Investigation of removal characteristics of heavy metal ions by natural adsorbents

by

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Dedicated to my honorable supervisor

Prof. Dr. Mohammad Abu Yousuf

Declaration

This is to certify that the thesis work entitled **"Investigation of removal characteristics of heavy metal ions by natural adsorbents"** has been carried out by Harabilash Golder in the Department of Chemistry, Khulna University of Engineering & Technology, Khulna, Bangladesh. The above thesis work or any part of this work has not been submitted anywhere for the award of any degree or diploma.

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This is to certify that the thesis work submitted by **Harabilash Golder** entitled **"Investigation of removal characteristics of heavy metal ions by natural adsorbents"** has been approved by the board of examiners for the partial fulfillment of the requirements for the degree of Masters of Philosophy in the Department of Chemistry, Khulna University of Engineering & Technology, Khulna, Bangladesh in December, 2011.

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Abstract

Rice husk, which is a relatively abundant and inexpensive material, is currently being used as the source of activated carbon an adsorbent for the removal of various pollutants from water. Activated carbon was prepared and characterized from rice husk. Rice husk was collected from local rice mill and used as raw material for producing activated carbon. Low-cost furnace was designed from locally available pottery and very slow burning of rice husk in presence of insufficient oxygen was applied for the preparation of activated carbon. The carbonized temperature and time was 350–550 $\circ$ C and about 3.0 hours respectively. The percentage of the yield in this method is about 25 (wt) %. Prepared activated carbon was characterized by a series of experiments such as, ash content, volatile content, moisture content, fixed carbon content, porosity and adsorption study. The prepared activated carbons contain 39.47% ash and 35.98% fixed carbon. SEM photographs exhibit that the prepared activated carbon possesses significant number of micropores, mesopores and macropores. XRD analysis provides information that the prepared samples are amorphous in nature. All these observed properties indicate that the prepared samples might be used as good adsorbent.

SEM photograph before and after adsorption clearly showed the evidence in favor adsorption. Prepared activated carbon used as potential adsorbent for methylene blue, Pb^{2+} and Hg^{2+} in aqueous solution. Methylene blue showed maximum adsorption at pH 6.0. With increasing the sizes of adsorbents the extent of adsorption decreased at two investigated temperatures, 30 °C and 40 °C. The equilibrium time for the adsorption was found to be about 180 minutes. Under all conditions amount of adsorbed increased with increasing the concentration of adsorbates. The experimental data have been found to be fit into Freundlich adsorption isotherm suggesting the chemisorption occurred.

Both Pb^{2+} and Hg^{2+} in aqueous solution showed maximum adsorption at pH 5.7 and 150 rev/min shaking frequency. The equilibrium time for the adsorption in both cases were found to be about 180 minutes under all conditions and the amount of adsorption increased with increasing the concentration of adsorbates. The experimental data have been found to be fit into Freundlich adsorption isotherm suggesting the chemisorption occurred.

Contents

1.1 Environment and its chemistry

Environmental chemistry is multidisciplinary science involving chemistry, physics, public health, sanitary engineering etc. In simple terms, it is the science of chemical phenomena in the environment. In broad terms, it is the study of the sources, reactions, transport and effect of chemical species in the air, water and soil and the effect of human activity upon these. Environmental education is important for the protection and conservation of environment and for the need to restrain human activities leading to indiscriminate release of pollutants into the environment. Recovering contaminated sites, treating industrial effluents and managing hazardous wastes are challenging topics in environmental science that demand a great deal of attention from chemists, biologists, and engineers. Therefore, laboratory introducing innovative aspects in environmental technology that is capable to solve the pollution of environment.

1.2 Environmental Pollution

Today, pollution is a prevalent phenomenon that is caused mostly by anthropogenenic activities. Pollution can be caused by the director indirect activity of human. Polluted water or land may he harmful to human health or the quality of aquatic ecosystems or terrestrial ecosystems directly depending on aquatic ecosystems, which result in damage to material property or interfere with amenities and other legitimate uses of the environment. Environmental pollution means any departure from a normal state of environment. Pollutant is a substance present in a nature in greater quantity than the natural substance due to human activity, which ultimately has a detrimental effect on the environment and therefore on living organism and mankind. The atmosphere, the hydrosphere, the lithosphere and the biosphere may be polluted by different reason. With increased population, ever growing urbanization and increased industrialization in urban areas the pollution problems began to have divesting effects on environment.

1.3 Pollutants:

Pollutants are usually those substances, which are added in the environment in amounts sufficient to cause harmful effects on the environment and therefore on human, other animals and vegetation. Pollutants like arsenic, lead, carbon monoxide, phenol etc. pollute the air, soil and water. A pollutant can cause a detrimental alteration of the physical, chemical or biological properties of air, soil and water. Thus, they can be classified as physical, chemical or biological pollutants:

a) **Physical pollutants:** color, odor, solids, temperature, the solubility of oxygen are the physical pollutants and these can affects the environment.

b) **Chemical pollutants:** They are classified into organic and inorganic:

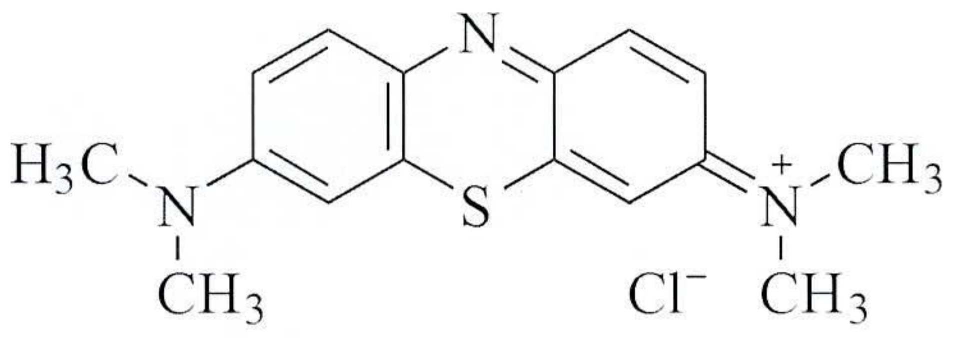
i) **Organic pollutants:** These are hydrocarbon and organic particulate matter such as, polycyclic aromatic hydrocarbons, domestic wastes (fats, oils, surfactants etc), agricultural wastes (pesticides, herbicides etc) and industrial wastes (phenols. oils, hydrocarbon etc).Contamination of ground and surface water by different organic pollutants is a major factor of environmental problems for the number of years. Among these organics pollutants, the phenolic compounds constitute eleven of one hundred and twenty six chemicals which have been designed as priority pollutants by the US environments.

ii) **Inorganic pollutants:** These are carbon monoxide (CO), nitrogen oxide (NO_x), sulphur oxides (SOx), inorganic salts, metallic salts (chlorides, sulfates, nitrates, etc.), heavy metals (Cu, Cd Cr, Hg Pb, As, etc.), mineral acids, finely divided metal or metallic compounds, trace elements, dissolved gases (methane, hydrogen sulfide, etc.), complexes of metals and organo-metallic compound. Some of them can be biodegraded by means of the microorganism present in the water. Some other has an extremely low biodegradability and is called refractory contaminants. They can remain in water for long periods in which its chemical action and/or toxicity may constitute a potential hazard.

c) **Biological pollutants:** the presence of determined microorganisms is necessary to complete the food chain. However, besides this group can be found pathogenic organisms, which can cause typhoid fever, dysentery, diarrhea and cholera.

1.4 Methylene blue as pollutant

Methylene blue is a heterocyclic aromatic chemical compound and IUPAC name is 3, 9–bis–dimethyl–aminophenazo–thionium chloride. It is a cationic dye. It is also called Swiss blue. Molecular formula and molecular mass of methylene blue are $C_{16}H_{18}N_3SCl.3H_2O$ and respectively. The structural formula is



Methylene blue is a dark green, combustible crystals or powder with bronze luster. It decomposes at 100–110 °C. It can react with oxidizing material. It is incompatible with alkalis, dichromates, alkali iodides and reducing agents. Heating produces toxic compounds of nitrogen and sulfur.

Methylene blue is a known animal mutagen. It is moderately to highly toxic by oral and intravenous routes. Eye contact can cause staining of the eye, stinging and lacrimation. Inhalations may cause dryness of mouth, flushed skin, rapid pulse, blurred vision, dizziness, hemolytic anemia, cyanosis and methaemaglobinaemia.

1.5 Lead as Pollutant

Lead (Pb) moves into and throughout ecosystems. The main sources of Pb entering an ecosystem are atmospheric Pb, paint, chips, used ammunition, fertilizers, pesticides, lead acid batteries or other industrial products, mining, plumbing, coal and gasoline. Pb deposited on the ground is transferred to the upper layers of the soil surface, where it maybe retained for many years. Atmospheric Pb in the soil will continue to move into the micro-organism and grazing food chains, until equilibrium is reached. Atmospheric Pb is deposited in vegetation, ground and water surface. The metal can affect all components of the environment and can move though the ecosystem until it reaches equilibrium. Pb accumulates in the environment but in certain chemical environments, it will be transformed in such a way as to increase its solubility. It slowing the rate of decomposition of matter plants, micro-organisms and invertebrates are affected by Pb. The addition of Pb to vegetation and animal surfaces can prevent the normal biochemical process. It may hinder the chemical breakdown of inorganic soil fragments and Pb in the soil may become more soluble, thus being more readily available to be taken up by plants.

Plants on land tend to absorb Pb from the soil and retain most of this in their roots. Pb pollution coats the surface of the leaf and reduces the amount of light reaching it. This results in stunting the growth and killing the plants by reducing the rate of photosynthesis, inhibiting respiration, encouraging an elongation of plant cells influencing root development. Pb affects the central nervous system of animals and inhibits their ability to synthesize red blood cells. Grazing animals are directly affected by the consumption of forage and feed contaminated by airborne Pb and somewhat indirectly by the up-take of Pb through plant roots. Pb shot and Pb weight can severely affect individual organisms and threaten ecosystems. Aquatic animals are affected by Pb at water connections lower than previously safe for wildlife.

The major biochemical effect of Pb is its interference with heme systhesis, which leads to hematological damage, Pb inhibits several of the key enzymes involved in the overall process of heme systesis whereby the metabolic intermediates accumulate. At higher levels of Pb in the blood (>0.8ppm) there will be symptoms of anaemia due to the deficiency of hemoglobin. Elevated Pb levels (>0.5-0.8ppm) in the blood cause kidney dysfunction and finally brain damage.

Lead is one of the metals to be removed, since it is harmful to central and peripheral nervous systems. It is generally known that adsorption with activated carbon is a good method to remove heavy metals. However, the high cost of activated carbon induces investigators to search for alternatives to replace it. Raw rice husk has been employed to remove heavy metals form aqueous solution.

1.6 Mercury as Pollutant

Mercury occurs uncombined in nature to a limited extent. It rarely occurs free in nature and is found mainly in cinnabar ore. Hg is an element that can be found naturally in the environment. It can be found in metal form, as Hg salts or as organic Hg compounds. Hg enters the environment as a result of normal breakdown of minerals in rocks and soil through exposure wind and water. Release of Hg from natural sources has remained fairly the same over the years. Still Hg concentrations in the environment are increasing; this is ascribed to human activities is released ito ari, through fossil fuel combustion mining, smelting and solid waste combustion. Some forms of human activity release mercury directly into soil or water, instance the application of agricultural fertilizers and industrial wastewater disposal. All Hg that is released the environment will eventually and up in soils or surface waters.

Hg is not naturally found in foodstuffs but in may turn up in food as it can be spread within food chains by smaller organisms that are consumed by humans, for instance through fish. Hg concentrations in the water they live in. Cattle breeding products can also contain eminent quantities of Hg. Hg is not commonly found in plant products, but it can enter human bodies through vegetables and other crops, when sprays that contain Hg are applied in agriculture. Hg from soils can accumulate in mushrooms. Acidic surface waters can contain significant amounts of Hg. When the pH values are between 5.0–7.0 the Hg concentrations in the water will increase due to mobilization in the ground. Once Hg is reached surface waters or soils, micro-organisms can convert it to methyl mercury, a substance that can be absorbed quickly by most organisms and is known to cause nerve damage. Fishes absorb a large amount of methyl mercury from surface waters every day. As a consequence, methyl mercury can accumulate in fish and in the food chins that they are part of. The effects that mercury has on animals are kidneys damage, stomach disruption, damage to intestines, reproductive failure and DNA alteration.

Hg^{2+} (mercury ion) however, is fairly toxic. Because of its high affinity for sulfur atoms, it easily attaches itself to the sulfur-containing amino acids of proteins. It also forms bonds with hemoglobin and serum albumin, both of which contain sulphydryl groups. Fish are the main source of food for many birds and other animals and Hg can seriously damage the health of these species. Because these predators rely on speed and co-ordination to obtain food, Hg may be particularly hazardous to these animals. Hg can cause harmful effects, such as nerve, brain and kidney damage lung irritation, eye irritation, skin rashes, vomiting and diarrhea. Hg has a number of effects on human that can all of them be simplified into main effects disruption of the nervous system, damage to brain functions, DNA damage and chromosomal damage, allergic reactions, resulting in skin rashes, tiredness and headaches, negative reproductive effects, such as sperm damage, birth defects and miscarriages.

1.7 Removal techniques of Pollutant

Various physical, chemical and biological pretreatment, main treatment and post treatment techniques can be employed to remove pollutants from water. Physicochemical techniques include membrane filtration, coagulation, flocculation, precipitation, flotation, adsorption, ion exchange, ion pair extraction, ultrasonic mineralization, electrolysis, advanced oxidation (chlorination, bleaching, ozonization, Fenton oxidation and photo catalytic oxidation) and chemical reduction. Biological techniques include bacterial and fungal biosorption and biodegradation in aerobic, anaerobic, anoxic or combined anaerobic/aerobic treatment processes. Among this adsorption is one of the well accepted and versatile techniques for the removal of dye and metal ions from aqueous system.

1.8 Low cost adsorbents

Adsorbents are used usually in the form of spherical pellets, rods, moldings, or monoliths with hydrodynamic diameters between 0.5 and 10 mm. They must have high abrasion resistance, high thermal stability and small pore diameters, which results in higher exposed surface area and hence high surface capacity for adsorption. The adsorbents must also have to have distinct porous structure which enables fast transport of the gaseous vapors.

Most industrial adsorbents fall into one of three classes:

i) Oxygen-containing compounds: These are typically hydrophilic and polar

ii) Carbon-based compounds: These are typically hydrophobic and non-polar; includes materials, such as, activated carbon and graphite.

iii) Polymer-based compounds: These are polar or non-polar functional groups in a porous polymer matrix.

Recently low cost adsorbents have been used for the removal of pollutants present in an industrial effluent such as heavy metal ion particles, chromium for tanks, industry effluents, dyes in textile industries effluents etc. Tea leaves, baggase, fish scales etc were used for above purpose.

1.9 Activated carbon as low–cost adsorbent

Activated carbon is a microcrystalline, non-graphitic from of carbon with a porous structure that has been processed to develop its internal porosity. This material can be characterized by a large specific surface area of 500-2500 m^2/g, the most important physical property of activated carbon which allows the physical adsorption of gases or vapors and dissolved or dispersed substances from liquids. Activated carbon is an effective adsorbent for many pollutant compounds (organic, inorganic and biological) of concern in water and wastewater treatment. The major use of activated carbon is in solution purification and for the removal of taste, color, odors and other objectionable and vegetable and animal oils. Activated carbon is a broad-spectrum agent that effectively removes toxic and bio refractive substances such as insecticides, herbicides, chlorinated hydrocarbons and phenols typically present in many water supplies.

Liquid-phase carbons, generally in the form of powdered or granular, are characterized as having larger pores (3 nm in diameter and larger) because of the need for rapid diffusion in the liquid decolorizing activated carbon are usually employed as powdered. Powdered activated carbon has an extremely high ratio of area to volume and since adsorption is a surface phenomenon, this increases its effectiveness but also makes it slow to settle and difficult to remove once added.

Any carbonaceous material (natural or synthetic) with high carbon content can be used as raw material for preparation of AC. The most common raw materials are agricultural byproducts such as wood, sawdust, rice husk, nut shell, fruit pits and charcoal, coconut shells, brown and bituminous coals, lignite, peat, bone, paper mill waste (lignin) and synthetic polymers like PVC are used for manufacturing of activated carbon. In fact, any carbonaceous low-cost materials (of animal, plant or mineral origin) with high carbon and low ash content can simply be changed into activated carbon under the thermal decomposition process. Activated carbon can also be used for removal of poisonous heavy metal ions from aqueous solutions.

Adsorption is due to the surface complex formation between the metal ions and the acidic surface function group of activated carbon. Removal efficiency is influenced by various factors such as, solution concentration, pH, ionic strength, nature adsorbate, adsorbent modification procedure, physical properties and chemical nature of the activated carbon. Removing of heavy metal ions adsorption using activated carbon can be increased by impregnation of activated carbon with suitable chemicals which is based on simple chemical reactions that are common in chemistry such as acid–base or neutralization, complex formation, redox, precipitation hydrolysis and catalytic reactions.

1.10 Adsorption

Adsorption is defined as the accumulation of a particular substance on interface. The species that is concentrated on the interface is called adsorbate and the material of the surface is called adsorbent. The reason of this accumulation is either a long range van der waals force of attraction or the electrostatic force of attraction or the unsaturation of the valence force on the surface i.e. the residual field of force. According to the nature of the force of attraction, two types of adsorption have been distinguished such as, (a) Physical adsorption and (b) Chemical adsorption. Chemisorption is classified into two groups as i) specific adsorption and ii) exchange adsorption.

The adsorbent may be solid or liquid. Surface of solid or liquid have certain properties that make them different from bulk. There is no chemical distinction between molecules or atoms on the surface and the molecules or atoms in the bulk. The molecules on the surface have unbalanced forces and they have tendency to satisfy this, whenever the opportunity comes. When this is accomplished, adsorption occurs. Adsorption takes place with a decrease in free energy and entropy. As the adsorption continuous, the adsorbed solute also tends to desorb. At one stage equal amount of solute is adsorbed and desorbed simultaneously. Eventually, the rates of adsorption and desorption will attain an equilibrium and the time at which such equilibrium is achieved is known as equilibrium time. Adsorption when involves the condensation of single layer of molecules on the surface is termed as monolayer and when involves the condensation of several layers is termed as multi-layer. The factors on which the extent of adsorption depends are:

(a) The pressure of adsorbate in case of gases.

(b) The nature of the active sites on the surface.

(c) The concentration of the liquid phase.

(d) The nature of adsorbent or adsorbate.

(e) The temperature of surroundings.

(f) pH of the liquid phase.

1.11 Adsorption on solid–liquid interface

There is a competition between the solute and solvent molecule for adsorption in the case of adsorption from solution. If the solute is adsorbed more than the solvent i.e., the concentration of solute is less in the bulk, the adsorption is termed as positive adsorption. But it is more in the bulk than in the interface, then negative adsorption results. If the solute is strong electrolyte, the cations and the anions are not likely to adsorb equally, particularly at low concentration in general, one of the ions, depending on the nature of the adsorbents is preferentially adsorbed and the surface will acquire electric charge accordingly.

The extent of adsorption is greatly influenced by the lyophobicity of compound. The higher is the solvophobicity greater is the adsorption and vice versa. Adsorption from solution is also influenced by the pH and the temperature of the solution. In case of adsorption from solution the effects of solvent on adsorption cannot be neglected especially, when the surface is polar in nature.

1.12 Adsorption equilibrium

The adsorbed solute tends to desorb into the solution during the adsorption from solution as the adsorption continues. At one stage equal amounts of solute is adsorbed and desorbed simultaneously. Eventually, the rates of adsorption and desorption attains an equilibrium. This is called adsorption equilibrium and the corresponding time at which such equilibrium achieved is called equilibrium time t_e. A number of mathematical expressions have been developed to give quantitative relationship between the amounts adsorbed with the equilibrium concentration of the solution.

$$x/m=f(c_e*T)$$

where, x/m = weight of adsorbate adsorbed per gram of adsorbent.

