Std. Dev.	3.44	R <sup>2</sup>	0.9827
Mean	54.56	Adjusted R <sup>2</sup>	0.9654
C.V. %	6.30	Predicted R <sup>2</sup>	0.9051
		Adeq Precision	25.9168

The Predicted R<sup>2</sup> of this research was found to be 0.9051 which is in reasonable agreement with the Adjusted R<sup>2</sup> of 0.9654; i.e. the difference between is less than 0.2. Adeq Precision measures the signal to noise ratio. A ratio greater than 4 is desirable. The ratio found in this work is 25.917 and indicates an adequate signal. Therefore, this model can be used to navigate the design space.

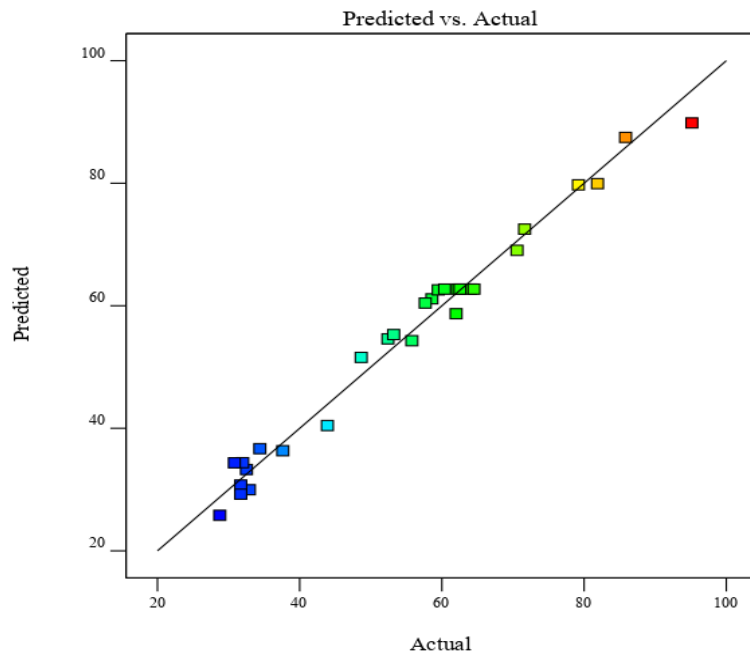


Figure 4.21: Comparison between the actual values and predicted values of model

Final equation in terms of coded factors is given in equation 4.3 below.

*Removal efficiency*

$$\begin{aligned}
 &= 62.72 - 10.24A + 11.45B + 2.79C + 15.10D - 9.5AD - 2056BC + 4.44CD \\
 &- 9.29A^2 + 5.55B^2 - 7.66C^2 - 8.34D^2 \quad (4.3)
 \end{aligned}$$

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## 4.14. Effects of Individual Factors on Pb (II) ions Removal Efficiency

### 4.14.1. Effects of pH

Figure 4.22 below shows the effect of change in solution pH on Pb(II) uptake by flax straw activated carbon. At a low pH values, the positively charged  $H^+$  ions are high and there is competition between positively charged  $H^+$  ions and Pb(II) metal ions for available active sites which result in lower removal efficiency [96]. The decrease in competition between protons ( $H^+$ ) and positively charged metal ions at the surface sites can explain the increase in metal removal when pH rises [7], [73]. Further increase of pH value above 4.53 causes the removal efficiency to decrease. The reason for this is that at high pH, there are more hydroxyl ions ( $OH^-$ ) in the solution which react with the Pb ions to form their insoluble hydroxides. Thus, precipitation takes place and it will clog the active site of the carbon which reduces the rate of adsorption [96].

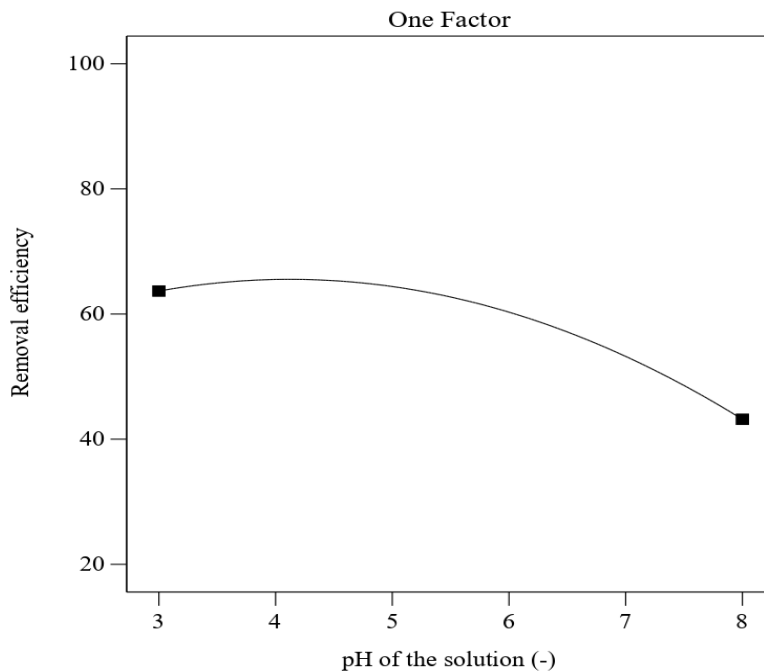


Figure 4.22: The Effects of pH on removal efficiency

### 4.14.2. Effects of Adsorbent Dosage

Adsorbent dose is another important parameter in the adsorption process. In this study, its effect on adsorption process can be seen from figure 4.23 as shown below. The removal of Pb (II) ions was increased while the adsorbent dose was increased from 1 to 4 g, as shown in figure 4.23.

This happens because increasing the adsorbent dosage results in a larger adsorption surface area [72]. A similar result was reported in the elimination of Pb (II) ion from paint industry wastewater using Activated Carbon Derived from African Arrowroot (Canna indica) Stem [7]. In the initial stages, the amount of lead adsorbed increased linearly with respect to the adsorbent mass showing that the increase in mass of adsorbent increased the amount of available adsorbent sites at which Pb(II) ions could adsorb [97].

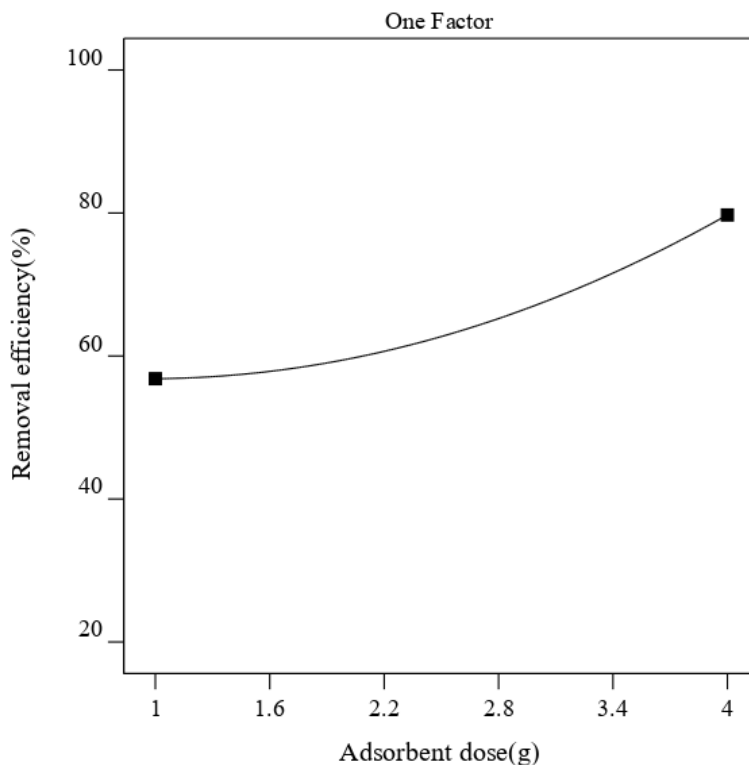


Figure 4.23: The effect of adsorbent dose on the removal efficiency of Pb (II)

#### 4.14.3. Effects of Initial Pb (II) Ions Concentration

The highest removal was achieved at a Pb (II) ion concentration between 61.1 and 66.3 mg/l as shown in figure 4.24. With the increase in concentration from 40.3 to 61.1 mg/l, the removal of Pb (II) ion was found to be small. The reason for this is that at low concentrations, the number of lead ions available in the solution is less compared to the available binding sites on the surface of activated carbon. As a result, all of the lead ions have the potential to interact with the active site. [97]. In contrast, higher initial concentrations resulted in more lead ions being linked to the adsorbent surface, causing the active site to become insufficient and adsorbent saturation, resulting in a drop in removal efficiency as it indicate in the figure 4.24 given below.

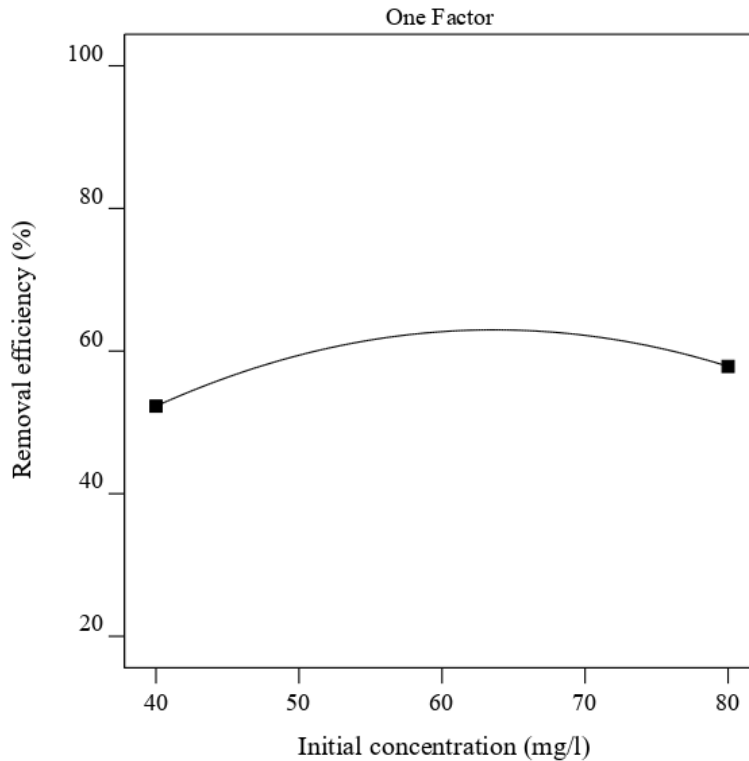


Figure 4.24: The effect of initial metal concentration on removal efficiency of Lead metal ions

#### 4.14.4. Effect of Contact Time on Removal Efficiency

Figure 4.25 depicts the effect of contact time on the adsorption efficiency of flax straw activated carbon. Because there are many active sites accessible on the adsorbent when the adsorption process begins, removal efficiency is usually increased initially as it indicates in the figure 4.25. The active sites of the adsorbent get exhausted as contact time rises, and the rate at which the adsorbate is transferred from the exterior to the internal sites of the adsorbent regulates the adsorption process [74].

In this research, there was a general increase in percentage removal of lead metal ions as contact time increased from 10 to 106 minutes until equilibrium was reached. Because there were more unoccupied spaces on the surface of the activated carbon at the beginning, and metal ion uptake was higher, there was a continuous increase in adsorption capacity by expanding the time slot from the start(10 minutes in this case) to 106 minutes. However, further increases would not be sufficient to create a sufficient change in metal adsorption because unoccupied areas have already been filled, and equilibrium has been reached.

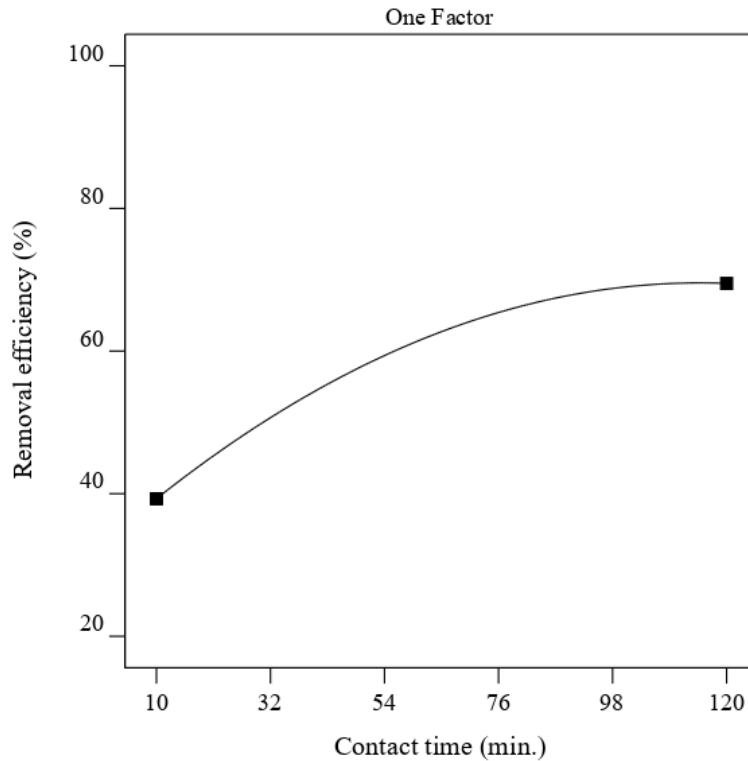


Figure 4.25: The effect of contact time on removal efficiency Pb(II) ions

#### 4.15. Interaction Effect of Parameters on Removal Efficiency

This section explains the combined (interaction) effects of parameters on the percentage removal efficiency. According to the results of ANOVA, A, B, C, D, AD, BC and CD are significant model terms. The effects of the first four parameters are already discussed before. Now, the discussion for the rest three model terms explained below.