C_e= equilibrium concentration of the solution

T = absolute temperature (K)

1.13 Adsorption Isotherm

At constant temperature the plot of amount adsorbed against equilibrium concentration is called the adsorption isotherm i.e.,

$$x/m = f(C_e) \text{ at constant T}$$

Although a number of theoretical and empirical isotherm have been proposed by Langmuir, Freundlich, and BET isotherms are considered to be the most useful. Adsorption is usually characterized by isotherms, that is, the amount of adsorbate on the adsorbent as a function of its pressure (if gas) or concentration (if liquid) at constant temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials. The most important aspects to adsorption isotherm are:

a) The shape of isotherm.

b) The significance of the plateau found in many isotherms.

c) Whether the adsorption is monomolecular or extends over several layers.

d) The orientation of the adsorbed molecules.

e) The effect of temperature etc.

1.13.1 Langmuir isotherm

The Langmuir isotherm is the best known of all isotherms. In 1916 Langmuir derived an equation based on the adsorption of gases on a solid considering the following assumption

1. The surface is homogeneous. No chemical interaction takes places between the adsorbate and adsorbent. So that the chemical and physical behavior of the adsorbate remain unchanged.
2. Each site accommodate only one adsorbate and forms a layer of one molecule thickness i.e. monolayer adsorption.
3. The dynamic equilibrium exists between adsorption and desorption. For gas-solid adsorption the Langmuir equation can be written as:

   $V= V_m bp(1+bp)$ and

$V = bp/(1+bp)$

Where, V = amount adsorbed at any pressure

V_m = amount adsorbed for monolayer formation

$b = k_a/k_d$, b = equilibrium constant

K_a = rate of adsorption

K_d = rate of desorption

P = pressure of adsorbate

1.13.2 Freundlich isotherm

An empirical relationship was given by Freundlich when pressure is neither to high nor too low, then

$$V \infty p^{\frac{1}{n}}$$

$$V = kp^{\frac{1}{n}}$$

Where, V = amount adsorbed at any pressure,

P = pressure of adsorbate

The Freundlich equation can be written as

$$log\frac{x}{m} = logk + \frac{1}{n}logC_e$$

[x/m = weight of adsorbate adsorbed per gram of adsorbent and C_e= equilibrium concentration of the solution]

This equation shows the variation of adsorption with concentration at constant temperature. It has been found to be valid over a limited range of concentration. A plot of $log\frac{x}{m}$ vs $logC_e$ gives a straight line of slope $\frac{1}{n}$.

CHAPTER II

Literature Review

2.1 Literature Review

Major contaminants found in wastewater include biodegradable, volatile, and recalcitrant organic compounds; toxic metals; microbial pathogens; and parasites [1, 2] causing deterioration of the surrounding medium that can present a great danger to the environment and human health [3, 4]. Several studies have been undertaken on the toxicity of dyes and their impact on ecosystems [5, 6]. These studies showed that certain dyes degrade and that their derived products can be toxic and carcinogenic even at low concentrations [7]. Azo dyes are important colorants having extensive application in the leather, textile, paper mill, additive, foodstuff, and cosmetic industries. However, approximately (10 to 15) % of the overall production of azo dyes is released into the environment, which is very dangerous because some of the azo dyes and their degradation products have a toxic, carcinogenic influence on living organisms. Therefore, there is considerable need to treat all effluents prior to their discharge into receiving waters [3, 4].

Biological treatment has been shown to be very efficient for the decrease of biological and chemical oxygen demand (BOD and COD), but it is ineffective for the elimination of dyes from wastewater [7]. Several techniques such as ultrafiltration, [8] catalytic photodegradation, [9, 10] oxidation by UV/H_2O_2, [11, 12] direct precipitation and separation of the pollutants by membrane processes, [13] and adsorption methods [7] have been studied. Among them, adsorption processes have been reported as a low-cost technique for the treatment of textile industry effluents and pigments. The most common adsorbents used for dye removal from wastewater are activated carbons [14]. Natural materials have been investigated as industrial adsorption media [11, 12, 15-17]. Regarding the adsorption of dyes using clay minerals, bentonites, montmorillonites, and sepiolites have been described as adsorbents [18–23].

The first study about the adsorption behavior of a cationic dye on clay particles was reported by Bergman and O'Konski [24]. The authors observed the presence of four distinct bands near (575, 610, 670, and 760) nm, using visible spectroscopy analysis, which were related to dye aggregates. Many other studies have been reported on dye-clay systems, relating the adsorption bands to several species. Yariv and Lurie [25] assigned the bands at (575 and 610) nm to the interaction between the ð-system of the dye and the lone pair of electrons of the oxygen atoms on the clay surface. Cenens and Schoonheydt [26] proposed that the band near 575 nm can be related to trimers on the external surface and that the band at 610 nm corresponded to dimers at the outer and inner surfaces of the clay. The investigators confirmed that the band at 670 nm was due to the adsorption of monomers and showed that the band at 765 nm is due to the protonated Methylene Blue (MB) adsorbed on the clay. Many of the aggregation and adsorption properties of clays can be related to the layer charge density [27]. Bujdak and Komadel [28] studied the interaction between modified clays with various layer charges and MB in aqueous suspensions. The authors concluded that the strong aggregation and redistribution of the molecules in the negative sites of clay surfaces suggests that the dye is adsorbed initially only on a small fraction of the clay particles, followed by a redistribution with time.

Recently, the use of cheap agricultural wastes such as rice straw [29], bark [30], Japanese green tea [31], wool, and coconut husks have been highlighted as potential adsorbents for metal removal from wastewater. Minamisawa *et al.* [32, 33] have used chitin and chitosan for the adsorption of metals such as Cu, Co, Au, and Mn ions. Chitin is universally present in the exoskeletons of arthropods and manufactured in large scale from crab and shrimp shell wastes. Chitin and chitosan are nontoxic and readily biodegradable and hence are environmentally acceptable. Y. Orhan and Buyukbungor [34] and G. Macchi [35] have reported that Turkish coffee, used coffee after extraction of coffee brew, and nut and walnut shells were useful for heavy metal removal. However, these agricultural materials have to be chemically treated prior to use as adsorbents, which proves costly. In a previous paper [36], we demonstrated that Cu(II) and Cd(II) were almost completely removed from aqueous solution by use of roasted coffee beans that had been pretreated simply by washing with water and drying. Thus, roasted coffee beans offer potential as a new low-cost material for the removal of heavy metals. For example, in Japan, coffee beans in the form of green beans are consumed at the rate of about 39000 tons/year, and the demand for coffee surpasses that of citrus fruit or Japanese tea. The used coffee grounds, green tea, tea, and coarse tea left after preparation of these beverages is almost completely disposed of as waste. Porous carbons have been widely used for gas separation, gas storage, catalysis, solvent recovery, and removal of pollutants. New activated carbons such as an activated carbon fiber [37] and a super-high-surface-area carbon [38] have considerably uniform slit-shaped micropores compared with conventional activated carbons [39, 40]. The pore walls are composed of basal planes of micrographites, and spaces between these micrographites provide micropores.

Many techniques have been proposed for the treatment of wastewater containing mercury. In the last years, there has been an increasing interest to develop new adsorbents for Hg, including resins [41] clays [42] and carbon materials obtained from nonconventional sources (biomass, agricultural products, and the like) [43-47]. A quite promising alternative for selective adsorption is the use of specific chelating organic groups that can be anchored to the surface of a solid support. Because mercury species show a high affinity toward sulfur, several thio-organic groups have been used for improving the adsorption efficiencies of different materials such as activated charcoal [45-47], clays [48, 49], or silica [50, 51]. Frequently, however, not all of the functional groups that are incorporated into the material are accessible to the mercury species because such materials present small and irregular pore sizes [48]. Meso structured silica materials obtained by surfactant assembly methods present high superficial areas and large uniform pores [52-55]. Therefore, they can be suitable supports for adsorbent design. For this potential application, hybrid organic-inorganic silicas have been developed by anchoring organic moieties to the mesoporous silica surface, allowing its use in specific adsorption [56]. For mercury treatment, these materials have been superficially modified by incorporating an organic monolayer that contains thiol and amine groups as specific sites of mercury anchorage [57-63].

Lead ions commonly exist in industrial and agricultural wastewater [64] and acidic leachate from landfill sites in relatively high concentration. They are quite harmful to human and living things, leading to a wide range of spectrum health problems, such as nausea, convulsions, coma, renal failure, cancer, and subtle effects on metabolism and intelligence [65]. Due to the importance of lead as a heavy metal ion contaminant in geochemical systems and its high toxicity, many techniques have been applied in the removal of Pb(II), such as flocculation [66], membrane filtration [67], solvent extraction [68], biosorption [69], chemical precipitation [70], reverse osmosis [71], adsorption [72], etc. Among these technologies, the adsorption method is considered to be highly effective and economical at present. In addition, it is an important method to understand the accumulation of metal ions at solid–liquid interfaces [73]. Carbon nanotubes [65], activated carbon [72], clay minerals [73, 74], microorganisms [75], plant wastes [69, 76], and industrial and agricultural byproducts [77] have been widely investigated as such adsorbents. Normally, adsorption is strongly dependent on the pore structure and surface area of the adsorbents [78], whereas metal ion uptake is largely ascribed to ion exchange or chemical adsorption on specific adsorption sites. At low concentrations, metal ion adsorption on solids is mainly a surface coordination process, which can be modeled thermodynamically as a complexation reaction between surface sites and metal ions. Thus, modification of the surface chemistry strongly influences the metal ion adsorption process [79]. As well-known notorious metal to the environment, Pb(II) has been adsropted onto γ-Al_2O_3 was determined using batch adsorption experiments over a pH range from 4 to 10 [80]. The adsorption of Pb(II), onto alumina, silica, and kaolinite, in the presence of salicylic acid also have been investigated [81–83].

Activated carbons can be widely used as industrial adsorbents for separation, purification, and recovery processes due to their highly porous texture and large adsorption capacity [84–86]. They are also used as catalyst supports, chromatography columns and electrode materials for batteries and capacitors [87–92]. Rice husk, a by-product of the rice milling industry, accounts for about 20% of the whole rice grain. The amount of rice husk was approximately 500 million tons in developing countries (Food and Agriculture Organization, 1995). Only 100 million tons were available annually for utilization. The amount of rice husk available is far in excess of any local uses and thus has posed disposal problems. Despite the increasing trend of the rice husk surplus, proper methods of disposal and utilization of rice husk have yet to be developed because of its high ash content [93]. Many papers have been published on this subject [94–97]. At the same time, coconut shell is one of the source of activated as mentioned by many workers [99–100]. The carbons have a large Brunauer–Emmett–Teller (BET) surface area. In addition, experimental results revealed that the BET surface area, pore volume, and average pore diameter of the resulting porous carbon generally increase with increasing activation time, activation temperature and ratio of activation agents.

In the present research, low cost adsorbent will be prepared from rice husk for the removal of methylene blue, Hg^{2+} and Pb^{2+} from aqueous solution. The influence of adsorbent loading and contact time will be considered. Kinetics of adsorption of methylene blue, Hg^{2+} and Pb^{2+} on activated carbon and their corresponding isotherms will also be studied.

2.2 Objectives

Various methods are currently used for removal of dyes and heavy metals from aquatic media including precipitation, coagulation, filtration, flotation, sedimentation, membrane process, electrochemical technique, ion-exchange and adsorption on solids. Among these techniques, the adsorption is the most easiest and efficient methods of removal these pollutants. Activated carbon having a very high surface area is widely used as an adsorbent in different industries including textile, knitting and dyeing factories as well as for removal of toxic metals. But preparation of activated carbon of high surface area from easy obtainable sources is very domineering from technological and cost-effective point of view. In this research work, preparation of activated carbon of high surface area and adsorption of methylene blue, Hg^{2+} and Pb^{2+} has been studied on with activated carbon the following aims:

- To prepare activated carbon from rice-husk in a low cost method.
- To characterize the locally produced activated carbon
- To determine the removal efficiency of heavy metal by the activated carbon.
- To determine the effectiveness of the produced activated carbon for removal of methylene blue
- To determine the effectiveness of the produced activated carbon for removal of methylene blue, Hg^{2+} and Pb^{2+} ions in aqueous system
- To study the kinetics of adsorption of methylene blue, Hg^{2+} and Pb^{2+} ions on activated carbon
- To study the adsorption isotherms
- To determine the effect of pH on adsorption
- To investigate the effect of temperature on adsorption.

CHAPTER III

3.1 Apparatus and Chemicals

3.1.1 Apparatus

1. A double beam UV–visible spectrophotometer (UV–160A, Shimadzu, Japan)
2. pH meter (HANNA instrument, Taiwan)
3. Electronic balance (OHAUS Balance- AR-2130 USA)
4. Electric Oven (DIN-12880 KI West Germany)
5. Electric shaker (GEMMY Orbit Shaker, Model: VRN-480)
6. Laboratory test sieve (MIC-200)
7. Muffle furnace (Lenton, AKF 13/5-2031 England)
8. Scanning electron microscope (HITACHI H-800EM).

3.1.2 Reagents

1. HCl (E–Merck, Germany)
2. Hexamine (E–Merck, Germany)
3. Double distilled water (DDW)
4. $(NH_4)OH$ solution (E–Merck, Germany)
5. Xylenol Orange tetrasodium salt (E–Merck, Germany)
6. Potassium nitrate (E–Merck, Germany)
7. EDTA (E–Merck, Germany)

3.1.3 Adsorbates

Methylene blue (AG–Merck, Germany), Lead (Pb^{2+}) ion (aqueous) and Mercury (Hg^{2+}) ion (aqueous).

3.1.4 Adsorbent

Prepared Activated Carbon (AC) from rice husk

3.2. Preparation of Activated Carbon (adsorbent) from rice husk

Dried and fresh rice husks were used as raw materials to produce the adsorbents. Rice husk was collected from local rice-mill. They were separately washed with hot tap water to remove dust like impurities and dried in sun. The porous carbons were prepared from rice husk by using a locally designed furnace as shown in Figure 2.1. 2.0 kg dried rice husks

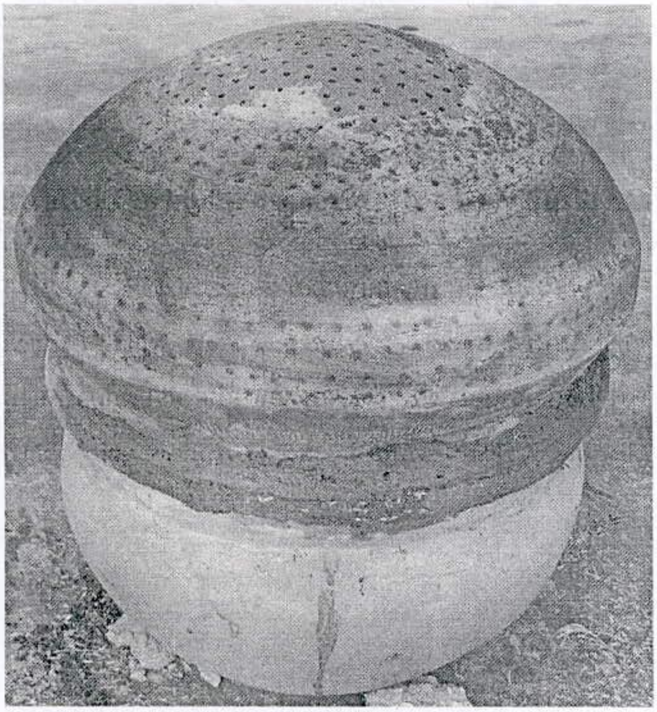


Figure 2.1: Photograph of locally designed furnace

were taken in the furnace carbonized at 350–550 ◦C in the presence of insufficient oxygen for about 3.0 hours. At end of combustion black colored activated product was collected from the bottom of the furnace. Then it was cooled to room temperature in desiccators. Then it was ground by a mortar and sieved. The prepared sample was placed in a glass screw-cap bottle and stored at room temperature. About 400 g AC was obtained from one batch combustion and was kept for the next adsorption experiment.

3.3 Characterization of activated carbon

3.3.1 The morphology and pore diameter of the samples were determined by scanning electron microscope (HITACHI H-800EM).

3.3.2 Moisture Content Determination

2.0 g activated carbon was taken in a crucible with lid. Then it was placed in a preheated ovenat 110 ^oC for 12 hours. Then it was transferred to a desiccator and was cooled to room temperature. The moisture content determined from:

$$\text{Moisture content} = [(A-B)/A]*100$$

Where,

A = Weight of the sample before heating

B = Weight of the sample after heating

3.3.3 Ash Content Determination

2.0 g activated carbon was taken in porcelain crucible and then heated the sample at about 750 ^oC for about one hour in a muffle furnace. The crucible and its content was retrieved and cooled to room temperature in a desiccator. The ash content was determined from:

$$\text{Ash content} = (B/A)*100$$

Where,

A = Weight of the sample before heating

B = Weight of the sample after heating

3.3.4 Determination of silica content

2.0 g of prepared AC was taken in a porcelain crucible with lid and was heated at 800 ^oC for about 5 hours for the silica and carbonaceous material content. The residue and the mass losses were considered as silica and carbonaceous materials respectively. The silica content was determined from:

$$\text{Silica content} = (B/A)*100$$

Where,

A = Weight of the sample before heating

B = Weight of the sample after heating

3.3.5 Volatile content determination

2.0 g activated carbon was taken in closed porcelain crucible and then the sample was heated at about 550 ^{o}C for 10 minutes in a muffle furnace. The crucible and its content was retrieved and cooled in a desiccator. The volatile content was determined from:

$$\text{Volatile content} = [(A-B)/A]*100$$

Where,

A = Weight of the sample before heating

B = Weight of the sample after heating

3.3.6 Fixed carbon determination of AC

The Fixed Carbon content (FC) is determined as:

$$\text{Fixed carbon content} = 100 - VC - U - Ash$$

Where,

VC = Volatile content (%)

U = Moisture content (%)

Ash = Ash content (%)

3.4 Preparation of stock solution of Adsorbates

3.4.1 Preparation of stock solution of methylene blue:

0.1598 g of methylene blue was taken in a 100 mL volumetric flask and it was dissolved in double distilled water to make 5×10^{-3} M stock solution. The volume was increased up to the mark by DDW. Further dilution was made whenever it is necessary. The concentration range used for experiments was 1.0×10^{-5} M to 4.0×10^{-5} M.

3.4.2 Preparation of stock solution of Pb^{2+} ion

331.20 is the molecular mass of lead nitrate. 8.28 g of salt was taken in 500 ml volumetric flask and it was dissolved in double distilled water to make 0.05 M solution. The volume was increased up to the mark by DDW. Then it was diluted to prepare 0.02 M, 0.01 M, 0.006 M and 0.003 M solution.

3.4.3 Preparation of stock solution of Hg^{2+} ion

271.59 is the molecular mass of mercuric chloride. 6.7898 g of salt was taken in 500 ml volumetric flask and it was dissolved in double distilled water to make 0.05 M solution. The volume was increased up to the mark by DDW. Then it was diluted to prepare 0.02 M, 0.01 M, 0.006 M and 0.003 M solution.

3.5 Absorption spectrum of stock solution

The absorption spectra of methylene blue solution at pH 6.0 under different concentration showed that the λ_{max} is independent of concentration and it was found to be 663.0 nm. Absorption spectrum was recorded by UV-160 spectrophotometer at 30 °C, using DDW as reference solution.

3.6 Determination of molar extinction coefficient

The calibration curve was constructed by plotting the absorbance of solutions versus the different concentrations of methylene blue within the range of 1.0×10^{-5} M to 4.0×10^{-5} M. The experiment data and corresponding standard curves have been shown in result and discussion section. From this calibration curves the molar absorption coefficient was calculated.

3.7 Adsorption experiments

3.7.1 Estimation of the equilibrium time for methylene blue adsorption

The equilibrium time of the adsorption of methylene blue on prepared AC obtained from rice husks was estimated at pH 6.0 and 10.0 respectively. 0.1 g of prepared AC and 25 mL of 3.0×10-5 M and 40 mL of methylene blue solution where used in each of the 12 bottles. For blank 0.1 g of prepared silica and 25.0 mL of DDW solution added in a bottle and after shaking for 5 hours at 30 °C. The bottles were shaken in a thermostatic mechanical shaker at 30 °C. After a definite interval of time each bottle was withdraw from the shaker. The supernatant of the bottle was transferred and centrifuge repeatedly until a clear liquid was obtained. The absorbance of the clear solution was measured spectrophotometrically at λ_{max} 663.0 nm. The amount adsorbed (x/m), mg/g versus the time of adsorption was plotted.