##### 4.15.1. Interaction Effect of pH and Contact Time

The combined effect of pH and contact time on the removal of lead metal ions is indicated in the figure 4.26 below. As shown in the figure 4.26, increase in the contact time provided more Pb (II) adsorbed on its surface of the adsorbent. And also, as the pH of the solution increases, the adsorption efficiency of flax straw activated carbon increases.

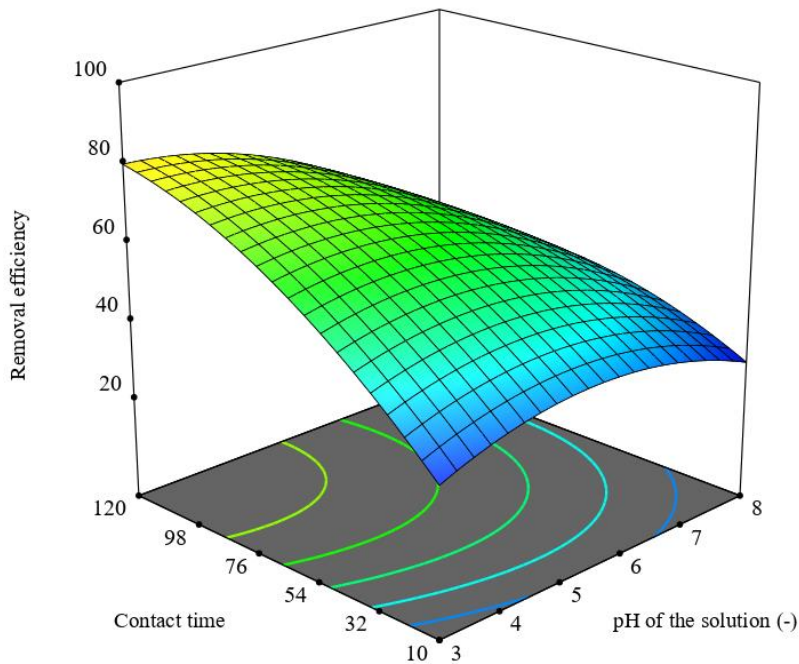


Figure 4.26: The combined effect of contact time and pH on removal efficiency

As the pH of the solution rises, the activated carbon's ability to remove impurities decreases. When the pH of the adsorbing medium was raised from 3 to 8, deprotonation of the adsorbent surface increased, resulting in a decrease in  $H^+$  ion on the adsorbent surface [5]. As it indicates in the figure 4.26 above, removal efficiency is low at maximum contact time and pH. More negative charges are created on the adsorbent surface, favoring the adsorption of positively charged species and positive sites on the adsorbent surface.

#### 4.15.2. Interaction Effect of Adsorbent Dose and Initial Concentration

The combined effects of adsorbent dose and initial concentration on the adsorption efficiency of Pb (II) are depicted in figure 4.27 as a 3D plot. Increase in the adsorbent dose provided more surface area or increased available adsorptions sites which increased the amount of Pb (II) adsorbed on its surface as seen in the figure 4.27.

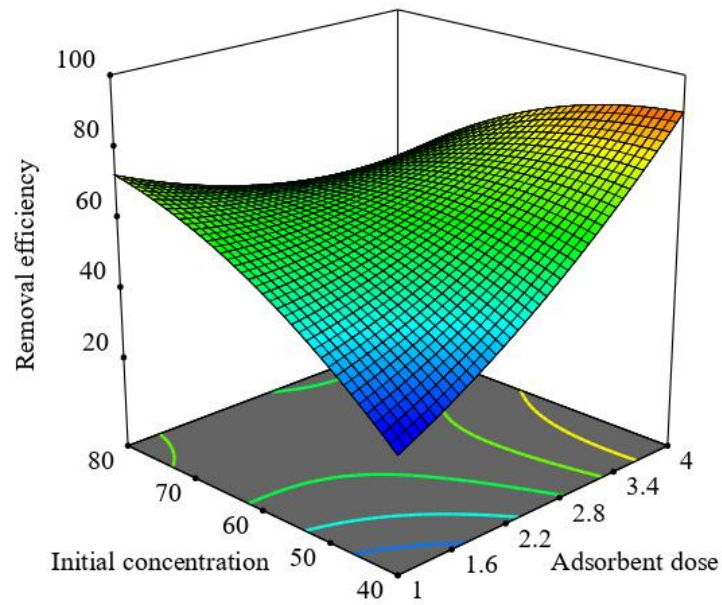


Figure 4.27: The effect of adsorbent dose and initial concentration on removal efficiency

#### 4.15.3. Interaction Effect of Contact Time and Initial Concentration

As shown in the figure 4.28, the removal efficiency of activated carbon was low at maximum values of contact time and initial lead metal concentration.

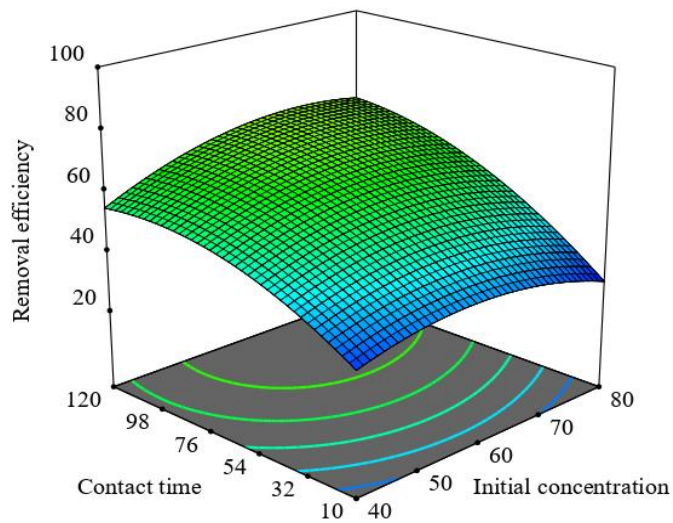


Figure 4.28: The interaction effect of contact time and initial concentration

## 4.16. Process Parameters Optimization in Adsorption Experiment

The constraints for parameters optimization is indicated in the table 4.15 given below. The optimum values of parameters were taken into account for the study of isotherm models, kinetic models, and Pb (II) ion removal from paint industry effluent.

Table 4.15: Constraints for optimization for adsorption process

Name	Goal	Lower Limit	Upper Limit
A: PH of the solution	is in range	3	8
B: Adsorbent dose	maximize	1	4
C: Initial concentration	is in range	40	80
D: Contact time	is target = 101	10	120
Removal efficiency	maximize	28.7634	95.1613

Table 4.16: Optimal processing conditions of batch adsorption experiment

Number	PH of the solution	Adsorbent dose	Initial concentration	Contact time	Removal efficiency	Desirability	
1	4.868	4.000	46.773	101	96.195	1.000	Selected

## 4.17. Adsorption Isotherm Study

Batch adsorption characteristics of lead metal ions removal by straw activated carbon were investigated in this study, and the results were applied to identify which isotherm model the adsorption process follows. The determination of the isotherm with the best correlation to the experimental results, as well as the assessment of the isotherm characteristics, is a well-known first step in the investigation of adsorption.

### 4.17.1. Langmuir Isotherm Model

Applicability of Langmuir isotherm model for lead metal ions removal was analyzed using the data obtained from batch adsorption experiment by plotting  $\frac{C_e}{q_e}$  versus  $C_e$ . Figure 4.29 below shows Langmuir plot of lead metal ions adsorption at room temperature, pH of 4.9, activated carbon of dosage of 4g and contact time of 101 minutes and for different initial lead metal

solution concentrations. The values of Langmuir constants,  $K_L$  and  $q_m$  were calculated from the intercept and slopes of the linear plot and summarized in table 4.18.

Table 4.17: Langmuir Isotherm for Adsorption of  $Pb^{2+}$  ions using flax straw activated carbon

Co(mg/l)	(Ce)	(qe)	Ce/qe	log (Ce)	Log(qe)
20	3.11828	0.42	7.38854	0.49392	-0.3746
30	9.46237	0.51	18.4293	0.976	-0.2895
40	12.6882	0.68	18.5827	1.1034	-0.1657
50	18.6022	0.78	23.6986	1.26956	-0.1052
60	25.3763	0.87	29.3168	1.40443	-0.0627
70	33.4409	0.91	36.5882	1.52428	-0.0391
80	37.957	1.05	36.1125	1.57929	0.02163
90	47.2043	1.07	44.1206	1.67398	0.02934

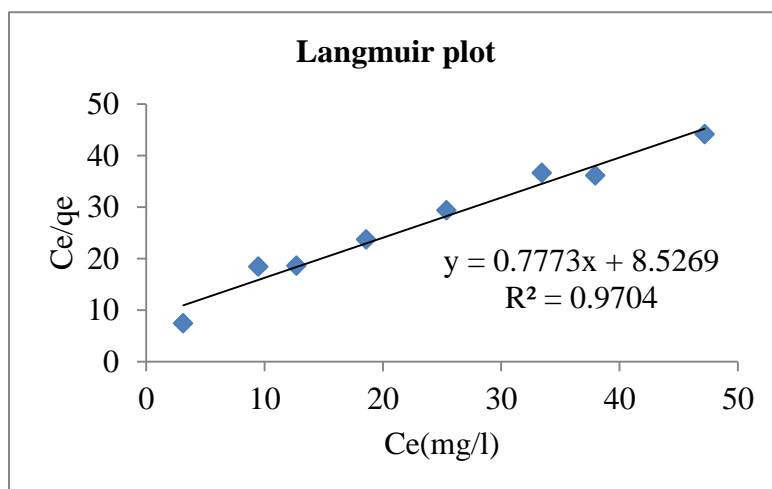


Figure 4.29: Langmuir adsorption isotherm plot for adsorption of  $Pb^{2+}$  on flax straw AC

As it indicated in figure 4.30 above, the plots has  $R^2$  value of 0.9704 with slope and intercept values of 0.77 and 8.53 respectively.

#### 4.17.2. Freundlich Isotherm

Figure 4.30 shows the linear plot of  $\log(C_e)$  versus  $\log q_e$  with  $R^2$  value of 0.9587, slope and intercept of 0.366 and -0.583 respectively.

The Freundlich parameters ( $K_F$  and  $n$ ) for the adsorption of Pb(II) ion on flax straw AC are determined from the slope and intercept values of the equilibrium data and are indicated in table 4.18.

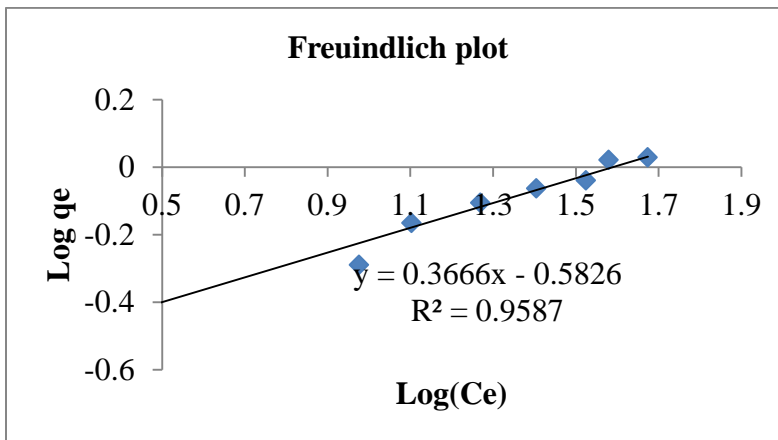


Figure 4.30: Freundlich isotherm model for adsorption of lead metal on flax straw AC

Table 4.18: Values of Langmuir and Freundlich adsorption Isotherm Parameters

Isotherm model	Parameters	Values
Langmuir	$q_m$ (mg/g)	1.29
	KL (L/g)	0.09
	$R^2$	0.9704
Freundlich	KF(mg/g)	0.26
	nF	2.727
	$R^2$	0.9587

From the table 4.18 above, the correlation coefficients ( $R^2$ ) of Langmuir and Freundlich isotherms models were found to be 0.9704 and 0.9587, respectively. Therefore, from this result we can say that the adsorption of Pb (II) on flax straw activated carbon fits the model of Langmuir very well. The Freundlich parameters,  $n$  gives an indication of the favorability and capacity of the adsorbent/ adsorbate system [72]. In table 4.18, the value of  $n$  for lead adsorption was found to be 2.727, for flax straw activated carbon. Similar results were found for the removal of Pb (II) from Aqueous Solutions using Lapsi seed stone and sugarcane bagasse [20, 98] the adsorption of lead metal ions from aqueous solution is best described by Langmuir isotherm model which is in consistent to this study.

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## 4.18. Adsorption Kinetic Study

Kinetic models (pseudo-first order and pseudo-second order) were investigated at optimum conditions (adsorbent dosage of 4g, initial lead solution concentration of 46.7 mg/l and pH of 4.9). Both kinetic models were based on the assumption that the rate of occupation of adsorption sites is proportional to the number of unoccupied sites. The best fit model was selected based on the linear regression correlation coefficient ( $R^2$ ).