3.7.2 Adsorption isotherm for methylene blue

Adsorption experiments were carried out on AC obtained from rice husks using different initial concentrations of methylene blue from 0.5×10^{-5} to 5.0×10^{-5} M at pH 6.0. 40 mL of each solution was taken in 6 bottles. The bottles were shaken in thermostatic mechanical shaker at 30 °C for 5 hours. The bottles were withdrawn at the stipulated time from the shaker and supernatant was transferred to the centrifuge tube for centrifugation. The absorbance of the clear solution was measured spectrophotomtrically.

3.7.3 Adsorption Experiments of Lead and Mercury

Adsorption experiments for lead were carried out in 850 mL high density polyethylene bottle employed in an orbital shaking shaker (GEMMY Orbit Shaker, Model: VRN-480). The adsorption bottles were filled with aqueous solution of lead of known concentration. The predetermined amounts of activated carbon were dropped into the bottles. Total volumes of the solutions were made 850 mL. pH of the solution was maintained by adding HCl or NH_4OH. Then the adsorption processes were performed at predefined shaking rate for anticipated time.

After a certain time, the adsorption was stopped and the slurry was filtered through a piece of ash less filter paper. The lead concentration in the filtrate was measured. The equilibrium time for adsorption, adsorption capacity and adsorption isotherm were determined by using standard and conventional methods.

CHAPTER IV

Result and Discussion

4.1 General

Efforts have been made to burn rice husk and coconut shell for the production of activated carbon as adsorption material under controlled temperature. Keeping the growing concern with environment pollution in vision, and the need to conserve energy and resources, the amount of rice husk available is far in excess of any local uses and, thus, has posed disposal problems. Rice husk and coconut shell were chosen to be applied as a precursor material, because of their granular structure, insolubility in water, chemical stability, high mechanical strength, and its local availability at almost no cost. The advantages in the application of rice husk, coconut shell and the produced activated carbon as adsorbents is that there is no need to regenerate them, because of their low production costs. The utilization of rice husk and coconut shell would solve both a disposal problem and also access to less-expensive material in the waste water treatment are of the subject of discussion of this chapter.

4.2 Properties of rice husk and activated carbon prepared from rice husk

Rice husk as shown in Figure 4.2.1 possesses a granular structure, is insoluble in water, and has

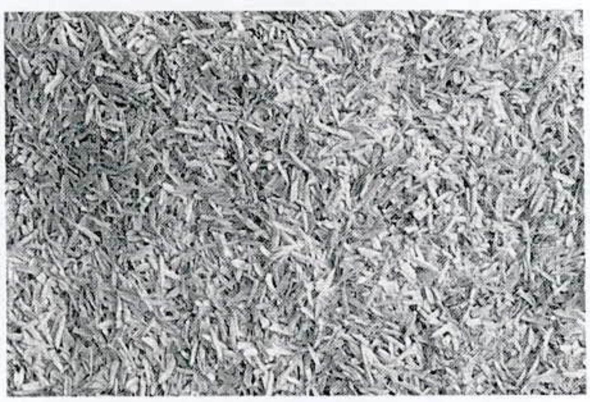


Figure 4.2.1: photograph of rice husk sample

chemical stability and high mechanical strength, making it a good adsorbent material for treating various wastes from water and wastewater. Tables 4.2.1 and 4.2.2 show the physico-chemical properties and chemical compositions of five different rice husk samples, respectively [101–102, 112–114]. It has been documented the morphology of rice husk may facilitate the adsorption of metals and other pollutants, because of the irregular surface of rice husk [112–114].

Table-4.2.1: Proximate and Ultimate Analyses of Rice Husk

Proximate Analysis (wt %)					Ultimate Analysis (wt %)					
Sample Number	**moisture**	**ash**	**Volatile matter**	**fixed carbon**	**C**	**H**	**O**	**N**	**S**	**Cl**
1	10.00	17.13	---	---	38.92	5.55	37.94	0.35	0.02	0.09
2	---	19.8	64.3	15.9	37.00	5.10	36.00	0.40	---	---
3	7.9	17.1	59.9	---	44.6	5.6	49.3	---	---	---
4	---	20.00	66.40	13.60	37.8	5.20	39.0	0.39	0.05	
5	---	17.9	72.8	9.3	48.9	6.2	44.1	0.8	---	---

The physical characterizations of rice husk have pointed out some properties such as the presence of functional groups (carboxyl, silanol, etc.) that make adsorption processes possible. As mentioned in Table 4.2.2, rice husk consists of cellulose, hemicelluloses, lignin, and

Table 4.2.2: Chemical Analysis of the Rice Husk

Composition	(%)				
/ Sample No	1	2	3	4	5
cellulose	32.24	34.4	29.20	32.24	33.47
hemicellulose	21.34	29.3	20.10	21.34	21.03
lignin	21.44	19.2	30.70	21.44	26.70
extractives	1.82			1.82	
water	8.11			8.11	
mineral ash	15.05	17.1		15.05	

mineral ash, and there is a high percentage of carbon in its mineral ash. The composition and properties of carbonaceous materials of rice husk or rice husk ash are shown in Tables 4.2.3. The chemical components of rice husk ash are found to be accompanied by some common ingredients such as, SiO_2, H_2O, Al_2O_3, Fe_2O_3, K_2O, Na_2O, CaO, and MgO. It has also been established that the presence of these oxide may increase the adsorption capacity to significant level [103–108]. At the same time it is seen from ultimate analysis of Table 4.2.1 rice husk contain 37–48.9% carbon which is the target material in our present research. Rice husk is a relatively abundant and inexpensive material, is currently being used as a source of activated carbon for the removal of methylene blue and some heavy metals.

Table 4.2.3: Chemical Analysis of the Rice Husk ash

Constituents	Values
SiO_2 (%)	92.00
Al_2O_3 (%)	0.29
Fe_2O_3(%)	0.10
CaO (%)	1.28
MgO (%)	0.37
Na_2O (%)	0.05
K_2O (%)	2.19
SO_3 (%)	0.94
Loss on Ignition (%)	3.43

Combustion processes always play the important role for making activated carbon. Because pretreatment of the rice husk can remove lignin and hemicellulose, reduce cellulose crystallinity, and increase the porosity or surface area [103−106]. Chemically modified or treated rice husk exhibited huge changes in its composition as well as adsorption properties. In this research, rice husk was collected from the local rice mill. Then it was washed and dried and combusted without any pretreatment in a self-designed furnace. Locally made potteries were used for making furnace. Samples are kept in a cup type pottery or receiver and covered by a sifter. To control the combustion process or to maximize the carbonization process some of the holes of the sifter were sealed by mud. The joining part of receiver and sifter are connected together by mud so the air cannot enter into the furnace frequently. Before connecting receiver and sifter rice husk samples were kept inside and it was fired. Activated carbon produced from rice husk is the combustion product of rice husk in other word; it may be termed as rice husk ash. 2.0 Kg rice husk samples were carbonized at 400 ^{o}C in the furnace shown in Figure 2.1. Thus, it is assumed that the optimum time for carbonization is 1 hour 40 minutes in order to eliminate most of the volatile organic constituents, leaving behind carbonaceous materials which were considered as activated carbon. The percentage of the yield is about 25 (wt) %.

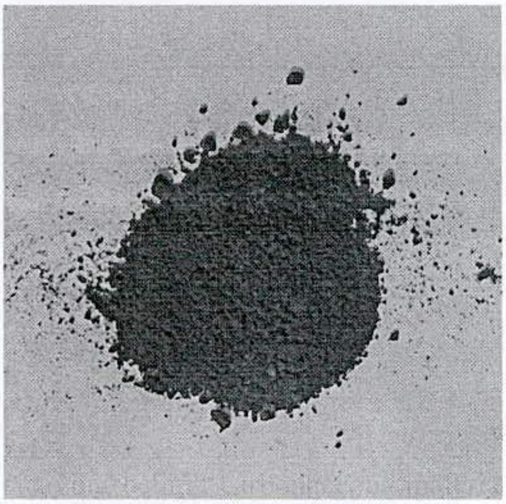


Figure 4.2.2: Schematic photograph of Activated Carbon obtained from rice husk sample

Activated carbons, the most widely used adsorbent are carbons which generally have a high surface area and complex pore structure and high adsorption capacity and high degree of surface reactivity resulting from carbonization processes. Figure 4.2.2 shows the activated carbon

Table 4.2.4: Physicochemical characteristics of Activated Carbon

Characteristics	Values
Bulk density (g/mL)	0.76
Solid density (g/mL)	1.49
Moisture content (%)	9.93
Ash Content (%)	39.47
Volatile content (%)	8.35
Fixed carbon content(%)	35.98
Particle size (mesh)	≤200
Surface area (m^2/g)	222.23
pore diameter (μm)	0<pore diameter≥8

prepared from rice husk. The physico-chemical properties of prepared activated carbon are shown in Table 4.2.4. These are black colored amorphous solid particles with abundant porosity. Figure 4.2.3 shows the SEM photograph of activated carbon before adsorption. In the photograph pores with different diameters are remarkable. The structure of an activated carbon is

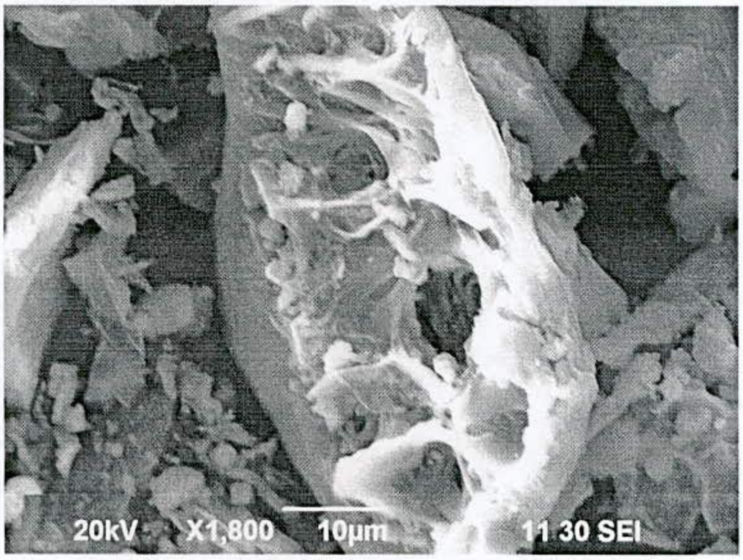


Figure 4.2.3: SEM photograph of Activated Carbon obtained from rice husk sample before adsorption.

composed of pores classified into three groups, namely micropores, mesopores and macropores. Micropores usually account for over 95% of the total surface area of activated carbons. The dimensions of the pores range are$0<$pore diameter≥ 8 μm. Conventional activated carbons are

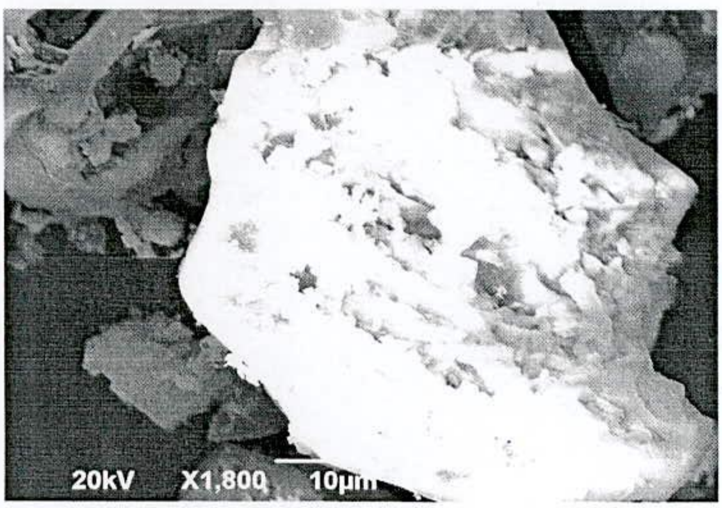


Figure 4.2.4: SEM photograph of Activated Carbon obtained from rice husk sample after adsorption.

tridisperse, having all three types of pores present within their structure is also evident from our investigation shown in SEM photograph. Adsorbate molecules penetrate through the wider pores and into the micropore structure [111]. The particles are sieved with 200 mesh sieve and are allowed for adsorption experiments. An evident result was obtained from SEM photograph after adsorption that almost all the pores were blocked due to the adsorption of metal ions such as Pb^{2+} or Hg^{2+} as shown by Figure 4.2.4.

The phase characterization or amorphous nature of the Activated Carbon was carried out using an X-ray diffractometer and the XRD pattern is shown in Figure 4.2.4. The XRD of Activated Carbon after thermal degradation in nitrogen or in an air medium was carried out. Copper was used as the target and nickel as the filter medium for the XRD. A quartz standard slide was run to check the instrument drift and obtain the accurate location of 2θpeaks. Broad, diffused peaks of the sample were observed, and these indicate the amorphous nature of the Activated Carbon. At the angles $2\theta = 20.5°$ and $2\theta = 23.7°$, the peaks indicate the presence of an amorphous form of carbon with minor components such as quartz, CaO and alumina, while the major components are silicates, and silicon oxide. This result correlates the chemical analysis report of rice husk ash as shown by earlier researchers in Table 4.2.3. Any sharp diffraction peak of crystalline carbon was observed. It was seen by the X-ray diffractrograms that the activated carbon has a heterogeneous surface.

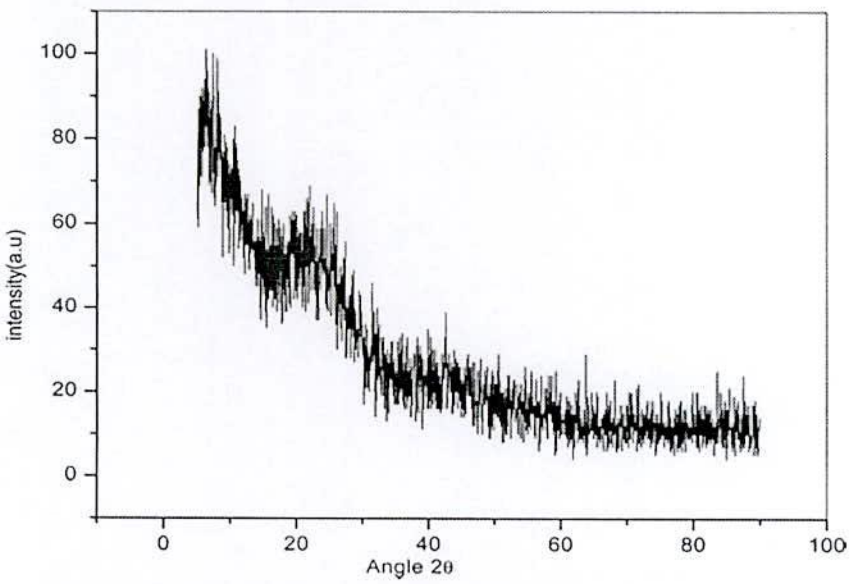


Figure 4.2.5: The XRD report of prepared Activated Carbon obtained from rice husk sample

4.3 Adsorption study of methylene blue on prepared activated carbon

Dyes and pigments are discharged into the water bodies from various industrial sources, mainly from the dye manufacturing and textile finishing industries. There are several methods available for the treatment of dye-containing wastewater. Among them, the adsorption process provides an attractive alternative for the treatment of wastewater, especially if the adsorbent is inexpensive and does not require additional pretreatment before its application. The adsorption characteristics of methylene blue onto prepared activated carbon from rice husk are discussed. Batch adsorption experiments were carried out under varying experimental conditions such as, particle size of adsorbent, contact time, initial concentration of MB and temperature of the solution.

4.3.1 Determination of Absorbance maxima (λ_{max}) of methylene blue:

Absorbance maxima (λ_{max}) of methylene blue at pH 6.0 were determined spectrophotomtrically from absorption spectrum. The maximum absorbance of methylene blue solution was found to be 663.0 nm and was found to be independent of different concentrations of methylene blue solution. The curves of λ_{max} are shown in the Figure 4.3.1.

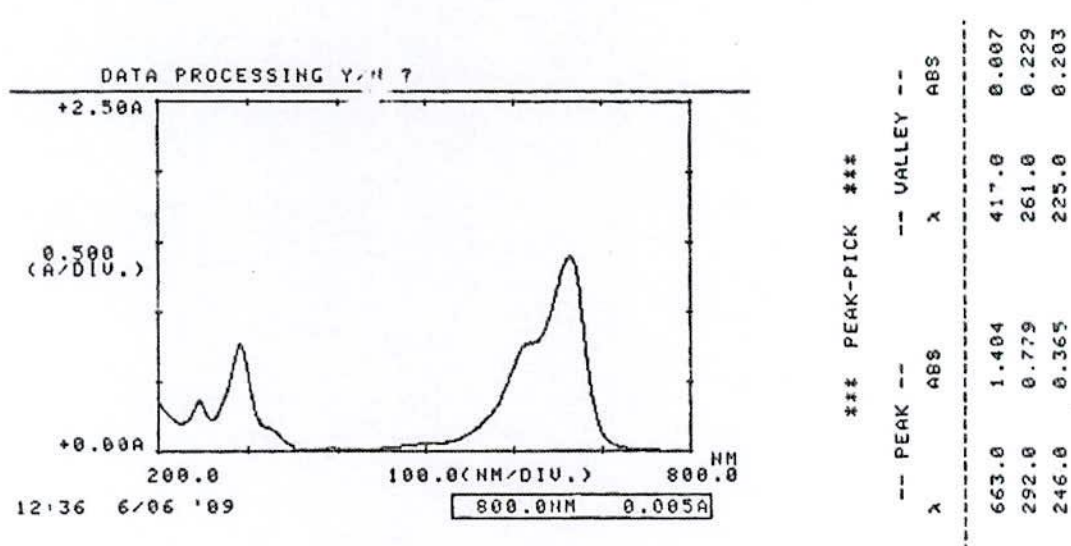


Figure 4.3.1: Absorbance maxima (λ_{max}) of methylene blue

4.3.2 Construction of a calibration curve and determination of molar absorption co-efficient of methylene blue

Using this λ_{max}, a calibration curve was constructed by maintaining the following conditions. The absorption spectrum of methylene blue solution was recorded and the value of absorbance maxima was found to be 663.0 nm. A calibration curve was prepared by plotting absorbance against different concentration of methylene blue solutions at pH 6.0. The straight line of the absorbance *vs.* concentration of methylene blue passing through the origin in Figure 4.3.2 suggested the validity of Beer-Lambert law. From the slope, the value of molar absorption co-efficient (ε) of methylene blue was estimated as 5.06×10^4 L $mol^{-1}cm^{-1}$. The unknown concentration of methylene blue solution was estimated using the value of molar absorption co-efficient (ε).

Experimental conditions:

Sample	: Methylene blue solution
Reference	: DDW
pH of the solution	: 6.0
Temperature	: 30 °C (±0.5 °C)
λ_{max} of methylene blue solution	: 663.0 nm

Table 4.3.1: Absorbance of methylene blue solution at different concentration at pH 6.0

No. of observations	Concentration × 10^5/M	Absorbance
1	1.0	0.522
2	2.0	1.100
3	2.5	1.318
4	3.0	1.593
5	3.5	1.808
6	4.0	1.990

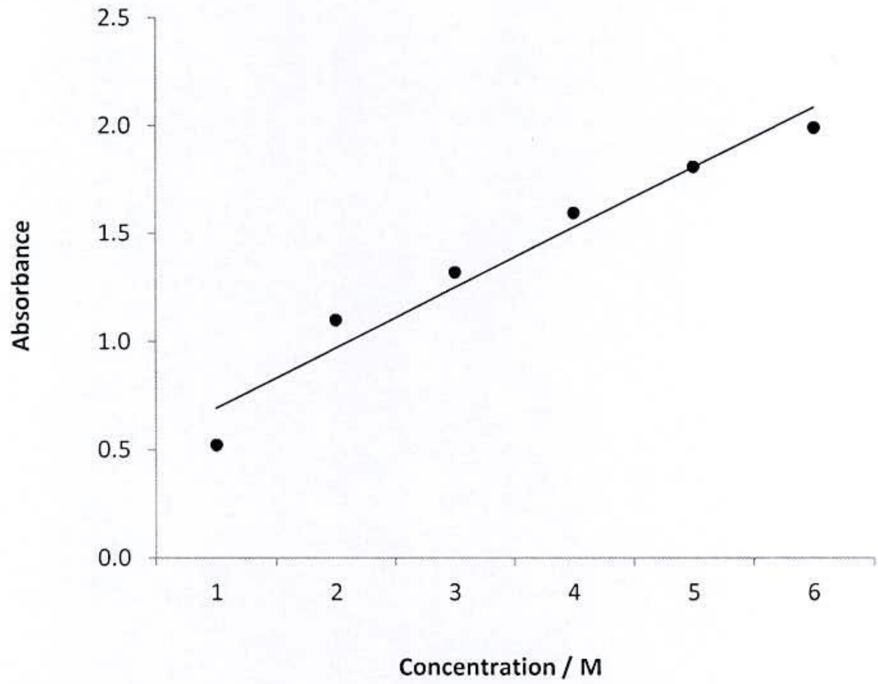


Figure 4.3.2: Construction of calibration curve for methylene blue

4.3.3 Estimation of equilibrium time for adsorption of methylene blue on prepared activated carbon (initial concentration of MB: 3.0×10^{-5} M and pH 6.0)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 6.0 and concentration of solution was 3.0×10^{-5} M at 30 °C. The amount of prepared activated carbon taken for each experiment was 0.1 g. Dimensions of the particles size were (200 mesh < particles diameter $\geq$ 400 mesh). Under this condition the equilibrium time for the adsorption of methylene blue onto prepared activated carbon was found to be about 3 hours. Results of equilibrium time were presented in Table 4.3.2 and in Figure 4.3.3.

Experimental conditions:

Sample	: Methylene blue
Initial concentration of methylene blue	: 3.0×10^{-5} M
Initial pH of the solution	: 6.0
Initial absorbance of the solution	: 1.404

Temperature	: 30 °C (±0.5 °C)
Rate of agitation	: 100 rpm
Amount of adsorbent	: 0.1 g
Particle size	: 200 mesh < particles diameter ≥ 400 mesh
Volume of methylene blue solution	: 25 mL
Reference	: DDW

Table 4.3.2: Data for the estimation of equilibrium time for the adsorption of methylene blue onto prepared activated carbon at 30 °C and pH 6.0 (initial concentration of methylene blue: 3.0×10^{-5} M)

No. of obs.	Contact time/min	Final absorbance	Initial conc. $C_0 \times 10^5$/M	Equilibrium conc. $C_e \times 10^5$/M	Amount adsorbed, x/m mg/g
1	5	0.884	2.775	1.747	0.832
2	10	0.761		1.504	1.024
3	20	0.665		1.314	1.151
4	30	0.512		1.012	1.407
5	45	0.421		0.832	1.567
6	60	0.302		0.597	1.727
7	90	0.256		0.506	1.823
8	120	0.212		0.419	1.887
9	150	0.216		0.427	1.887
10	180	0.216		0.427	1.887
11	240	0.214		0.423	1.887
12	300	0.213		0.421	1.887

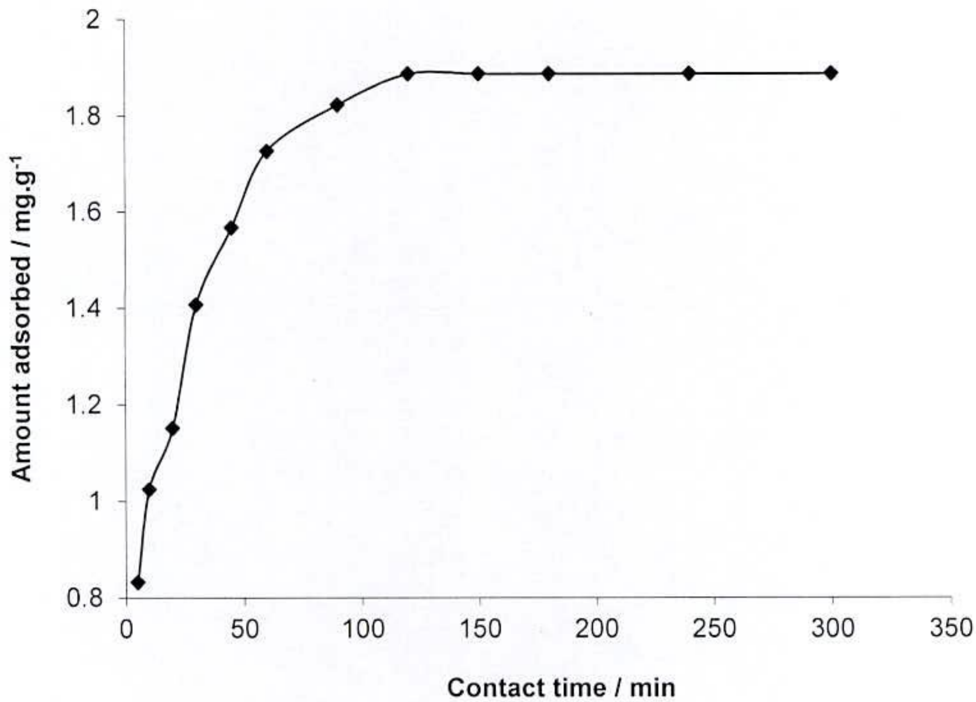


Figure 4.3.3.: Estimation of equilibrium time for adsorption of methylene blue onto prepared activated carbon (temperature: 30 °C; pH 6.0; initial concentration of methylene blue 3.0×10^{-5} M).

4.3.4 Estimation of equilibrium time for adsorption of MB on prepared activated carbon (initial concentration of methylene blue: 5.0×10^{-5} M and pH 6.0)

Following the similar procedure as describe in section 4.3.3, estimation of equilibrium time, the adsorption experiment was also carried out at pH 6.0 and concentration of solution was 5.0×10^{-5} M at 30 °C. The amount of prepared activated carbon taken for each experiment was 0.1 g. Dimensions of the particles size were (particles diameter ≤ 200 mesh). The equilibrium time for the adsorption of methylene blue onto prepared activated carbon was found to be about 3 hours. The overlaid adsorption spectra were shown in Figure 4.3.4. Results of equilibrium time were presented in Table 4.3.3 and in Figure 4.3.5. During estimating the equilibrium time, it was observed that removal of methylene blue on prepared activated carbon was found to be about 92.98% within 3 hours for all concentrations. It is obvious from section 4.3.3 and section 4.3.4

that as the particle sizes increased from (particles diameter ≤ 200 mesh) to (200 mesh < particles diameter ≥ 400 mesh) the extent of adsorption of methylene blue had been decreased

Experimental conditions:

Sample	: Methylene blue
Initial concentration of methylene blue	: 5.0×10^{-5} M
Initial pH of the solution	: 6.0
Temperature	: 30 °C (±0.5 °C)
Rate of agitation	: 100 rpm
Amount of adsorbent	: 0.1 g
Particle size	: particles diameter ≤ 200 mesh
Volume of methylene blue solution	: 40 mL
Reference	: DDW

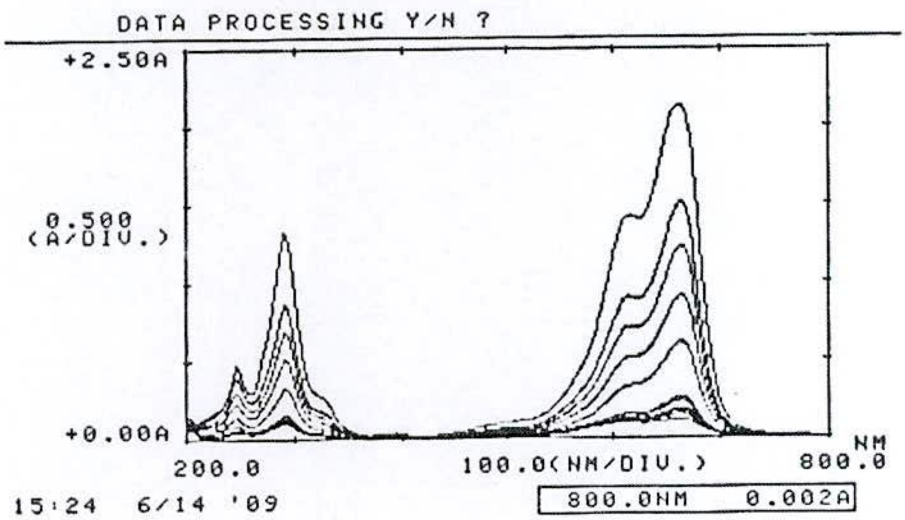


Figure 4.3.4: Overlay spectra for the adsorption of methylene blue on prepared activated carbon with time (temperature: 30 °C; pH 6.0; initial concentration of MB: 5.0×10^{-5} M).

Table 4.3.3: Data for the estimation of equilibrium time for the adsorption of MB on prepared activated carbon at 30 °C and pH 6.0 (initial concentration of MB: 5.0×10^{-5} M)

No. of obs.	Contact time/min	Final absorbance	Equilibrium conc. $C_e\times 10^5$/M	Amount adsorbed, x/m mg/g	% Removal
1	5	1.552	3.067	2.473	38.66
2	10	1.23	2.431	3.287	51.38
3	20	0.915	1.808	4.084	63.84
4	30	0.607	1.199	4.863	76.02
5	45	0.438	0.866	5.289	82.68
6	60	0.238	0.47	5.796	90.6
7	90	0.183	0.362	5.934	92.76
8	120	0.21	0.415	5.866	91.7
9	150	0.189	0.373	5.92	92.54
10	180	0.173	0.342	5.959	93.16
11	240	0.188	0.371	5.922	92.58
12	300	0.177	0.351	5.948	92.98
13	360	0.177	0.351	5.948	92.98

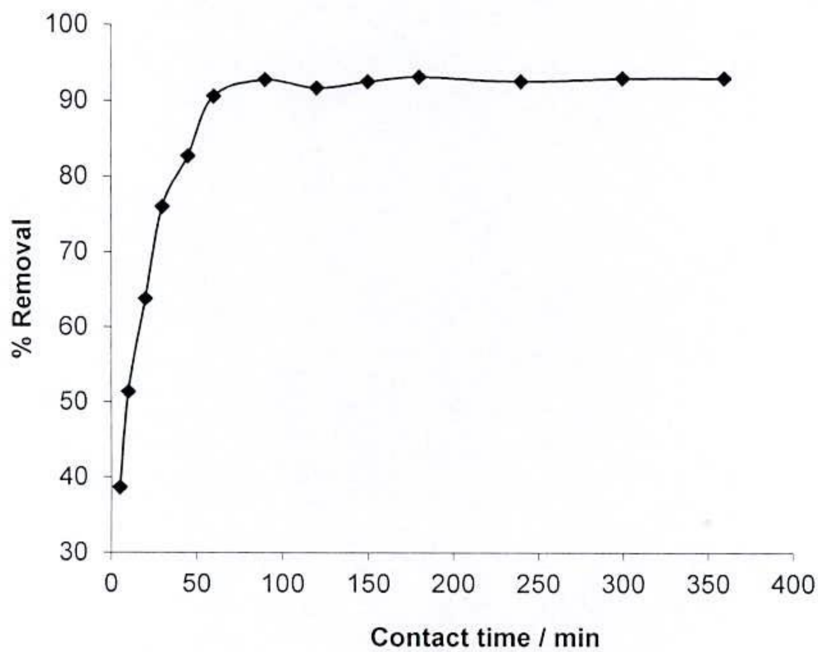


Figure 4.3.5: Estimation of equilibrium time for adsorption of methylene blue on prepared activated carbon (temperature: 30 °C; pH 6.0; initial concentration of MB: 5.0×10^{-5} M).

4.3.5 Adsorption isotherms

4.3.5.1 Adsorption isotherms of methylene blue on prepared activated carbon at pH 6.0 and different temperatures

Adsorption isotherms were determined at two different temperatures i.e., 30 °C and 40 °C using methylene blue solutions of pH 6.0. Results are presented in Table 4.3.4 and 4.3.5. The respective isotherms were shown in Figure 4.3.6 and 4.3.7. Under all conditions amount adsorbed increased with the increase of equilibrium concentration. The experimental data of two isotherms have been found to be fit into Freundlich isotherm (Figure 4.3.8 and 4.3.9). The value of n varies from 1 to 1.5. It is also seen from the results the isotherm of temperature 30 °C and 40 °C suggested that chemisorptions of methylene blue were occurred on prepared activated carbon.

The experiment has been carried out under the following experimental condition:

Initial pH of the solution	: 6.0
Temperature	: 30 °C and 40 °C (±0.5 °C)
Amount of adsorbent	: 0.1 g
Particle size	: particles diameter ≤ 200 mesh
Volume of methylene blue solution	: 40 mL
Rate of agitation	: 100 rpm
Time of adsorption	: 5.0 hours
Reference	: DDW

Table 4.3.4: Variation of amount adsorbed of methylene blue on prepared activated carbon with different concentration at 30 °C and pH 6.0

No. of obs.	Initial abs.	Final abs.	Initial conc. $C_0 \times 10^5$/M	Equilibrium conc. $C_e \times 10^5$/M	Amount adsorbed, per gram $\times 10^5$/mole	Amount absorbed mg/g
1	0.522	0.036	1.032	0.071	0.384	1.228
2	1.100	0.050	2.174	0.108	0.826	2.642
3	1.318	0.062	2.605	0.122	0.993	3.176
4	1.593	0.082	3.148	0.162	1.194	3.819
5	1.808	0.086	3.573	0.169	1.361	4.353
6	2.196	0.120	4.340	0.237	1.641	5.250

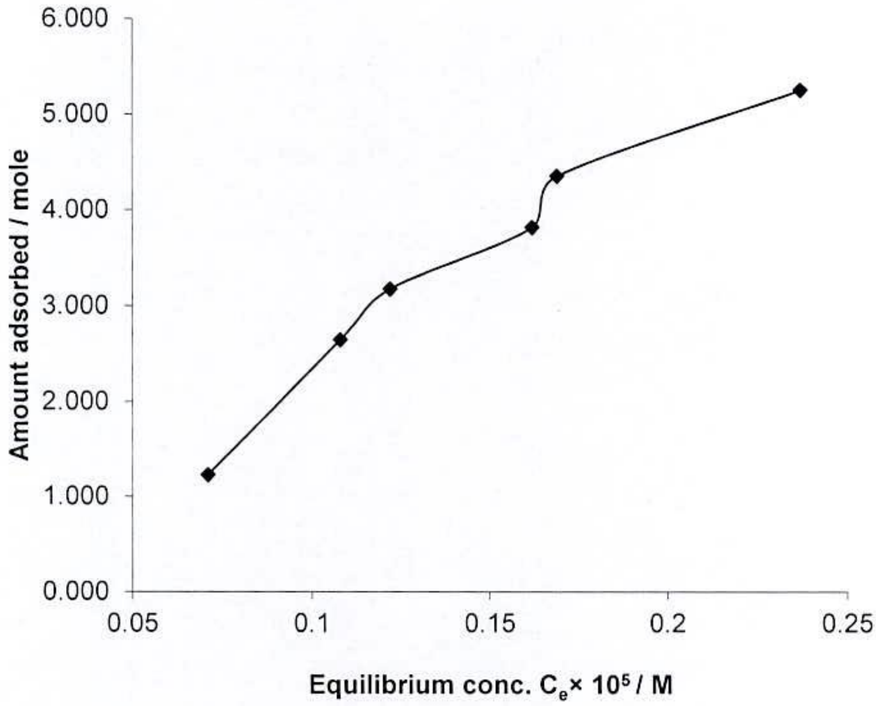


Figure 4.3.6: Adsorption isotherm of methylene blue on prepared activated carbon at 30 °C and pH 6.0

Table 4.3.5: Variation of amount adsorbed of methylene blue on prepared activated carbon with different concentration at 40 °C and pH 6.0

No. of obs.	Initial abs.	Final abs.	Initial conc. $C_0 \times 10^5$/M	Equilibrium conc. $C_e \times 10^5$/M	Amount adsorbed, per gram $\times 10^5$/mole	Amount absorbed mg/g
1	0.522	0.026	1.032	0.051	0.392	1.254
2	1.100	0.051	2.174	0.100	0.829	2.651
3	1.318	0.056	2.605	0.110	0.998	3.192
4	1.593	0.070	3.148	0.138	1.204	3.851
5	1.808	0.081	3.573	0.160	1.365	4.366
6	1.990	0.092	3.933	0.182	1.500	4.798
7	2.196	0.110	4.340	0.217	1.650	5.277

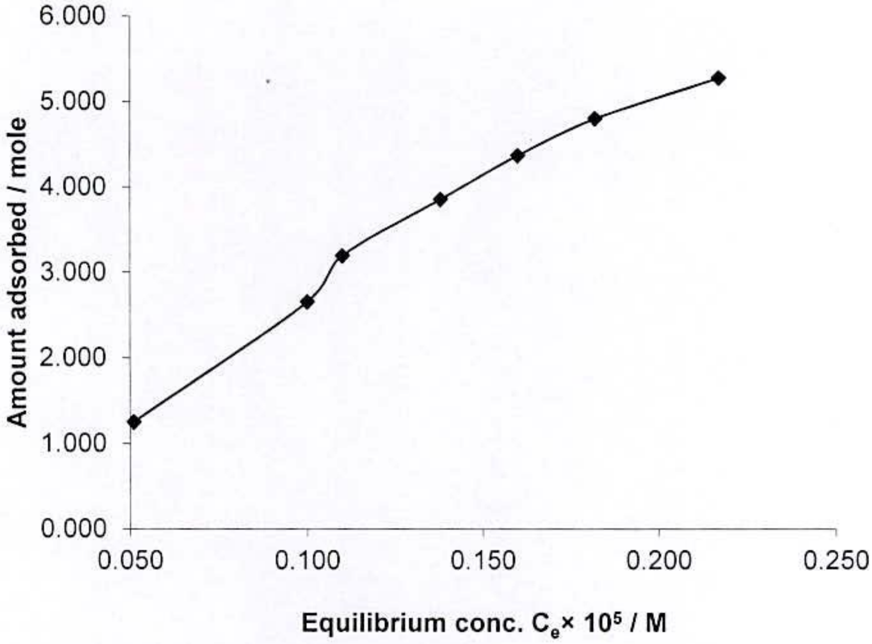


Figure 4.3.7: Adsorption isotherm of methylene blue on prepared activated carbon at 40 °C and pH 6.0

Table 4.3.6: Data for Freundlich isotherm for the adsorption of methylene blue on prepared activated carbon at 30 °C and pH 6.0

No. of obs.	Amount adsorbed, X/m (mg/g)	Equilibrium conc. $C_e \times 10^3$ (M)	Log x/m	-log C_e	$C_e/(x/m) \times 10^3$
1	1.228	7.100	0.089	2.149	-0.024
2	2.642	10.800	0.422	1.967	-4.661
3	3.176	12.200	0.502	1.914	-3.813
4	3.819	16.200	0.582	1.790	-3.077
5	4.353	16.900	0.639	1.772	-2.774
6	5.250	23.700	0.720	1.625	-2.257

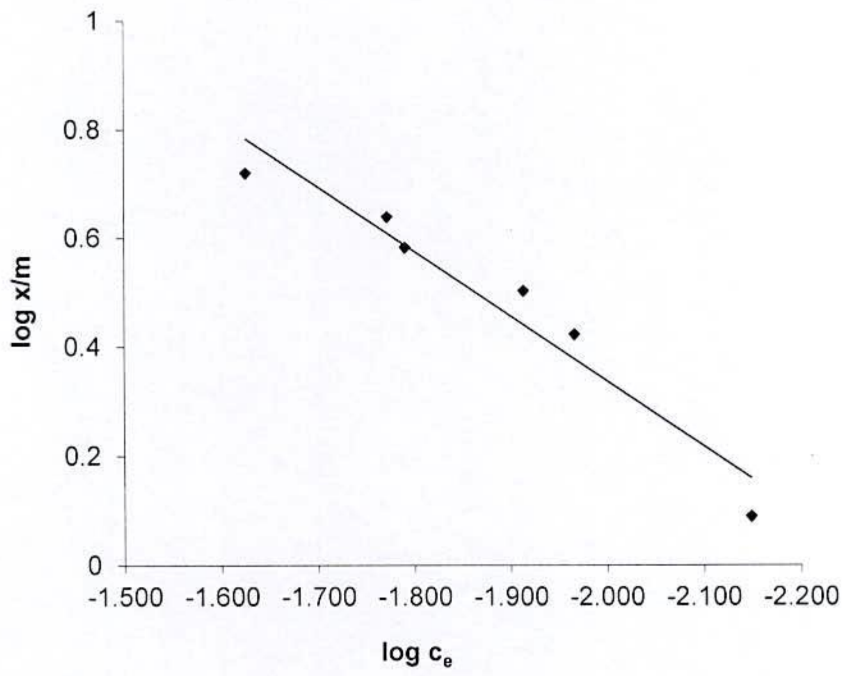


Figure 4.3.8: Freundlich isotherm for the adsorption of methylene blue on prepared activated carbon at 30 °C and pH 6.0