### 4.18.1. Pseudo-First Order Kinetic Model

Pseudo-first order kinetic model and its respective adsorption model parameters were determined from slope and intercept of linear plot of  $\log(Q_e - Q_t)$  versus time as shown in the figure 4.31 below.

Table 4.19: Pseudo-first order kinetics experimental data

Time(minutes)	Ct(ppm)	Qt(ppm)	Qe	Qe-Qt	log(Qe-Qt)
10	34.193	0.197	0.45	0.253	-0.596
20	29.247	0.259	0.45	0.191	-0.718
30	21.935	0.35	0.45	0.1	-1
40	19.569	0.38	0.45	0.07	-1.154
50	16.344	0.421	0.45	0.029	-1.537
60	14.301	0.446	0.45	0.004	-2.397
70	14.516	0.443	0.45	0.007	-2.154
80	14.408	0.444	0.45	0.006	-2.221

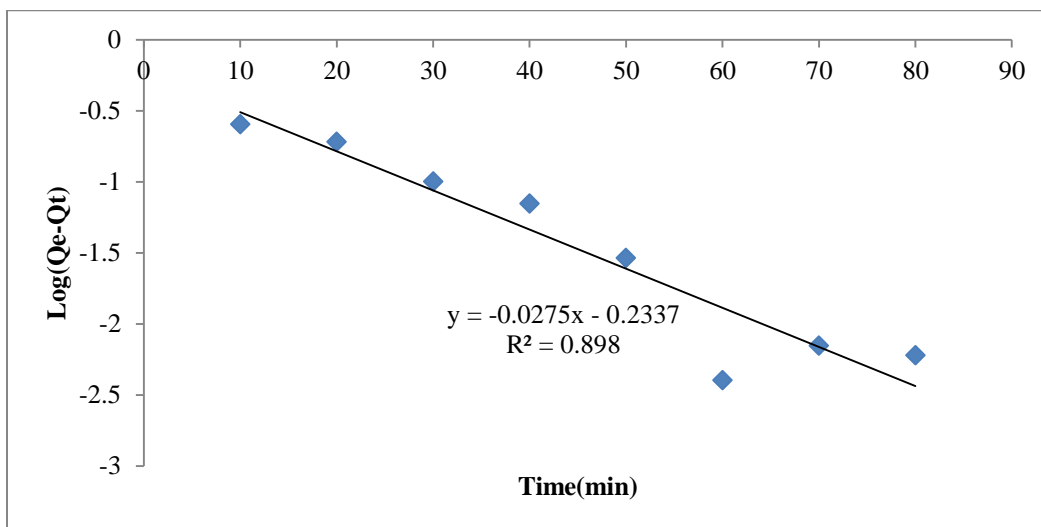


Figure 4.31: Linear plots of  $\log(Q_e - Q_t)$  versus time for Pseudo-first order kinetics. The  $K_1$  and  $Q_e$  values were determined from the slope and intercept of the linear plots respectively and given in table 4.21.

#### 4.18.2. Pseudo-Second Order Kinetic Model

The pseudo second order kinetic model was studied with different contact time and the results were described in figure 4.32 shown below. The figure does give the linear plots of  $\frac{t}{qt}$  against  $t$ . The values of  $n$  and  $K_2$  were calculated from the slope and intercept of the respective plots and are presented in table 4.21.

Table 4.20: Pseudo-second order kinetics data

Time(minutes)	Ct(ppm)	qt(ppm)	t/qt
10	34.193	0.197	50.612
20	29.247	0.259	77.098
30	21.935	0.350	85.517
40	19.569	0.380	105.159
50	16.344	0.421	118.849
60	14.301	0.446	134.457
70	14.516	0.443	157.818
80	14.408	0.444	179.818

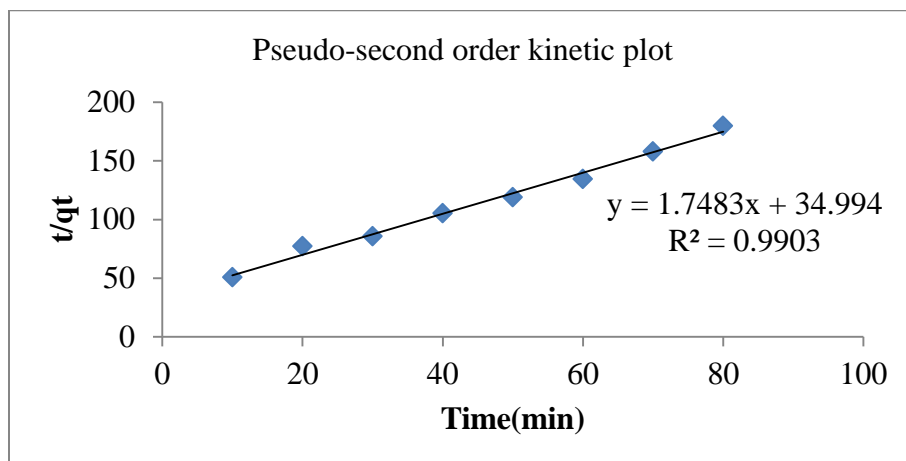


Figure 4.32: Pseudo-second order kinetic plot

According to the values of correlation coefficient,  $R^2$  as indicated in the table 4.21 below, pseudo-second order model showed a higher ( $R^2 = 0.9903$ ) value which indicate that the kinetics of adsorption of lead metal adsorption by flax straw activated carbon could be better described by pseudo-second order model.

Table 4.21: Kinetics Model Parameters and Correlation Coefficient for adsorption kinetics study

Kinetic models	Pseudo-first order model			Pseudo-second order model		
	$K_1(\text{min}^{-1})$	$q_e(\text{mg/g})$	$R^2$	$K_2(\text{min}^{-1})$	$q_e(\text{mg/g})$	$R^2$
parameters						
values	0.061	0.556	0.898	0.087	0.572	0.9903

Similar reports were found about the removal of Pb (II) ions from aqueous solutions by activated carbon prepared from a waste biomass was best described by the pseudo-second-order kinetic model [6]. Furthermore, the kinetics data for adsorption of lead (II) ions by activated carbons is best described by the pseudo second-order kinetics [5], [66], [67].

#### 4.19. Flax Straw AC Performance for Lead Removal from Real Wastewater

After optimization was carried out using synthetic wastewater prepared from stock solution of lead, analysis were conducted for removal of Pb(II) from the real wastewater. Table 4.22 given below show the characteristics of untreated and treated wastewater collected from KADISCO paint industry. Except temperature, other parameters in untreated wastewater exceeded the standard established EEPA [99].