Table 4.3.7: Data for Freundlich isotherm for the adsorption of methylene blue on prepared activated carbon at 40 °C and pH 6.0

No. of obs.	Amount adsorbed, X/m (mg/g)	Equilibrium conc. $C_e \times 10^3$ (M)	Log x/m	-log C_e	$C_e/(x/m) \times 10^3$
1	1.254	5.100	0.098	2.292	-0.023
2	2.651	10.000	0.423	2.000	-4.724
3	3.192	11.000	0.504	1.959	-3.886
4	3.851	13.800	0.586	1.860	-3.177
5	4.366	16.000	0.640	1.796	-2.806
6	4.798	18.200	0.681	1.740	-2.555
7	5.277	21.700	0.722	1.664	-2.303

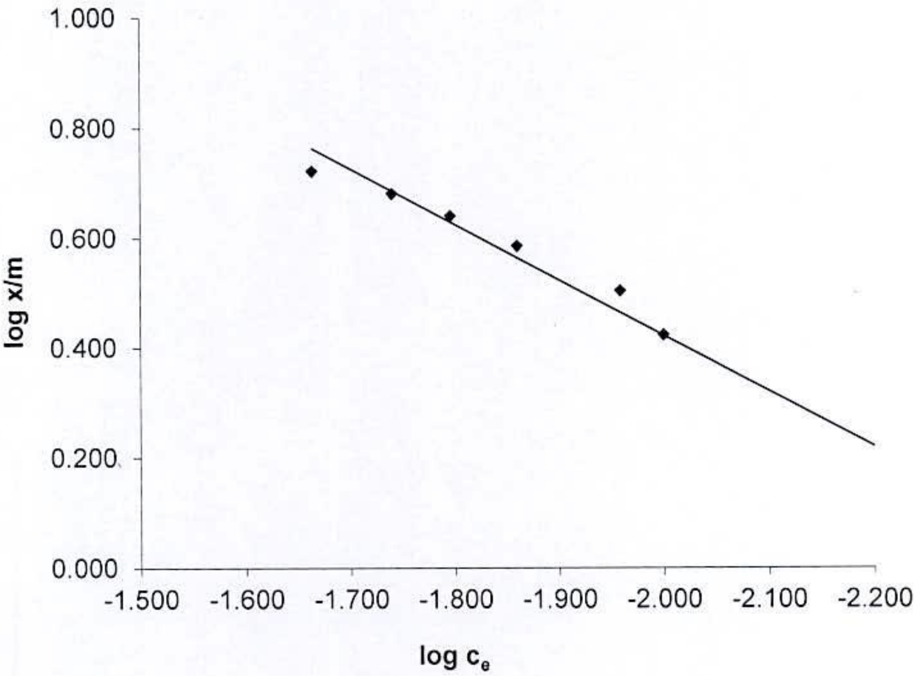


Figure 4.3.9: Freundlich isotherm for the adsorption of methylene blue on prepared activated carbon at 40 °C and pH 6.0

4.4 Adsorption study of Lead on prepared activated carbon

Lead metals are discharged into the water bodies from various industrial sources. There are several methods available for the removal of metal ions from wastewater. Among them, the adsorption process provides an attractive alternative for the treatment of wastewater, especially if the adsorbent is inexpensive and does not require additional pretreatment before its application. The adsorption characteristics of lead ions onto prepared activated carbon from rice husk are discussed. Batch adsorption experiments were carried out under varying experimental conditions such as, particle size of adsorbent, contact time, initial concentration of lead solution.

Five series of experiments varying the amount of activated carbon were carried out to study the effects of initial lead concentration, pH and shaking frequency on the removal of lead from aqueous solution. The equilibrium time for adsorption of aqueous Pb^{2+} ions onto prepared activated carbon is 3 hours which is almost same as found for methylene blue discussed in section 4.3.3. The adsorption processes were performed at different pH and shaking frequency. The pH and shaking frequency at which maximum adsorption are 5.7 and 150 rev/min respectively. The attained data are presented as the plots of removal of lead (mg/g), q, against adsorption time, t. The percentage removal of Pb^{2+} at equilibrium is also important, for different adsorption conditions. The removal of Pb^{2+}, q, and percentage removal of Pb^{2+} used are defined as follows:

Removal of Pb^{2+}: $q = \frac{(C_0 - C)V}{w}$

Percentage removal of Pb^{2+}: $\% \, Removal = \frac{(C_0 - C)}{C_0} \times 100$

$$= \frac{qw}{VC_0} \times 100$$

Where, C and C_0 are the Pb^{2+} concentrations (mg/g) at $t=t$ and $t=0$, respectively; V is the volume of aqueous solution (L); and w is the mass of activated carbon used (g).

4.4.1 Effect of pH on adsorption of lead onto prepared activated carbon

The adsorption experiments were carried out at different pH to optimize the adsorption capabilities of prepared activated carbon.

The experiment has been carried out under the following experimental condition:

pH of the solutions	: 3.0, 4.0, 5.0, 5.7 and 6.0
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Pb^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Pb^{2+} solution	: 700 mg/L

The results representing the effect of pH are tabulated in Table 4.4.1 and Figure 4.4.1. The results are close to those of other reports [115—117]. The experimented pH values are 3.0, 4.0, 5.0, 5.7 and 6.0. To fix the pH value 5.7 more experiments were done at pH 5.1, 5.2, 5.3, 5.4, 5.5, 5.6, 5.7, 5.8 and 6.0. It was seen from the results that pH value 5.7 showed the maximum adsorption almost in all cases. The removal of Pb^{2+} ions are increased with an increase of pH reaching the maximum at pH 5.7 it decreased again. No effect of initial concentration of Pb^{2+} on pH was observed. The effect of pH observed here can probably be explained by the ion-exchange mechanism proposed by Daifullah *et al.* [118].

$$Pb^{2+} + 2(\text{-SiOH}) \Leftrightarrow Pb(\text{—SiO})_2 + 2H^+$$

where Pb^{2+}, —SiOH, and H+ are lead ion, silanol group, and proton, respectively.

As discussed in section 4.3 that our prepared activated carbon contains 39.47% ash (Table 4.2.4) and ash comprise a significant amount of SiO_2 and the value is 92% (Table 4.2.3). The silanol group on the surface of activated carbon was produced through the hydrolysis of silicon oxide and the Pb^{2+} ion in solution substituted the proton of the silanol group [118]. At low pH, the surface of the activated carbon was surrounded by proton (H^+), which prevented the Pb^{2+} from approaching the silanol group and, consequently, reduced the adsorption of lead ion. With increasing pH value, electrostatic repulsion decreased due to the reduction of positive charge density around the silanol group, thus resulting in enhancement of lead adsorption [116].

Table 4.4.1 : Effect of pH on adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at shaking frequency: 150 rev/min, initial concentration of Pb^{2+} ions: 3.34×10^{-3} M and 30 °C.

pH	Shacking Frequency (rev/min)	Amount of carbon (g)	Initial Concentration (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
3	150	1.00	3.34×10^{-3}	622.37	77.63	11.09	65.99
		2.00		469.14	230.86	32.98	98.12
		3.00		369.60	330.40	47.20	93.61
		4.00		279.37	420.63	60.09	89.38
		5.00		189.28	510.72	72.96	86.82
4	150	1.00	3.34×10^{-3}	599.06	100.94	14.42	85.80
		2.00		454.09	245.91	35.13	104.51
		3.00		338.45	361.55	51.65	102.44
		4.00		252.77	447.23	63.89	95.04
		5.00		164.29	535.71	76.53	91.07
5	150	1.00	3.34×10^{-3}	577.85	122.15	17.45	103.83
		2.00		432.74	267.26	38.18	113.59
		3.00		321.16	378.84	54.12	107.34
		4.00		226.24	473.76	67.68	100.67
		5.00		147.07	552.93	78.99	94.00
5.7	150	1.00	3.34×10^{-3}	546.07	153.93	21.99	130.84
		2.00		410.76	289.24	41.32	122.93
		3.00		289.45	410.55	58.65	116.32
		4.00		193.62	506.38	72.34	107.61
		5.00		109.83	590.17	84.31	100.33
6	150	1.00	3.34×10^{-3}	573.65	126.35	18.05	107.40
		2.00		423.29	276.71	39.53	117.60
		3.00		309.96	390.04	55.72	110.51
		4.00		234.36	465.64	66.52	98.95
		5.00		147.84	552.16	78.88	93.87

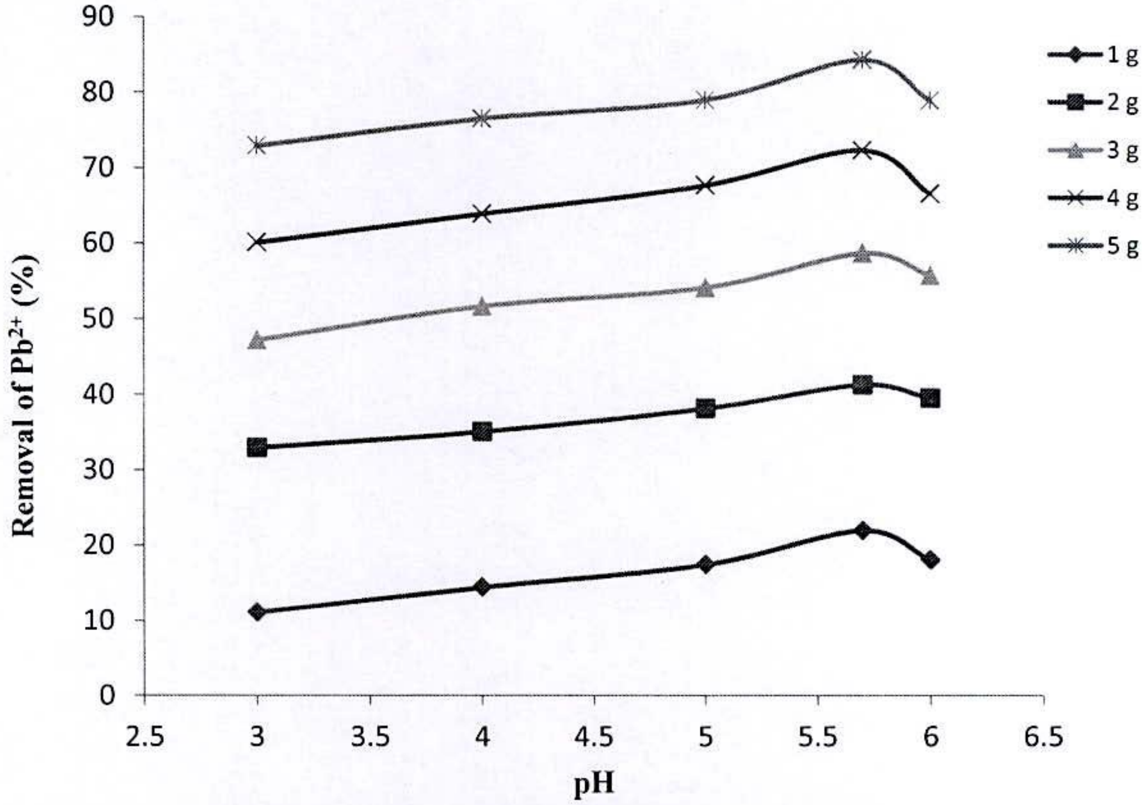


Figure 4.4.1 : Effect of pH on adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at shaking frequency: 150 rev/min, initial concentration of Pb^{2+} ions: 3.34×10^{-3} M and 30 °C.

4.4.2 Effect of shaking frequency on adsorption of lead onto prepared activated carbon

The adsorption experiments were carried out at different shaking frequency to optimize the adsorption capabilities of prepared activated carbon.

The experiment has been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter $\leq$ 200 mesh
Volume of aqueous Pb^{2+} solution	: 850 mL
Rate of shaking	: 50, 100, 150 and 200 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Pb^{2+} solution	: 700 mg/L

The results showing the effect of shaking frequency are tabulated in Table 4.4.2 and Figure 4.4.2. The results are close to those of other reports [116]. The experimented shaking frequencies are 50, 100, 150 and 200 rev/min. It was seen from the results that shaking frequency value of 150 rev/min showed the maximum adsorption almost in all cases. The removal of Pb^{2+} ions are increased with an increase of shaking frequency reaching the maximum value of 150 rev/min it decreased again. No effect of initial concentration of Pb^{2+} on shaking frequency was observed. It is seen that the higher the shaking frequency, the faster the removal and higher removal at equilibrium. Wong *et al.* [116] reported the same tendency for the system of Pb^{2+} tartaric acid modified rice hull. The reason for this is that when the shaking frequency is increased, the rate of mass transfer of lead ion through an artificial liquid film outside activated carbon particles is increased. It is also noted from Figure 4.4.2 that when the shaking frequency is over 150 rev/min, the effect of stroke speed becomes insignificant. This implies the resistance of the mass transfer of lead ion through the liquid film becomes null.

Table 4.4.2 : Effect of shaking frequency on adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at pH: 5.7, initial concentration of Pb^{2+} ions: 3.34×10^{-3} M and 30 °C.

pH	Shacking Frequency (rev/min)	Amount of carbon (g)	Initial Concentration (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	50	1.00	3.34×10^{-3}	596.54	103.46	14.78	87.94
		2.00		464.31	235.69	33.67	100.17
		3.00		341.39	358.61	51.23	101.61
		4.00		262.43	437.57	62.51	92.98
		5.00		186.13	513.87	73.41	87.36
5.7	100	1.00	3.34×10^{-3}	568.75	131.25	18.75	111.56
		2.00		438.69	261.31	37.33	111.06
		3.00		312.62	387.38	55.34	109.76
		4.00		218.89	481.11	68.73	102.24
		5.00		133.28	566.72	80.96	96.34
5.7	150	1.00	3.34×10^{-3}	546.07	153.93	21.99	130.84
		2.00		410.76	289.24	41.32	122.93
		3.00		289.45	410.55	58.65	116.32
		4.00		193.62	506.38	72.34	107.61
		5.00		109.83	590.17	84.31	100.33
5.7	200	1.00	3.34×10^{-3}	556.29	143.71	20.53	122.15
		2.00		419.09	280.91	40.13	119.39
		3.00		297.78	402.22	57.46	113.96
		4.00		201.74	498.26	71.18	105.88
		5.00		116.97	583.03	83.29	99.12

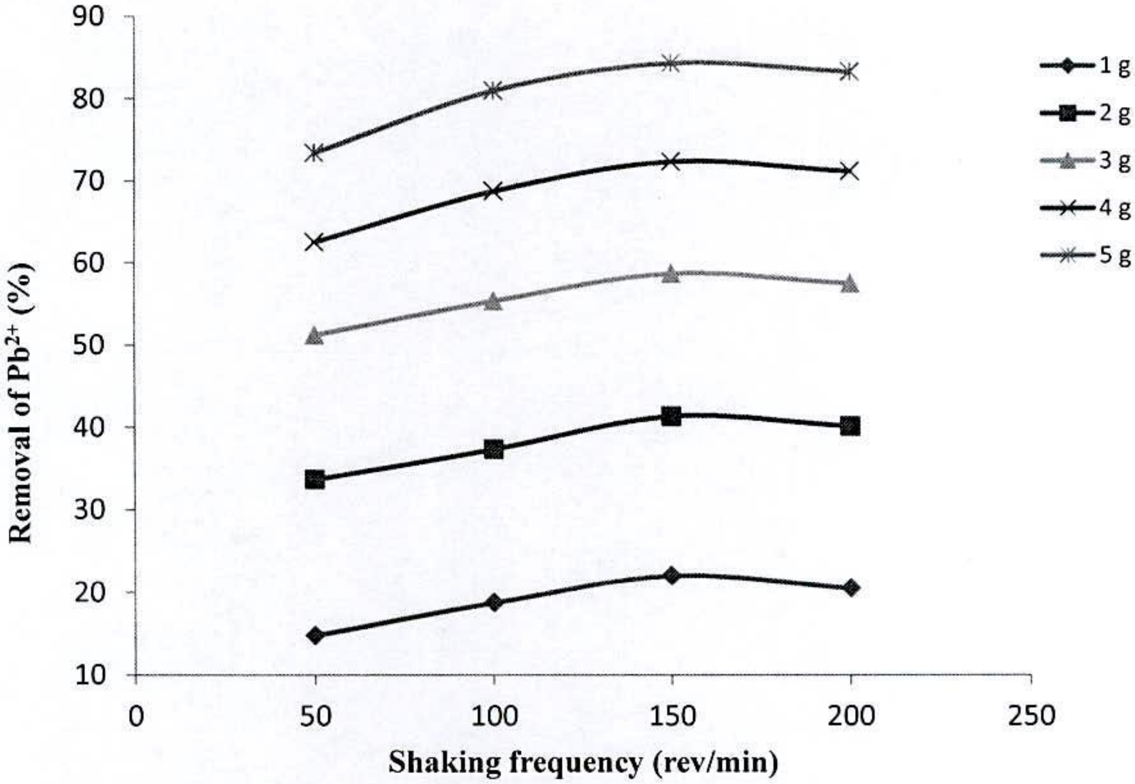


Figure 4.4.2 : Effect of shaking frequency on adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at pH: 5.7, initial concentration of Pb^{2+} ions: 3.34×10^{-3} M and 30 °C.

4.4.3 Estimation of equilibrium time for adsorption of aqueous Pb^{2+} ions onto prepared activated carbon (initial concentration of Pb^{2+}: 1.48×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 1.48×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Pb^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.4.3 and in Figure 4.4.3. It is observed from Table 4.4.3 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 40 to 60 mg/g while the initial concentration of Pb^{2+} is 1.48×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Pb^{2+} ions onto prepared activated carbon is found to be 91.00% within 3 hours and that for minimum is 22.10%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Pb^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.4.4). The value of n is 6.45. It is also seen from the results the isotherm that chemisorption of Pb^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Pb^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Pb^{2+} solution	: 1.48×10^{-3} M

Table 4.4.3 : **Determination of equilibrium time for the adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+} ions: 1.48×10^{-3} M and 30 °C.**

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	1.48×10^{-3}	233.70	66.30	22.10	56.36
			2.00		182.70	117.30	39.10	49.85
			3.00		134.46	165.54	55.18	46.90
			4.00		97.09	202.91	67.64	43.12
			5.00		62.73	237.27	79.09	40.34
5.7	150	60	1.00	1.48×10^{-3}	230.40	69.60	23.20	59.16
			2.00		172.99	127.01	42.34	53.98
			3.00		122.37	177.63	59.21	50.33
			4.00		81.13	218.87	72.96	46.51
			5.00		56.91	243.09	81.03	41.33
5.7	150	90	1.00	1.48×10^{-3}	227.94	72.06	24.02	61.25
			2.00		167.40	132.60	44.20	56.36
			3.00		118.13	181.87	60.62	51.53
			4.00		71.01	228.99	76.33	48.66
			5.00		47.40	252.60	84.20	42.94
5.7	150	120	1.00	1.48×10^{-3}	225.03	74.97	24.99	63.72
			2.00		162.60	137.40	45.80	58.39
			3.00		108.62	191.38	63.79	54.22
			4.00		65.40	234.60	78.20	49.85
			5.00		40.38	259.62	86.54	44.14
5.7	150	150	1.00	1.48×10^{-3}	223.38	76.62	25.54	65.13
			2.00		160.50	139.50	46.50	59.29
			3.00		104.39	195.61	65.20	55.42
			4.00		55.29	244.71	81.57	52.00
			5.00		36.00	264.00	88.00	44.88
5.7	150	180	1.00	1.48×10^{-3}	221.64	78.36	26.12	66.61
			2.00		154.98	145.02	48.34	61.63
			3.00		104.39	195.61	65.20	55.42
			4.00		55.32	244.68	81.56	51.99
			5.00		27.03	272.97	90.99	46.40
5.7	150	210	1.00	1.48×10^{-3}	221.64	78.36	26.12	66.61
			2.00		155.10	144.90	48.30	61.58
			3.00		104.36	195.64	65.21	55.43
			4.00		55.32	244.68	81.56	51.99
			5.00		27.00	273.00	91.00	46.41

Continued

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	1.48×10^{-3}	221.55	78.45	26.15	66.68
			2.00		154.80	145.20	48.40	61.71
			3.00		104.33	195.67	65.22	55.44
			4.00		55.29	244.71	81.57	52.00
			5.00		27.24	272.76	90.92	46.37
5.7	150	270	1.00	1.48×10^{-3}	221.55	78.45	26.15	66.68
			2.00		154.80	145.20	48.40	61.71
			3.00		104.39	195.61	65.20	55.42
			4.00		55.32	244.68	81.56	52.00
			5.00		27.09	272.91	90.97	46.39
5.7	150	300	1.00	1.48×10^{-3}	221.64	78.36	26.12	66.61
			2.00		155.10	144.90	48.30	61.58
			3.00		104.39	195.61	65.20	55.42
			4.00		55.32	244.68	81.56	52.00
			5.00		27.15	272.85	90.95	46.38
5.7	150	330	1.00	1.48×10^{-3}	221.64	78.36	26.12	66.61
			2.00		154.80	145.20	48.40	61.71
			3.00		104.39	195.61	65.20	55.42
			4.00		55.32	244.68	81.56	52.00
			5.00		27.03	272.97	90.99	46.40
5.7	150	360	1.00	1.48×10^{-3}	221.67	78.33	26.11	66.58
			2.00		155.10	144.90	48.30	61.58
			3.00		104.43	195.57	65.19	55.41
			4.00		55.29	244.71	81.57	52.00
			5.00		27.00	273.00	91.00	46.41

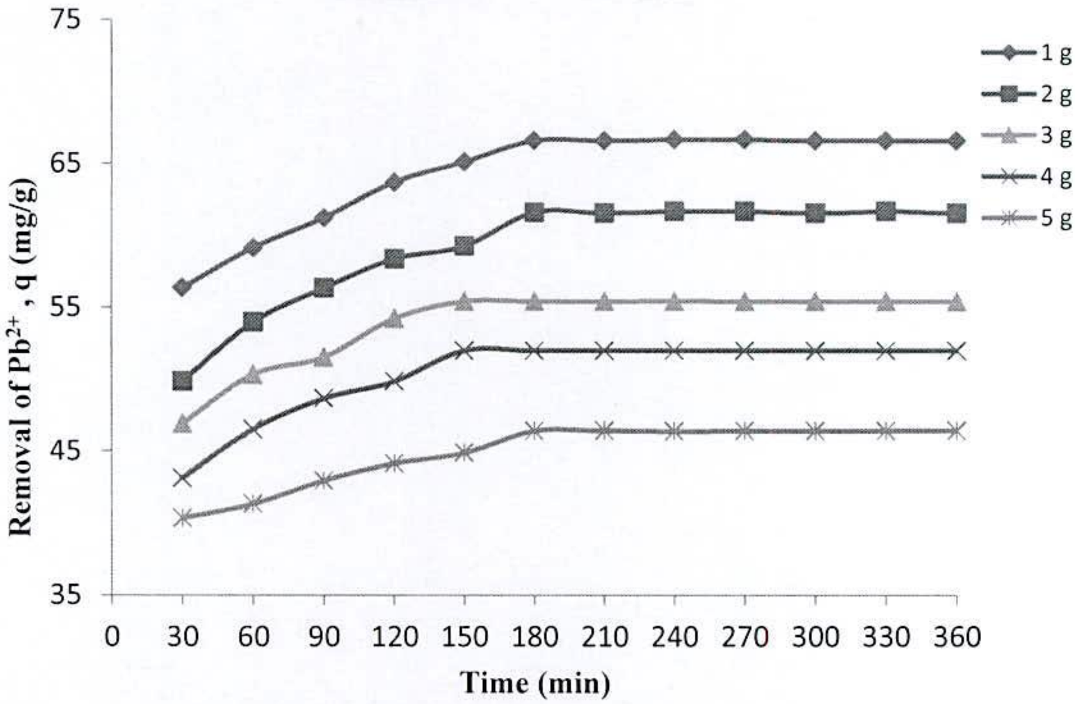


Figure 4.4.3 : Plot of removal of Pb^{2+} against time to determine equilibrium time of adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 1.48×10^{-3} M and 30 °C.

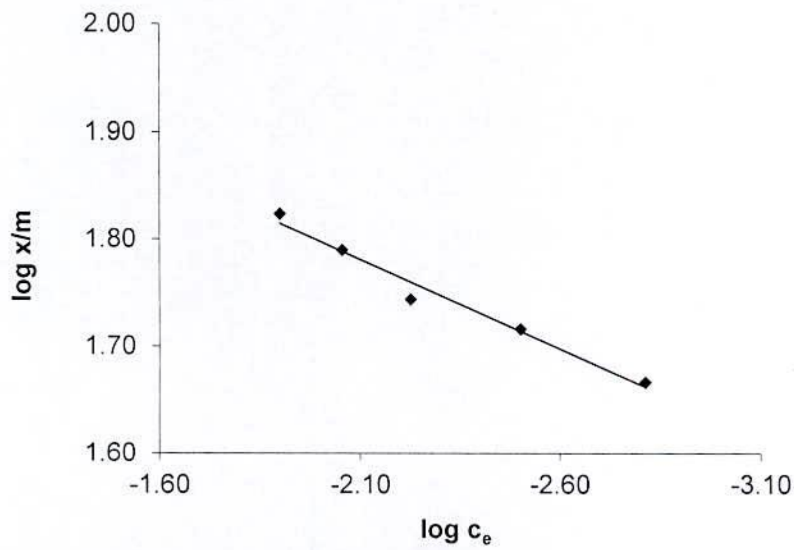


Figure 4.4.4 : Freundlich isotherm for the adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 1.48×10^{-3} M and 30 °C.

4.4.4 Estimation of equilibrium time for adsorption of aqueous Pb^{2+} ions onto prepared activated carbon (initial concentration of Pb^{2+}: 1.93×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 1.93×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Pb^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.4.4 and in Figure 4.4.5. It is observed from Table 4.4.4 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 60 to 80 mg/g while the initial concentration of Pb^{2+} is 1.93×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Pb^{2+} ions onto prepared activated carbon is found to be 88.34% within 3 hours and that for minimum is 20.78%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Pb^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.4.6). The value of n is 7.25. It is also seen from the results the isotherm that chemisorption of Pb^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Pb^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Pb^{2+} solution	: 1.93×10^{-3} M

Table 4.4.4 : Determination of equilibrium time for the adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+} ions: 1.93×10^{-3} M and 30 °C.

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	1.93×10^{-3}	316.88	83.12	20.78	70.65
			2.00		243.03	156.97	39.24	66.71
			3.00		187.99	212.01	53.00	60.07
			4.00		134.61	265.39	66.35	56.40
			5.00		91.16	308.84	77.21	52.50
5.7	150	60	1.00	1.93×10^{-3}	311.16	88.84	22.21	75.51
			2.00		235.92	164.08	41.02	69.73
			3.00		177.87	222.13	55.53	62.94
			4.00		124.33	275.67	68.92	58.58
			5.00		84.04	315.96	78.99	53.71
5.7	150	90	1.00	1.93×10^{-3}	307.64	92.36	23.09	78.51
			2.00		226.00	174.00	43.50	73.95
			3.00		172.83	227.17	56.79	64.36
			4.00		112.29	287.71	71.93	61.14
			5.00		76.52	323.48	80.87	54.99
5.7	150	120	1.00	1.93×10^{-3}	304.52	95.48	23.87	81.16
			2.00		220.00	180.00	45.00	76.50
			3.00		162.05	237.95	59.49	67.42
			4.00		100.98	299.02	74.76	63.54
			5.00		69.32	330.68	82.67	56.22
5.7	150	150	1.00	1.93×10^{-3}	302.76	97.24	24.31	82.65
			2.00		212.04	187.96	46.99	79.88
			3.00		155.41	244.59	61.15	69.30
			4.00		88.04	311.96	77.99	66.29
			5.00		56.88	343.12	85.78	58.33
5.7	150	180	1.00	1.93×10^{-3}	299.80	100.20	25.05	85.17
			2.00		210.86	189.14	47.29	80.39
			3.00		155.41	244.59	61.15	69.30
			4.00		88.08	311.92	77.98	66.28
			5.00		47.20	352.80	88.20	59.98
5.7	150	210	1.00	1.93×10^{-3}	299.80	100.20	25.05	85.17
			2.00		210.90	189.10	47.28	80.37
			3.00		155.41	244.59	61.15	69.30
			4.00		88.08	311.92	77.98	66.28
			5.00		46.80	353.20	88.30	60.04

Continued

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	1.93×10^{-3}	299.60	100.40	25.10	85.34
			2.00		210.81	189.19	47.30	80.41
			3.00		155.50	244.50	61.12	69.27
			4.00		88.04	311.96	77.99	66.29
			5.00		47.20	352.80	88.20	59.98
5.7	150	270	1.00	1.93×10^{-3}	299.64	100.36	25.09	85.31
			2.00		210.81	189.19	47.30	80.41
			3.00		155.54	244.46	61.11	69.26
			4.00		88.08	311.92	77.98	66.28
			5.00		47.20	352.80	88.20	59.98
5.7	150	300	1.00	1.93×10^{-3}	299.80	100.20	25.05	85.17
			2.00		210.81	189.19	47.30	80.41
			3.00		155.54	244.46	61.11	69.26
			4.00		88.08	311.92	77.98	66.28
			5.00		46.64	353.36	88.34	60.07
5.7	150	330	1.00	1.93×10^{-3}	299.68	100.32	25.08	85.27
			2.00		210.81	189.19	47.30	80.41
			3.00		155.54	244.46	61.11	69.26
			4.00		88.08	311.92	77.98	66.28
			5.00		47.20	352.80	88.20	59.98
5.7	150	360	1.00	1.93×10^{-3}	299.80	100.20	25.05	85.17
			2.00		210.80	189.20	47.30	80.41
			3.00		155.52	244.48	61.12	69.27
			4.00		88.12	311.88	77.97	66.27
			5.00		47.20	352.80	88.20	59.98

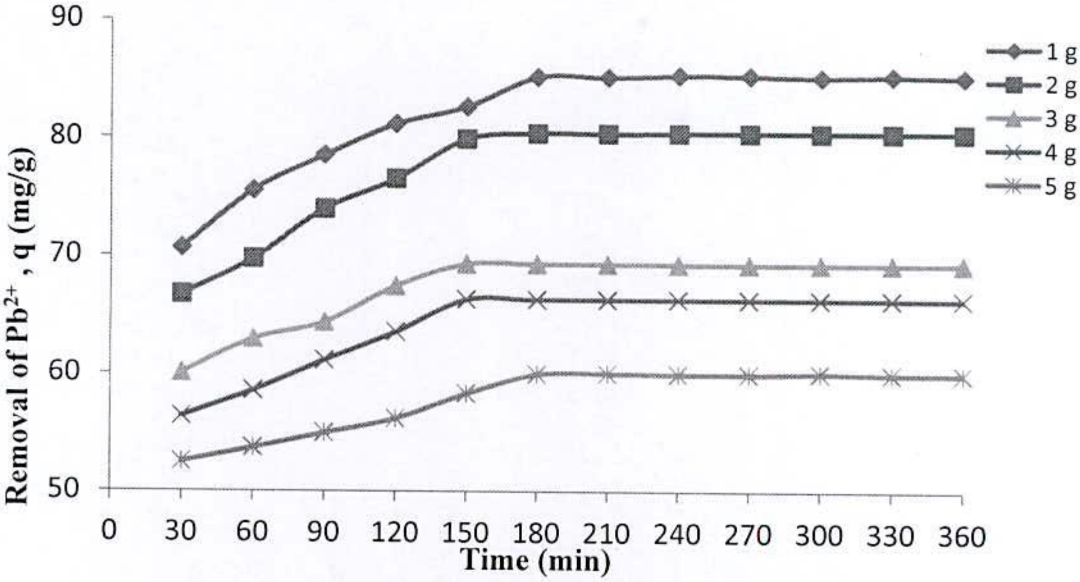


Figure 4.4.5 : Plot of removal of Pb^{2+} against time to determine equilibrium time of adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 1.93×10^{-3} M and 30 °C.

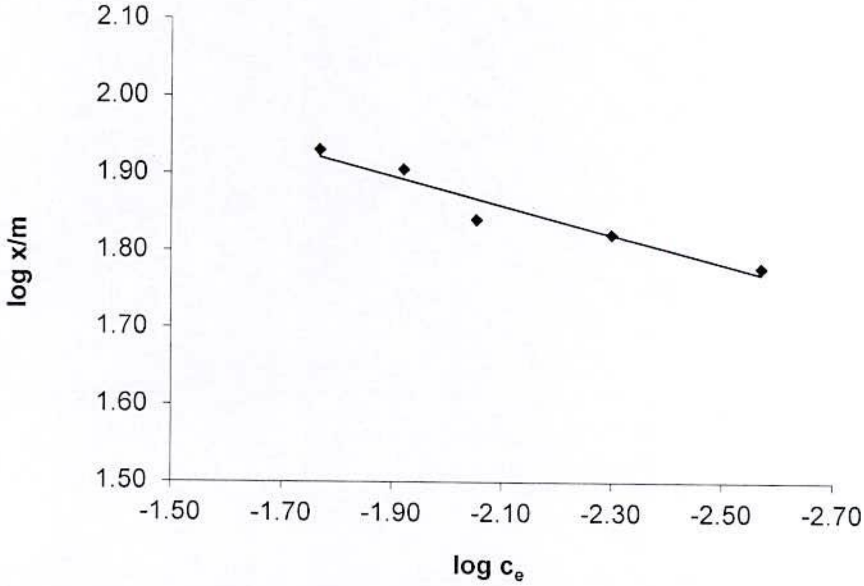


Figure 4.4.6 : Freundlich isotherm for the adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 1.93×10^{-3} M and 30 °C.

4.4.5 Estimation of equilibrium time for adsorption of aqueous Pb^{2+} ions onto prepared activated carbon (initial concentration of Pb^{2+}: 2.42×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 2.42×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Pb^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.4.5 and in Figure 4.4.7. It is observed from Table 4.4.5 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 70 to 85 mg/g while the initial concentration of Pb^{2+} is 2.42×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Pb^{2+} ions onto prepared activated carbon is found to be 87.83% within 3 hours and that for minimum is 19.99%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Pb^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.4.8). The value of n is 5.43. It is also seen from the results the isotherm that chemisorption of Pb^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Pb^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Pb^{2+} solution	: 2.42×10^{-3} M

Table 4.4.5: **Determination of equilibrium time for the adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+} ions: 2.42×10^{-3} M and 30 °C.**

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	2.42×10^{-3}	400.05	99.95	19.99	84.96
			2.00		312.64	187.37	37.47	79.63
			3.00		233.94	266.07	53.21	75.39
			4.00		170.97	329.03	65.81	69.92
			5.00		119.55	380.45	76.09	64.68
5.7	150	60	1.00	2.42×10^{-3}	394.80	105.20	21.04	89.42
			2.00		302.34	197.67	39.53	84.01
			3.00		223.24	276.77	55.35	78.42
			4.00		156.82	343.18	68.64	72.93
			5.00		104.60	395.40	79.08	67.22
5.7	150	90	1.00	2.42×10^{-3}	389.00	111.00	22.20	94.35
			2.00		289.85	210.15	42.03	89.31
			3.00		209.32	290.68	58.14	82.36
			4.00		142.69	357.32	71.46	75.93
			5.00		90.50	409.50	81.90	69.62
5.7	150	120	1.00	2.42×10^{-3}	384.50	115.50	23.10	98.18
			2.00		279.90	220.10	44.02	93.54
			3.00		196.02	303.98	60.80	86.13
			4.00		131.14	368.87	73.77	78.38
			5.00		84.55	415.45	83.09	70.63
5.7	150	150	1.00	2.42×10^{-3}	381.50	118.50	23.70	100.73
			2.00		274.60	225.40	45.08	95.80
			3.00		183.77	316.23	63.25	89.60
			4.00		119.90	380.10	76.02	80.77
			5.00		72.95	427.05	85.41	72.60
5.7	150	180	1.00	2.42×10^{-3}	378.40	121.60	24.32	103.36
			2.00		269.65	230.35	46.07	97.90
			3.00		183.77	316.23	63.25	89.60
			4.00		118.34	381.66	76.33	81.10
			5.00		60.85	439.15	87.83	74.66
5.7	150	210	1.00	2.42×10^{-3}	378.35	121.65	24.33	103.40
			2.00		269.46	230.54	46.11	97.98
			3.00		183.82	316.18	63.24	89.58
			4.00		118.34	381.66	76.33	81.10
			5.00		60.85	439.15	87.83	74.66

Continued

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	2.42×10^{-3}	378.40	121.60	24.32	103.36
			2.00		269.41	230.60	46.12	98.00
			3.00		183.82	316.18	63.24	89.58
			4.00		118.40	381.61	76.32	81.09
			5.00		60.85	439.15	87.83	74.66
5.7	150	270	1.00	2.42×10^{-3}	378.45	121.55	24.31	103.32
			2.00		269.46	230.55	46.11	97.98
			3.00		183.77	316.23	63.25	89.60
			4.00		118.40	381.61	76.32	81.09
			5.00		60.85	439.15	87.83	74.66
5.7	150	300	1.00	2.42×10^{-3}	378.40	121.60	24.32	103.36
			2.00		269.46	230.55	46.11	97.98
			3.00		183.77	316.23	63.25	89.60
			4.00		118.40	381.61	76.32	81.09
			5.00		60.85	439.15	87.83	74.66
5.7	150	330	1.00	2.42×10^{-3}	378.45	121.55	24.31	103.32
			2.00		269.46	230.55	46.11	97.98
			3.00		183.77	316.23	63.25	89.60
			4.00		118.40	381.61	76.32	81.09
			5.00		60.85	439.15	87.83	74.66
5.7	150	360	1.00	2.42×10^{-3}	378.45	121.55	24.31	103.32
			2.00		269.55	230.45	46.09	97.94
			3.00		183.80	316.20	63.24	89.59
			4.00		118.35	381.65	76.33	81.10
			5.00		60.85	439.15	87.83	74.66

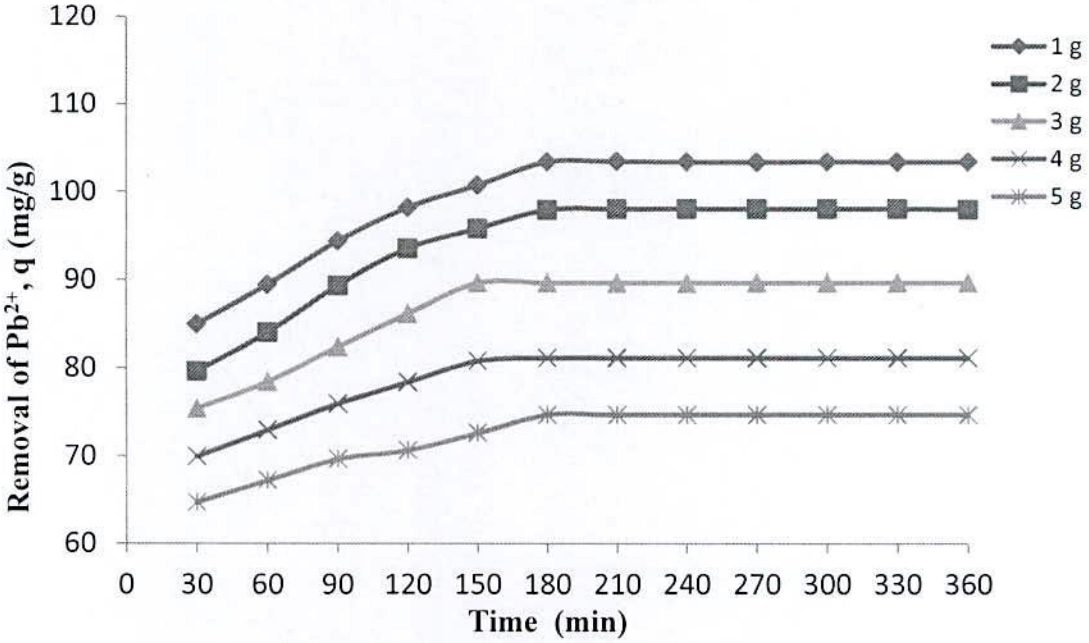


Figure 4.4.7 : **Plot of removal of Pb^{2+} against time to determine equilibrium time of adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 2.42×10^{-3} M and 30 °C.**