The concentration of Pb (II) ions, in particular, was found to be much greater than the standard, indicating that it must be removed to avoid environmental vulnerabilities. The results indicate that highly polluted effluent of the Pb (II) value was 3.95 mg/L as against the maximum limited Pb (II) which is 0.50 mg/L [99].

Table 4.22: Physiochemical analysis of the treated paint effluent

Parameters	Untreated wastewater	Treated wastewater	Max permissible Limit
Lead(Pb), mg/L	3.95	0.84	0.5
Turbidity,NTU	3394	20.55	25
pH	4.5	6.9	6 - 8.5
BOD <sub>5</sub> ,mg/L	158.52	31.97	50
COD, mg/L	2482	162.4	150
DO, mg/L	0.152	7.25	5 - 20
TSS, mg/L	652.667	145	50
TDS, mg/L	604.444	144.444	-
Temperature( <sup>o</sup> C)	20.2	20.1	Ambient temperature $\pm$ 3

The treated wastewater characteristics were below the standard set by WHO (2017) except for COD and TSS. Hence, further treatment is needed to remove the excess COD and TSS before disposing to the environment. In this research, it was found that the removal efficiencies of Pb (II) ion from aqueous solution is 95.16% and from the paint industry wastewater it was found to be 78.73%.

As the result of this research show, the performance of the flax straw activated carbon for the removal of Pb (II) ion in the paint industry wastewater was found to be much lower than the aqueous solution. The reason for this to happen could be paint industry wastewater contained different types of heavy metals like arsenic, chromium, copper, mercury, nickel, and silver, biological oxygen demand (BOD<sub>5</sub>), chemical oxygen demand (COD), total suspended solids and turbidity that affected the Pb (II) ion removal efficiency by competing one another in the flax straw activated carbon active site.

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## 5. Conclusions and Recommendation

### 5.1. Conclusion

Depending on the finding of this study, the following conclusions can be drawn:

- agricultural wastes of different varieties could have different carbon content and volatile matter. Therefore, it is important selecting the raw materials having high amount carbon and volatile matter for the preparation of activated carbon in order to save energy, cost and time.
- the physico- chemical analysis of flax straw AC results were found as that the flax straw AC had 8.04% of moisture, 6.04% of ash, 18.615% of volatile matter, 79.421% of fixed carbon, 459.807 mg/g of iodine number and surface area of 489.455 m<sup>2</sup>/g.
- physico-chemical characteristics revealed that raw wastewater had a concentration of 3.95 mg/L Pb (II), 158.52 mg/L BOD<sub>5</sub>, 2482 mg/L COD, and 652.667 mg/L TSS.
- the optimum conditions of Pb (II) removal from paint wastewater on the flax straw AC were obtained as pH 4.9, adsorbent dose 4 g, contact time 101minutes and initial concentration 46.77 mg/L.
- the removal efficiency of flax straw activated carbon from aqueous or synthetic solution is 95.16% and from the paint industry is 78.73%. This percentage removal of lead metal ion was achieved by employing the optimum parameters.
- the removal performance of the flax straw activated carbon for Pb (II) ion is lower in the paint industry wastewater than the removal in aqueous solution due to the occurrence of various types of heavy metals and pollutants like zinc, copper nikel, BOD, COD.
- in this study, the adsorption of Pb (II) on flax straw activated carbon fits the model of Langmuir very well. And also, the kinetics study of lead metal adsorption was better described by pseudo-second order model.
- to sum up, this research showed that, the adsorption process using flax straw activated carbon were inexpensive, environmental friendly and has potential to adsorb pollutants and toxic heavy metals from both synthetic solution and paint industry wastewater.

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## 5.2. Recommendation

The following recommendations were made for the additional study in the preparation and application of activated carbon from flax straw for the removal of pollutants and heavy metals from paint industry wastewater.

- In this research, activation time, activation temperature and activating agent concentration were considered for activated carbon preparation by keeping impregnation ratio constant in order to get maximum yield and iodine adsorption, therefore it is recommended for further study using different impregnation ratio, in order to get maximum yield and iodine number adsorption.
- In this study activated carbon was prepared by single step chemical activation method, therefore, it is recommended that future study can prepare activated carbon from flax straw by two step activation methods in order to get higher pore volume and surface area.
- In this study phosphoric acid was used as activating agent to prepare activated carbon, for future study it is recommended to use other activating agent in order to get high yield and high iodine adsorption capacity.
- This study was conducted by considering Pb(II) metal ion concentration, pH of the solution, adsorbent dose and contact time, for future research, it is recommended to do adsorption study at various temperatures and agitation speed for lead adsorption on flax straw activated carbon.
- Lead metal ion removal was the main interest in this research; therefore, future research can focus on other metals removal by flax straw AC from wastewater.
- This research was conducted only the removals of lead metal ions from paint wastewater, further work should be conducted on removal of other heavy metals by using flax straw activated carbon.

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## Appendices

### Appendix A. Solution preparation calculation for different phosphoric acid solution concentrations

- In this study phosphoric acid solutions of different concentration were prepared based on the following steps.
- Solution from of 85% phosphoric acid was bought from the market and its density was 4.71 g/ml and MW 98 g/mol at 20<sup>0</sup>C.

**Step 1:** Calculate the mass of solution

$$\text{Density} \left( \frac{g}{ml} \right) = \frac{\text{Mass of solution}(g)}{\text{Volume of solution}(ml)} \quad \text{A.0.1}$$

From equation A.1, Mass of solution is given by:

$$\text{Mass of solution}(g) = \text{Density} \left( \frac{g}{ml} \right) \times \text{volume of solution}(ml) \quad \text{A.0.2}$$

$$\text{Mass of solution}(g) = 4.71 \left( \frac{g}{ml} \right) \times 1000(ml) = 4710g$$

**Step 2:** Determine concentration in percent by mass of the solute in solution

$$\text{Mass of } H_3PO_4 = \%H_3PO_4 \times \text{Mass of solution}$$

$$\text{Mass of } H_3PO_4 = 0.85 \times 4710g = 4003.5g$$

**Step 3:** Calculate the molar mass of the compound, MM

- ◆ Molar mass of H<sub>3</sub>PO<sub>4</sub> is 98 g/mol

**Step 4:** Find the number of moles of solute present in the whole solution

$$\text{Mole} = \frac{\text{Mass of } H_3PO_4}{\text{Molar mass of } H_3PO_4} = \frac{4003.5}{98 \left( \frac{g}{mol} \right)} = 40.85 \text{ mol}$$

**Step 5:** Find the molarity of the solution

$$\text{Molarity of the solution} = \frac{\text{moles of solute}}{\text{Volume of the solution}(L)} = \frac{40.85}{1L} = 40.85 M$$

Now, let's see how to prepare 30% concentration of phosphoric acid solution:

- ◆ Volume of solution needed to prepare is 30.3 ml (Because, IR is set 2.75 and mass of treated flax straw is 11 g).

$$\text{Mass of } H_3PO_4 = \% H_3PO_4 \times \text{Mass of solution}$$

$$\text{Mass of } H_3PO_4 = 0.3 \times 4710 \text{ g} = 1413 \text{ g}$$

$$\text{Mole} = \frac{\text{mass of } H_3PO_4}{98 \left(\frac{\text{g}}{\text{mol}}\right)} = \frac{1413 \text{ g}}{98} = 14.418$$

$$\text{Molarity} = \frac{\text{Mole of solution}}{\text{Volume of solution}} = \frac{14.418}{1 \text{ L}} = 14.418 \text{ M}$$

- ◆ By using dilution formula:  $M_1V_1 = M_2V_2$

Where:

- ✓  $M_1$  is the molarity of 85% phosphoric acid,
- ✓  $V_1$  is the volume of 85% phosphoric acid required for dilution
- ✓  $M_2$  is the molarity of the required solution to be prepared and  $V_2$  is the volume needed to prepare.

Now, the volume of 85% phosphoric acid solution is calculated as:

$$V_1 = \frac{M_2}{M_1} \times V_2 = \frac{14.418}{40.852} \times 30.3 = 10.69 \text{ ml}$$

- Therefore, the amount of distilled water required to form 30.3 ml was found to be 19.61 ml.
- ❖ All other solutions of phosphoric acid with different concentration was calculated by this method described here above.

## Appendix B: Proximate analysis of flax straw and the prepared activated carbon

### Appendix B1: Raw material Characterization:

#### 1. Ash content

$$\text{Ash content} = \frac{(M_c + M_a) - M_c}{(M_c + M_s) - M_c} \times 100$$

Mass of empty dry crucible(g)	Mass of crucible plus mass of sample(g)	Mass of crucible plus sample after complete ashing(g)
40.000	43	40.1685

$$\text{Ash content} = \frac{(40.1685) - 40.000}{(43) - 40.000} \times 100 = 5.616\%$$

## 2. Moisture Content

The moisture content was determined in weight % according to Equation;

$$\text{Moisture content} = \frac{M_1 - M_2}{M_1} \times 100$$

Mass of empty dry crucible(g)	Mass of crucible plus mass of sample(g)	Mass of crucible plus sample after drying(g)
40	43	39.942

$$\text{Moisture content} = \frac{43 - 39.942}{43} \times 100 = 7.116\%$$

## 3. Volatile content

The volatile matter content (VM) was determined in % according to:

$$\text{Volatile matter} = \frac{100(B - C) - M_c(B - A)}{(B - A)(100 - M_c)} \times 100$$

Mass of empty dry crucible(g)	weight of crucible plus sample (g)	weight of crucible plus sample after heating (g)
40	43	40.837

$$\text{Volatile matter} = \frac{100(43 - 40.836) - 7.116(43 - 40)}{(43 - 40)(100 - 7.116)} \times 100 = 69.96\%$$

## 4. Fixed carbon content:

Fixed carbon is a calculated value and it is the resultant of summation of percentage moisture, ash, and volatile matter subtracted from 100.

$$\text{Fixed carbon}(\%) = 100 - (\text{Moisture, \%} - \text{Ash, \%} + \text{Volatile matter})$$

$$\text{Fixed carbon(\%)} = 100 - (7.116 + 5.616 + 69.96) = 17.31\%$$

## Product Characterization: Proximate Analysis

### 1. Ash Content:

The ash content is determined in % according to Equation:

$$\text{Ash content} = \frac{(M_c + M_a) - M_c}{(M_c - M_a) - M_c} \times 100$$

Mass of empty dry crucible(g)	Mass of crucible plus mass of sample(g)	Mass of crucible plus sample after complete ashing(g)			
		Trial 1	Trial 2	Trial 3	Average
40	45	40.352	40.425	40.128	40.302

$$\text{Ash content} = \frac{(40.302) - 40.000}{(45) - 40.000} \times 100 = 6.04\%$$

### 2. Moisture Content

The moisture content was determined in weight % according to Equation;

$$\text{Moisture content} = \frac{M_1 - M_2}{M_1} \times 100$$

Mass of empty dry crucible(g)	Mass of crucible plus mass of sample(g)	Mass of crucible plus sample after drying(g)			
		Trial 1	Trial 2	Trial 3	Average
40	45	41.262	41.946	40.986	41.398

$$\text{Moisture content} = \frac{45 - 41.398}{45} \times 100 = 8.004$$

### 3. Volatile content

The volatile matter content (VM) was determined in % according to:

$$\text{Volatile matter} = \frac{100(B - C) - M_c(B - A)}{(B - A)(100 - M_c)} \times 100$$

Mass of empty dry crucible(g)	weight of crucible plus sample (g)	weight of crucible plus sample after heating (g)
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		Trial 1	Trial 2		Average
40	45	43.262	44.436		43.849

$$\text{Volatile matter} = \frac{100(45 - 43.849) - 5.412(45 - 40)}{(45 - 40)(100 - 5.412)} \times 100 = 18.615\%$$

#### 4. Fixed carbon content:

Fixed carbon is a calculated value and it is the resultant of summation of percentage moisture, ash, and volatile matter subtracted from 100.

$$\text{Fixed carbon}(\%) = 100 - (\text{Moisture, \%} - \text{Ash, \%} + \text{Volatile matter})$$

$$\text{Fixed carbon}(\%) = 100 - (8.004 - 6.04 + 18.615) = 79.421\%$$

#### 5. Bulk Density determination:

- Mass of empty pycnometre (g),  $m_1 = 29.2074$
- Mass of pycnometre with water (g),  $m_2 = 79.3837$
- Mass of pycnometre with sample (g),  $m_3 = 32.2074$
- Mass of pycnometre containing water and sample(g),  $m_4 = 79.6303$

$$V_1 = \frac{[(79.3837 - 29.2074) - (79.6303 - 32.2074)]}{1 \text{ g/ml}} = 2.7534 \text{ ml}$$

$$\rho = \frac{32.2074 - 29.2074}{2.7534} = 1.0895 \text{ g/ml}$$

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## Appendix C: Experimental data for batch adsorption experiment

### Calculation C – 1: Preparation of Aqueous Solution

- The required chemical is Lead Nitrate,  $\text{Pb}(\text{NO}_3)_2$  of molecular weight 331.21 and distilled water.
- The lead (II) stock solution (500 mg/L) was prepared by dissolving 0.8 g of  $\text{Pb}(\text{NO}_3)_2$  (99.8%) with 500 ml distilled water.
- Standard working solution was prepared by serial dilution of the stock solution by using dilution formula:

$$\text{Dilution formula: } C_1V_1 = C_2V_2$$

Where:

- $C_1$  is the initial concentration,  $C_2$  is the final concentration,  $V_1$  is the initial volume, and  $V_2$  is the final volume.
- Now let's see how to prepare 40 ppm of working solution of Lead nitrate solution. The volume required is 100 ml working solution.
- Applying dilution formula:

$$V_1 = \frac{C_2}{C_1} \times V_2 = \frac{40}{80} \times 100 = 50 \text{ ml}$$

- Thus, 50 ml of distilled water is required to have 100 ml of 40 ppm lead nitrate working solution.
- All other concentration of the working solution was prepared by this method.