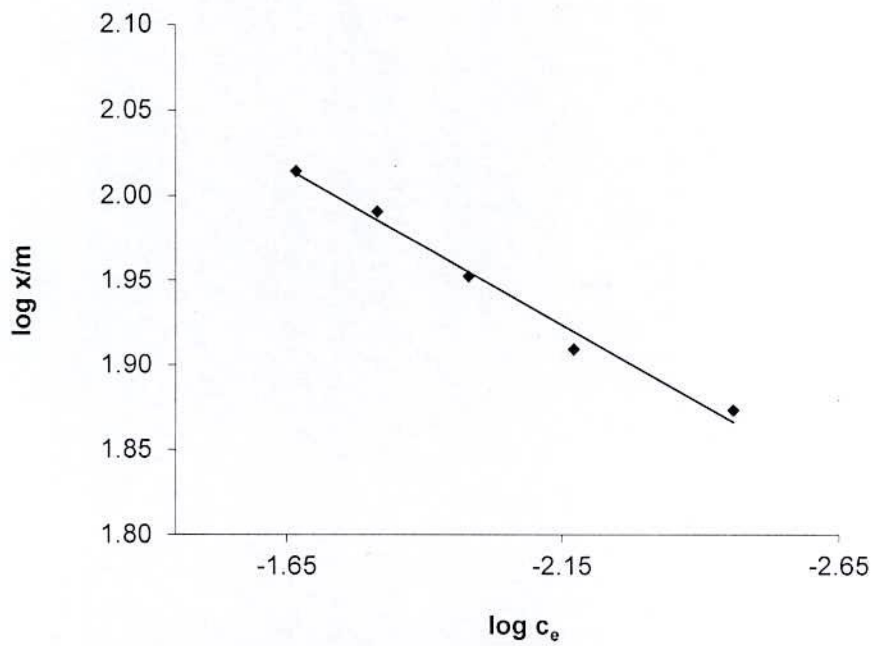


Figure 4.4.8 : **Freundlich isotherm for the adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 2.42×10^{-3} M and 30 °C.**

4.4.6 Estimation of equilibrium time for adsorption of aqueous Pb^{2+} ions onto prepared activated carbon (initial concentration of Pb^{2+}: 2.90×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 2.90×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Pb^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.4.6 and in Figure 4.4.9. It is observed from Table 4.4.6 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 90 to 105 mg/g while the initial concentration of Pb^{2+} is 2.90×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Pb^{2+} ions onto prepared activated carbon is found to be 86.91% within 3 hours and that for minimum is 17.32%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Pb^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.4.10). The value of n is 5.29. It is also seen from the results the isotherm that chemisorption of Pb^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Pb^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Pb^{2+} solution	: 2.90×10^{-3} M

Table 4.4.6 : Determination of equilibrium time for the adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+} ions: 2.90×10^{-3} M and 30 °C.

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	2.90×10^{-3}	496.08	103.92	17.32	88.33
			2.00		400.56	199.44	33.24	84.76
			3.00		314.82	285.18	47.53	80.80
			4.00		239.82	360.18	60.03	76.54
			5.00		167.28	432.72	72.12	73.56
5.7	150	60	1.00	2.90×10^{-3}	486.72	113.28	18.88	96.29
			2.00		391.50	208.50	34.75	88.61
			3.00		293.22	306.78	51.13	86.92
			4.00		211.32	388.68	64.78	82.59
			5.00		149.64	450.36	75.06	76.56
5.7	150	90	1.00	2.90×10^{-3}	482.04	117.96	19.66	100.27
			2.00		379.38	220.62	36.77	93.76
			3.00		280.62	319.38	53.23	90.49
			4.00		193.92	406.08	67.68	86.29
			5.00		131.70	468.30	78.05	79.61
5.7	150	120	1.00	2.90×10^{-3}	474.00	126.00	21.00	107.10
			2.00		365.04	234.96	39.16	99.86
			3.00		264.66	335.34	55.89	95.01
			4.00		180.06	419.94	69.99	89.24
			5.00		111.30	488.70	81.45	83.08
5.7	150	150	1.00	2.90×10^{-3}	467.64	132.36	22.06	112.51
			2.00		348.18	251.82	41.97	107.02
			3.00		257.10	342.90	57.15	97.16
			4.00		162.00	438.00	73.00	93.08
			5.00		95.58	504.42	84.07	85.75
5.7	150	180	1.00	2.90×10^{-3}	466.74	133.26	22.21	113.27
			2.00		346.50	253.50	42.25	107.74
			3.00		240.12	359.88	59.98	101.97
			4.00		151.98	448.02	74.67	95.20
			5.00		78.66	521.34	86.89	88.63
5.7	150	210	1.00	2.90×10^{-3}	466.80	133.20	22.20	113.22
			2.00		346.62	253.38	42.23	107.69
			3.00		240.12	359.88	59.98	101.97
			4.00		152.04	447.96	74.66	95.19
			5.00		78.66	521.34	86.89	88.63

Continued

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	2.90×10^{-3}	466.74	133.26	22.21	113.27
			2.00		346.50	253.50	42.25	107.74
			3.00		240.24	359.76	59.96	101.93
			4.00		151.98	448.02	74.67	95.20
			5.00		78.54	521.46	86.91	88.65
5.7	150	270	1.00	2.90×10^{-3}	466.74	133.26	22.21	113.27
			2.00		346.62	253.38	42.23	107.69
			3.00		240.12	359.88	59.98	101.97
			4.00		151.98	448.02	74.67	95.20
			5.00		78.60	521.40	86.90	88.64
5.7	150	300	1.00	2.90×10^{-3}	466.74	133.26	22.21	113.27
			2.00		346.50	253.50	42.25	107.74
			3.00		240.06	359.94	59.99	101.98
			4.00		151.92	448.08	74.68	95.22
			5.00		78.66	521.34	86.89	88.63
5.7	150	330	1.00	2.90×10^{-3}	466.80	133.20	22.20	113.22
			2.00		346.50	253.50	42.25	107.74
			3.00		240.18	359.82	59.97	101.95
			4.00		151.98	448.02	74.67	95.20
			5.00		78.66	521.34	86.89	88.63
5.7	150	360	1.00	2.90×10^{-3}	466.80	133.20	22.20	113.22
			2.00		346.56	253.44	42.24	107.71
			3.00		240.12	359.88	59.98	101.97
			4.00		151.98	448.02	74.67	95.20
			5.00		78.66	521.34	86.89	88.63

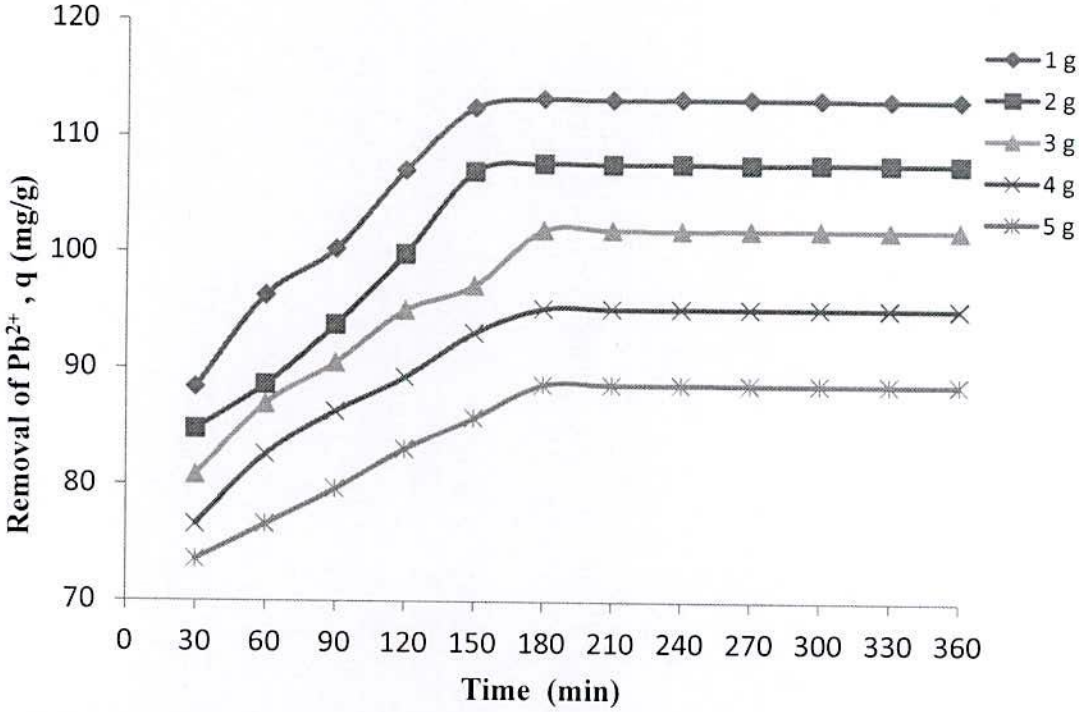


Figure 4.4.9 : **Plot of removal of Pb^{2+} against time to determine equilibrium time of adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 2.90×10^{-3} M and 30 °C.**

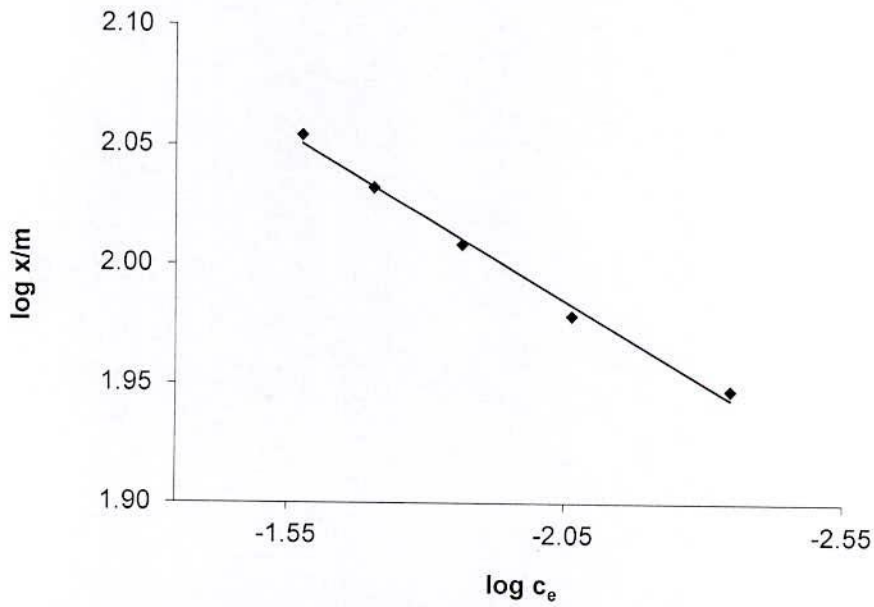


Figure 4.4.10 : **Freundlich isotherm for the adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 2.90×10^{-3} M and 30 °C.**

4.4.7 Estimation of equilibrium time for adsorption of aqueous Pb^{2+} ions onto prepared activated carbon (initial concentration of Pb^{2+}: 3.34×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 3.34×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter $\leq$ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Pb^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.4.7 and in Figure 4.4.11. It is observed from Table 4.4.7 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 98 to 125 mg/g while the initial concentration of Pb^{2+} is 3.34×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Pb^{2+} ions onto prepared activated carbon is found to be 84.32% within 3 hours and that for minimum is 16.45%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Pb^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.4.12). The value of n is 6.02. It is also seen from the results the isotherm that chemisorption of Pb^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter $\leq$ 200 mesh
Volume of aqueous Pb^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Pb^{2+} solution	: 3.34×10^{-3} M

Table 4.4.7 : Determination of equilibrium time for the adsorption of aqueous Pb^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+} ions: 3.34×10^{-3} M and 30 °C.

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	3.34×10^{-3}	584.85	115.15	16.45	97.88
			2.00		476.00	224.00	32.00	95.20
			3.00		377.72	322.28	46.04	91.31
			4.00		288.40	411.60	58.80	87.47
			5.00		216.65	483.35	69.05	82.17
5.7	150	60	1.00	3.34×10^{-3}	576.47	123.53	17.65	105.00
			2.00		461.09	238.91	34.13	101.54
			3.00		350.91	349.09	49.87	98.91
			4.00		258.58	441.42	63.06	93.80
			5.00		188.44	511.56	73.08	86.97
5.7	150	90	1.00	3.34×10^{-3}	568.71	131.29	18.76	111.60
			2.00		446.67	253.33	36.19	107.67
			3.00		335.93	364.07	52.01	103.15
			4.00		238.77	461.23	65.89	98.01
			5.00		166.67	533.33	76.19	90.67
5.7	150	120	1.00	3.34×10^{-3}	561.26	138.74	19.82	117.93
			2.00		428.19	271.81	38.83	115.52
			3.00		318.22	381.78	54.54	108.17
			4.00		220.78	479.22	68.46	101.83
			5.00		142.31	557.69	79.67	94.81
5.7	150	150	1.00	3.34×10^{-3}	556.22	143.78	20.54	122.21
			2.00		419.37	280.63	40.09	119.27
			3.00		301.07	398.93	56.99	113.03
			4.00		202.79	497.21	71.03	105.66
			5.00		123.69	576.31	82.33	97.97
5.7	150	180	1.00	3.34×10^{-3}	549.36	150.64	21.52	128.04
			2.00		410.76	289.24	41.32	122.93
			3.00		289.45	410.55	58.65	116.32
			4.00		193.62	506.38	72.34	107.61
			5.00		109.83	590.17	84.31	100.33
5.7	150	210	1.00	3.34×10^{-3}	549.36	150.64	21.52	128.04
			2.00		410.69	289.31	41.33	122.96
			3.00		289.45	410.55	58.65	116.32
			4.00		193.69	506.31	72.33	107.59
			5.00		109.83	590.17	84.31	100.33

Continued

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	3.34×10^{-3}	549.29	150.71	21.53	128.10
			2.00		410.76	289.24	41.32	122.93
			3.00		289.45	410.55	58.65	116.32
			4.00		193.69	506.31	72.33	107.59
			5.00		109.76	590.24	84.32	100.34
5.7	150	270	1.00	3.34×10^{-3}	549.36	150.64	21.52	128.04
			2.00		410.76	289.24	41.32	122.93
			3.00		289.38	410.62	58.66	116.34
			4.00		193.62	506.38	72.34	107.61
			5.00		109.83	590.17	84.31	100.33
5.7	150	300	1.00	3.34×10^{-3}	549.36	150.64	21.52	128.04
			2.00		410.69	289.31	41.33	122.96
			3.00		289.45	410.55	58.65	116.32
			4.00		193.62	506.38	72.34	107.61
			5.00		109.83	590.17	84.31	100.33
5.7	150	330	1.00	3.34×10^{-3}	549.29	150.71	21.53	128.10
			2.00		410.76	289.24	41.32	122.93
			3.00		289.38	410.62	58.66	116.34
			4.00		193.62	506.38	72.34	107.61
			5.00		109.76	590.24	84.32	100.34
5.7	150	360	1.00	3.34×10^{-3}	549.36	150.64	21.52	128.04
			2.00		410.76	289.24	41.32	122.93
			3.00		289.45	410.55	58.65	116.32
			4.00		193.69	506.31	72.33	107.59
			5.00		109.76	590.24	84.32	100.34

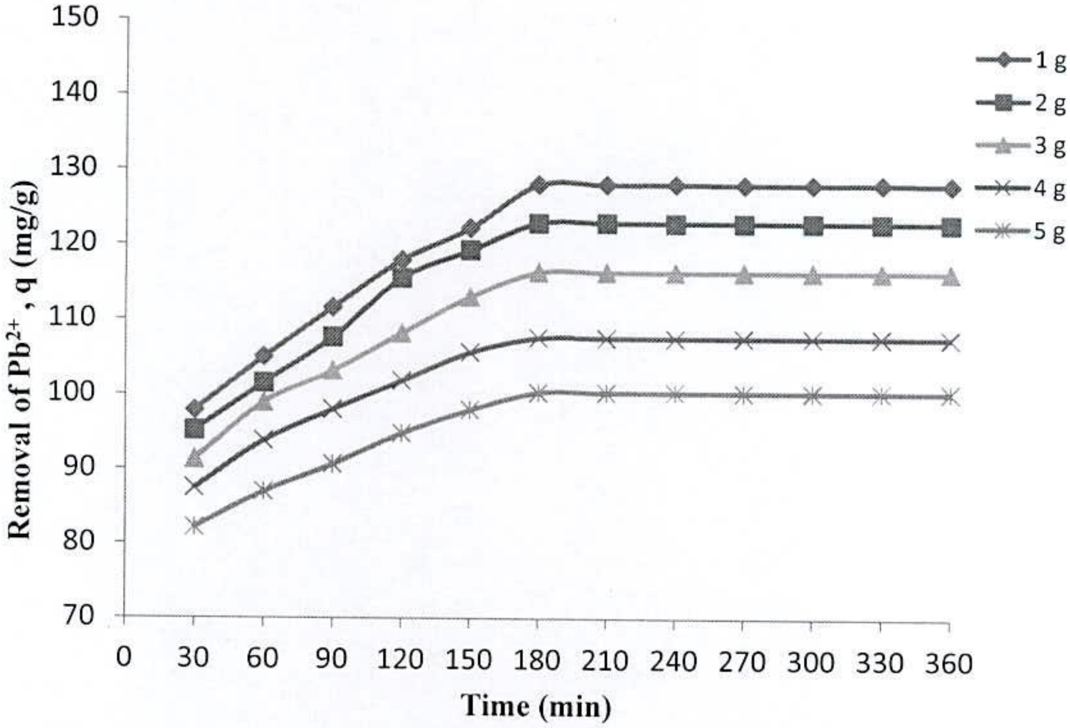


Figure 4.4.11 : **Plot of removal of Pb^{2+} against time to determine equilibrium time of adsorption of Pb^{2+} on activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 3.34×10^{-3} M and 30 °C.**

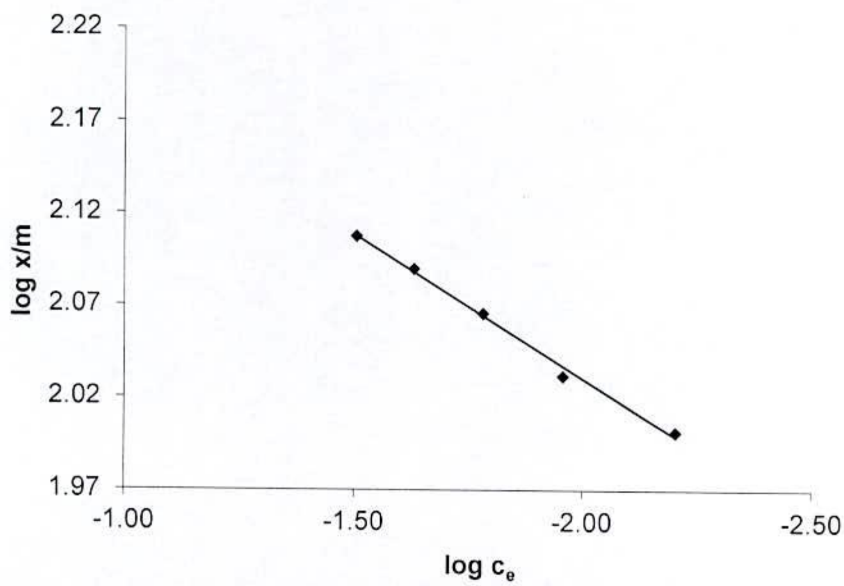


Figure 4.4.12 : **Freundlich isotherm for the adsorption of Pb^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Pb^{2+}: 2.90×10^{-3} M and 30 °C.**

4.4.8 Quantification of the adsorption isotherm data

The linear form of Freundlich equation, which was also applied for the adsorption of Pb^{2+} is given as:

$$log\frac{x}{m} = logk + \frac{1}{n}logC_e$$

Where, k is roughly an indicator of the adsorption capacity and 1/n the adsorption intensity. In general, as the k value increases the adsorption capacity of the adsorbent for a given dye increases. The magnitude of the exponent 1/n gives an indication of the favorability of adsorption. Values, n>1 represent favorable condition for adsorption. Linear plots of $log\frac{x}{m}$ vs. log Ce (Figure 4.4.4, 4.4.6, 4.4.8, 4.4.10 and 4.4.12) show that the adsorption of activated carbon onto also follow the Freundlich isotherm. Data for the Freundlich isotherm are shown in Table 4.4.8. Values of k and n were calculated from the intercepts and slopes of the plots and are listed in Table 4.4.9. The results suggest that the activated carbon is favorably adsorbed by activated carbons prepared from rice husk.