**Appendix D: The experimental result of flax straw activated carbon**

Run	Processing parameters			Response variables	
	Activation time (min.)	Activation temperature(°C)	Activating agent Concentration (%)	Yield (%)	Iodine number(mg/g)
1	30	500	70	32.2	289.7293
2	90	600	50	26.8	357.2933
3	60	400	30	39.3	328.5067
4	30	600	50	35.9	265.8533
5	60	500	50	53.3	460.5867
6	60	500	50	52.2	460.5867
7	30	400	50	32.6	318.3467
8	90	400	50	45.5	414.8667
9	60	500	50	52.2	436.88
10	90	500	30	41.5	425.0267
11	90	500	70	30.2	441.96
12	30	500	30	33.6	348.8267
13	60	500	50	52.2	460.5867
14	60	500	50	52.2	460.5867
15	60	600	30	29	311.5733
16	60	400	70	32.68	360.68
17	60	600	70	28.5	189.6533

**Appendix E: Experimental design matrix for flax straw AC for Pb<sup>+2</sup> removal efficiency**

Run	Factors				Absorbance	Slope	Volume	Ce	Co-Ce	Removal efficiency (%)
	PH	Adsorbent dose(g)	Adsorbate Concentration (ppm)	Contact time(min)						
1	8	2.5	80	65	0.488	0.0093	0.1	52.47	27.53	34.41
2	5.5	2.5	60	65	0.221	0.0093	0.1	23.76	36.24	60.39
3	3	2.5	80	65	0.315	0.0093	0.1	33.87	46.13	57.66
4	5.5	4	60	10	0.261	0.0093	0.1	28.06	31.94	53.23
5	8	4	60	65	0.231	0.0093	0.1	24.84	35.16	58.60
6	5.5	4	60	120	0.079	0.0093	0.1	8.49	51.51	85.84
7	5.5	1	60	120	0.226	0.0093	0.1	24.30	35.70	59.50
8	5.5	1	80	65	0.211	0.0093	0.1	22.69	57.31	71.64
9	8	2.5	60	10	0.381	0.0093	0.1	40.97	19.03	31.72
10	5.5	2.5	80	120	0.219	0.0093	0.1	23.55	56.45	70.56
11	8	1	60	65	0.348	0.0093	0.1	37.42	22.58	37.63
12	5.5	4	80	65	0.329	0.0093	0.1	35.38	44.62	55.78
13	5.5	2.5	80	10	0.499	0.0093	0.1	53.66	26.34	32.93
14	3	2.5	40	65	0.191	0.0093	0.1	20.54	19.46	48.66
15	5.5	4	40	65	0.018	0.0093	0.1	1.94	38.06	95.16
16	5.5	1	60	10	0.386	0.0093	0.1	41.51	18.49	30.82
17	8	2.5	60	120	0.313	0.0093	0.1	33.66	26.34	43.91
18	3	1	60	65	0.212	0.0093	0.1	22.80	37.20	62.01
19	8	2.5	40	65	0.253	0.0093	0.1	27.20	12.80	31.99
20	3	2.5	60	10	0.381	0.0093	0.1	40.97	19.03	31.72
21	5.5	2.5	40	120	0.177	0.0093	0.1	19.03	20.97	52.42
22	5.5	2.5	60	65	0.211	0.0093	0.1	22.69	37.31	62.19
23	5.5	2.5	60	65	0.201	0.0093	0.1	21.61	38.39	63.98
24	5.5	2.5	60	65	0.198	0.0093	0.1	21.29	38.71	64.52
25	5.5	1	40	65	0.265	0.0093	0.1	28.49	11.51	28.76
26	3	4	60	65	0.116	0.0093	0.1	12.47	47.53	79.21
27	5.5	2.5	40	10	0.251	0.0093	0.1	26.99	13.01	32.53
28	3	2.5	60	120	0.101	0.0093	0.1	10.86	49.14	81.90
29	5.5	2.5	60	65	0.209	0.0093	0.1	22.47	37.53	62.54

Appendix D: Some selected Laboratory setups and their photos

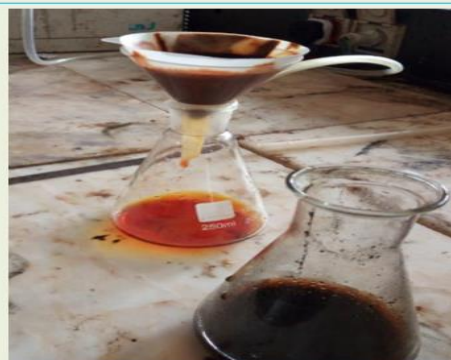


Raw material pretreatment for characterization and production of AC





Main steps of the preparation of activated carbon



Experimental setup during iodine number determination, AAiT, AAU-research Lab



Standard working solution



UV-Vis Spectrophotometer (Model-UVD-3200)



Laboratory samples before and after adsorption process



**Point of Zero Charge Determination for flax straw activated carbon**

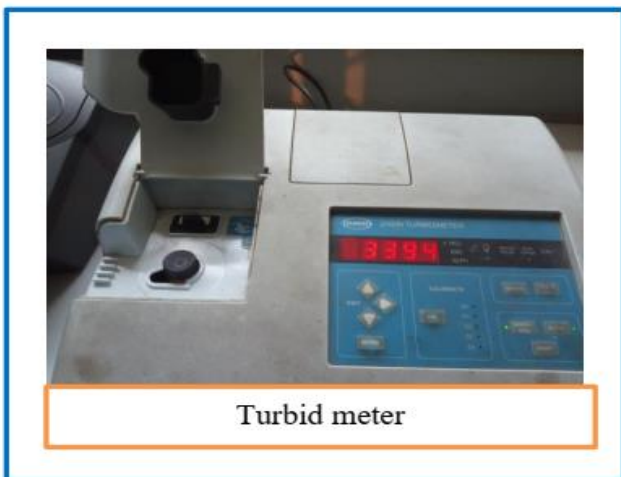


**Bulk Density measurement of activated carbon by pycnometer**

## Appendix E: Wastewater sample collection and treatment



Collected wastewater samples from KADISCO paint factory



Turbid meter



DO meter



Samples for BOD test