Table 4.4.8 : Freundlich constants for the adsorption of aqueous Pb^{2+} ions onto prepared activated carbon

Amount of carbon (g)	Initial Conc. (mg/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)	Equilibrium Conc., C_e (M)	log C_e	log x/m
1.00	3.34×10^{-3}	549.36	150.64	21.52	128.04	0.03	-1.51	2.11
2.00		410.76	289.24	41.32	122.93	0.02	-1.63	2.09
3.00		289.45	410.55	58.65	116.32	0.02	-1.78	2.07
4.00		193.62	506.38	72.34	107.61	0.01	-1.96	2.03
5.00		109.83	590.17	84.31	100.33	0.01	-2.21	2.00
1.00	2.90×10^{-3}	466.74	133.26	22.21	113.27	0.03	-1.58	2.05
2.00		346.50	253.50	42.25	107.74	0.02	-1.71	2.03
3.00		240.12	359.88	59.98	101.97	0.01	-1.87	2.01
4.00		151.98	448.02	74.67	95.20	0.01	-2.06	1.98
5.00		78.66	521.34	86.89	88.63	0.00	-2.35	1.95
1.00	2.42×10^{-3}	378.40	121.60	24.32	103.36	0.02	-1.67	2.01
2.00		269.65	230.35	46.07	97.90	0.02	-1.82	1.99
3.00		183.77	316.23	63.25	89.60	0.01	-1.98	1.95
4.00		118.34	381.66	76.33	81.10	0.01	-2.17	1.91
5.00		60.85	439.15	87.83	74.66	0.00	-2.46	1.87

Continued

Amount of carbon (g)	Initial Conc. (mg/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)	Equilibrium Conc., C_e (M)	log C_e	log x/m
1.00	1.93×10^{-3}	299.80	100.20	25.05	85.17	0.02	-1.77	1.93
2.00		210.86	189.14	47.29	80.39	0.01	-1.92	1.91
3.00		155.41	244.59	61.15	69.30	0.01	-2.05	1.84
4.00		88.08	311.92	77.98	66.28	0.01	-2.30	1.82
5.00		47.20	352.80	88.20	59.98	0.00	-2.57	1.78
1.00	1.48×10^{-3}	221.64	78.36	26.12	66.61	0.01	-1.90	1.82
2.00		154.98	145.02	48.34	61.63	0.01	-2.06	1.79
3.00		104.39	195.61	65.20	55.42	0.01	-2.23	1.74
4.00		55.32	244.68	81.56	51.99	0.00	-2.50	1.72
5.00		27.03	272.97	90.99	46.40	0.00	-2.81	1.67

Table 4.4.9 : Freundlich constants for the adsorption of aqueous Pb^{2+} ions onto prepared activated carbon

Initial Conc. of Pb^{2+} (mol/L)	Value of k	Value of n
1.48×10^{-3}	2.13	6.02
1.93×10^{-3}	2.25	5.29
2.42×10^{-3}	2.32	5.43
2.90×10^{-3}	2.26	7.25
3.34×10^{-3}	2.34	6.45

4.5 Adsorption study of Hg on prepared activated carbon

Mercury discharged into the water bodies from various industrial sources. There are several methods available for the removal of metal ions from wastewater. Among them, the adsorption process provides an attractive alternative for the treatment of wastewater, especially if the adsorbent is inexpensive and does not require additional pretreatment before its application. The adsorption characteristics of Hg ions onto prepared activated carbon from rice husk are discussed. Batch adsorption experiments were carried out under varying experimental conditions such as, particle size of adsorbent, contact time, initial concentration of Hg solution.

Five series of experiments varying the amount of activated carbon were carried out to study the effects of initial Hg concentration, pH and shaking frequency on the removal of Hg from aqueous solution. The equilibrium time for adsorption of aqueous Hg^{2+} ions onto prepared activated carbon is 3 hours which is almost same as found for methylene blue and Pb^{2+} discussed in section 4.3 and 4.4 respectively. The adsorption processes were performed at different pH and shaking frequency. The pH and shaking frequency at which maximum adsorption are 5.7 and 150 rev/min respectively. The attained data are presented as the plots of removal of Hg (mg/g), q, against adsorption time, t. The percentage removal of Hg^{2+} at equilibrium is also important, for different adsorption conditions. The removal of Hg^{2+}, q, and percentage removal of Hg^{2+} used are defined as follows:

Removal of Hg^{2+}: $q = \frac{(C_0 - C)V}{w}$

Percentage removal of Hg^{2+}: $\% \, Removal = \frac{(C_0 - C)}{C_0} \times 100$

$$= \frac{qw}{VC_0} \times 100$$

Where, C and C_0 are the Hg^{2+} concentrations (mg/g) at $t=t$ and $t=0$, respectively; V is the volume of aqueous solution (L); and w is the mass of activated carbon used (g).

4.5.1 Effect of pH on adsorption of Hg onto prepared activated carbon

The adsorption experiments were carried out at different pH to optimize the adsorption capabilities of prepared activated carbon.

The experiment has been carried out under the following experimental condition:

pH of the solutions	: 3.0, 4.0, 5.0, 5.7 and 6.0
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter $\leq$ 200 mesh
Volume of aqueous Hg^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Hg^{2+} solution	: 700 mg/L

The results representing the effect of pH are tabulated in Table 4.5.1 and Figure 4.5.1. The results are close to those of other reports [115−117]. The experimented pH values are 3.0, 4.0, 5.0, 5.7 and 6.0. To fix the pH value 5.7 more experiments were done at pH 5.1, 5.2, 5.3, 5.4, 5.5, 5.6, 5.7, 5.8 and 6.0. It was seen from the results that pH value 5.7 showed the maximum adsorption almost in all cases. The removal of Hg^{2+} ions are increased with an increase of pH reaching the maximum at pH 5.7 it decreased again. No effect of initial concentration of Hg^{2+} on pH was observed. The effect of pH observed here can probably be explained by the ion-exchange mechanism proposed by Daifullah *et al.* [118].

$$Hg^{2+} + 2(\text{-SiOH}) \Leftrightarrow Hg(\text{—SiO})_2 + 2H^+$$

where Hg^{2+}, —SiOH, and H+ are Hg ion, silanol group, and proton, respectively.

As discussed in section 4.3 that our prepared activated carbon contains 39.47% ash (Table 4.2.4) and ash comprise a significant amount of SiO_2 and the value is 92% (Table 4.2.3). The silanol group on the surface of activated carbon was produced through the hydrolysis of silicon oxide and the Hg^{2+} ion in solution substituted the proton of the silanol group [118]. At low pH, the surface of the activated carbon was surrounded by proton (H^+), which prevented the Hg^{2+} from approaching the silanol group and, consequently, reduced the adsorption of Hg ion. With increasing pH value, electrostatic repulsion decreased due to the reduction of positive charge density around the silanol group, thus resulting in enhancement of Hg adsorption [116].

Table 4.5.1 : Effect of pH on adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at shaking frequency: 150 rev/min, initial concentration of Hg^{2+} ions: 3.34×10^{-3} M and 30 °C.

pH	Shacking Frequency (rev/min)	Amount of carbon (g)	Initial Concentration (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
3	150	1.00	3.34×10^{-3}	614.32	85.68	12.24	72.83
		2.00		503.16	196.84	28.12	83.66
		3.00		406.56	293.44	41.92	83.14
		4.00		314.65	385.35	55.05	81.89
		5.00		257.67	442.33	63.19	75.20
4	150	1.00	2.98×10^{-3}	590.52	109.48	15.64	93.06
		2.00		467.32	232.68	33.24	98.89
		3.00		378.98	321.02	45.86	90.96
		4.00		294.91	405.09	57.87	86.08
		5.00		245.07	454.93	64.99	77.34
5	150	1.00	2.42×10^{-3}	580.86	119.14	17.02	101.27
		2.00		460.53	239.47	34.21	101.77
		3.00		368.06	331.94	47.42	94.05
		4.00		286.02	413.98	59.14	87.97
		5.00		228.34	471.66	67.38	80.18
5.7	150	1.00	1.93×10^{-3}	556.77	143.23	20.46	121.74
		2.00		437.15	262.85	37.55	111.71
		3.00		347.20	352.80	50.40	99.96
		4.00		257.32	442.68	63.24	94.07
		5.00		191.94	508.06	72.58	86.37
6	150	1.00	1.48×10^{-3}	581.07	118.93	16.99	101.09
		2.00		481.53	218.47	31.21	92.85
		3.00		391.16	308.84	44.12	87.50
		4.00		285.32	414.68	59.24	88.12

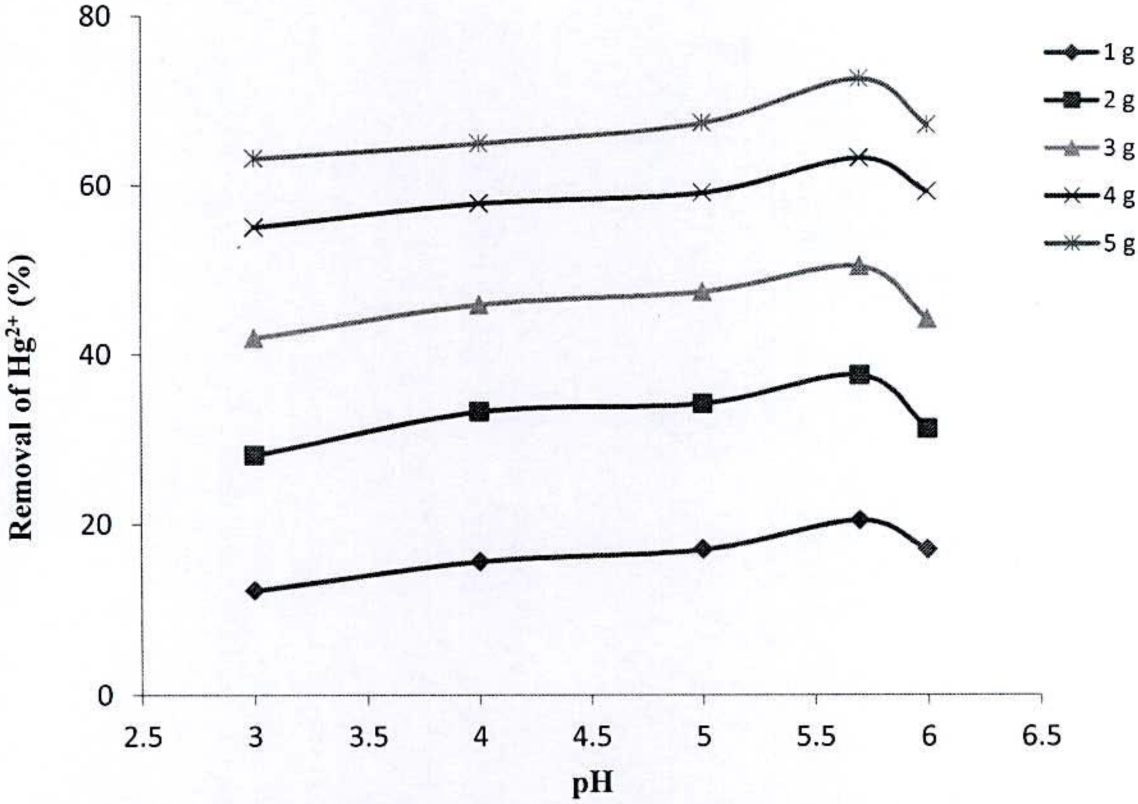


Figure 4.5.1 : **Effect of pH on adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at shaking frequency: 150 rev/min, initial concentration of Hg^{2+} ions: 3.34×10^{-3} M and 30 °C.**

4.5.2 Effect of shaking frequency on adsorption of Hg onto prepared activated carbon

The adsorption experiments were carried out at different shaking frequency to optimize the adsorption capabilities of prepared activated carbon.

The experiment has been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Hg^{2+} solution	: 850 mL
Rate of shaking	: 50, 100, 150 and 200 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Hg^{2+} solution	: 700 mg/L

The results showing the effect of shaking frequency are tabulated in Table 4.5.2 and Figure 4.5.2. The results are close to those of other reports [116]. The experimented shaking frequencies are 50, 100, 150 and 200 rev/min. It was seen from the results that shaking frequency value of 150 rev/min showed the maximum adsorption almost in all cases. The removal of Hg^{2+} ions are increased with an increase of shaking frequency reaching the maximum value of 150 rev/min it decreased again. No effect of initial concentration of Hg^{2+} on shaking frequency was observed. It is seen that the higher the shaking frequency, the faster the removal and higher removal at equilibrium. Wong *et al.* [116] reported the same tendency for the system of Hg^{2+} tartaric acid modified rice hull. The reason for this is that when the shaking frequency is increased, the rate of mass transfer of Hg ion through an artificial liquid film outside activated carbon particles is increased. It is also noted from Figure 4.5.2 that when the shaking frequency is over 150 rev/min, the effect of stroke speed becomes insignificant. This implies the resistance of the mass transfer of Hg ion through the liquid film becomes null.

Table 4.5.2 : **Effect of shaking frequency on adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at pH: 5.7, initial concentration of Hg^{2+} ions: 3.34×10^{-3} M and 30 °C.**

pH	Shacking Frequency (rev/min)	Amount of carbon (g)	Initial Concentration (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	50	1.00	3.34×10^{-3}	596.54	103.46	14.78	87.94
		2.00		464.31	235.69	33.67	100.17
		3.00		341.39	358.61	51.23	101.61
		4.00		262.43	437.57	62.51	92.98
		5.00		186.13	513.87	73.41	87.36
5.7	100	1.00	3.34×10^{-3}	568.75	131.25	18.75	111.56
		2.00		438.69	261.31	37.33	111.06
		3.00		312.62	387.38	55.34	109.76
		4.00		218.89	481.11	68.73	102.24
		5.00		133.28	566.72	80.96	96.34
5.7	150	1.00	3.34×10^{-3}	556.77	143.23	20.46	121.74
		2.00		437.15	262.85	37.55	111.71
		3.00		347.20	352.80	50.40	99.96
		4.00		257.32	442.68	63.24	94.07
		5.00		191.94	508.06	72.58	86.37
5.7	200	1.00	3.34×10^{-3}	556.29	143.71	20.53	122.15
		2.00		419.09	280.91	40.13	119.39
		3.00		297.78	402.22	57.46	113.96
		4.00		201.74	498.26	71.18	105.88
		5.00		116.97	583.03	83.29	99.12

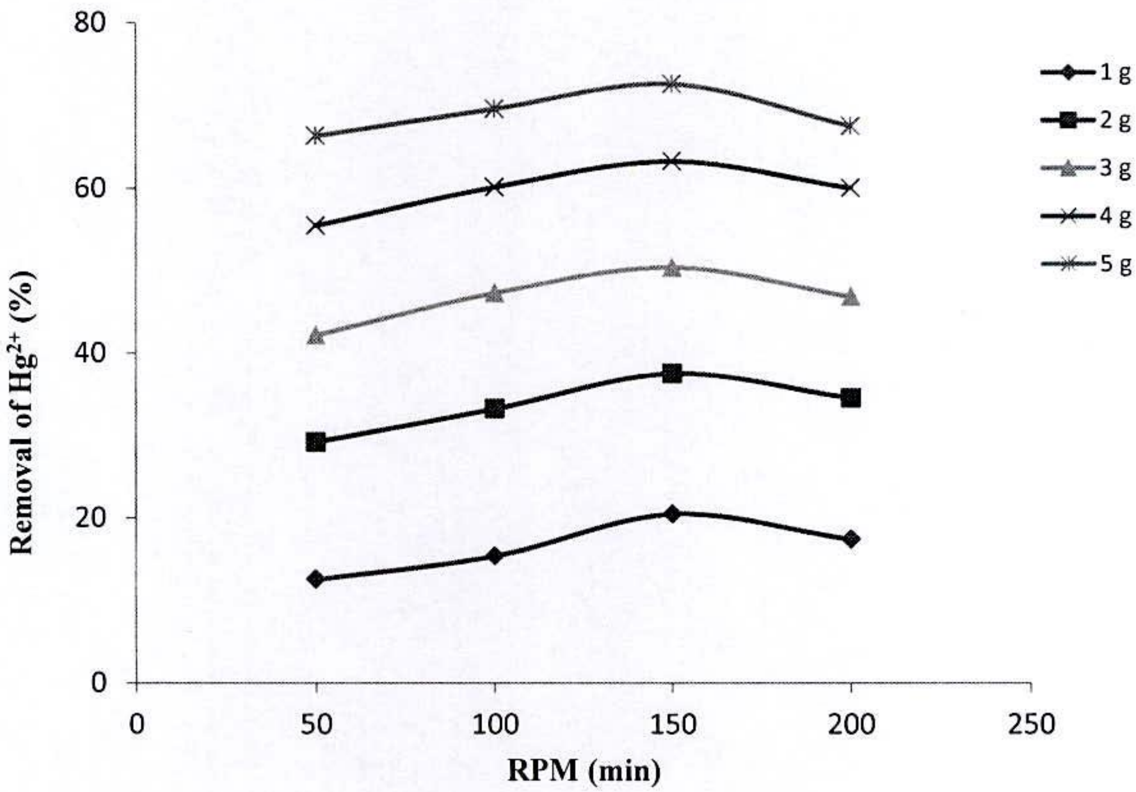


Figure 4.5.2 : **Effect of shaking frequency on adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at pH: 5.7, initial concentration of Hg^{2+} ions: 3.34×10^{-3} M and 30 °C.**

4.5.3 Estimation of equilibrium time for adsorption of aqueous Hg^{2+} ions onto prepared activated carbon (initial concentration of Hg^{2+}: 1.48×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 1.48×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Hg^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.5.3 and in Figure 4.5.3. It is observed from Table 4.5.3 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 43 to 55 mg/g while the initial concentration of Hg^{2+} is 1.48×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Hg^{2+} ions onto prepared activated carbon is found to be 83.61% within 3 hours and that for minimum is 21.33%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Hg^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.5.4). The value of n is 2.83. It is also seen from the results the isotherm that chemisorption of Hg^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Hg^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Hg^{2+} solution	: 1.48×10^{-3} M

Table 4.5.3 : **Determination of equilibrium time for the adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+} ions: 1.48×10^{-3} M and 30 °C.**

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	1.48×10^{-3}	236.01	63.99	21.33	54.39
			2.00		185.01	114.99	38.33	48.87
			3.00		136.77	163.23	54.41	46.25
			4.00		99.40	200.60	66.87	42.63
			5.00		77.64	222.36	74.12	37.80
5.7	150	60	1.00	1.48×10^{-3}	233.67	66.33	22.11	56.38
			2.00		179.49	120.51	40.17	51.22
			3.00		131.07	168.93	56.31	47.86
			4.00		93.43	206.57	68.86	43.90
			5.00		71.94	228.06	76.02	38.77
5.7	150	90	1.00	1.48×10^{-3}	231.33	68.67	22.89	58.37
			2.00		173.31	126.69	42.23	53.84
			3.00		124.65	175.35	58.45	49.68
			4.00		87.07	212.93	70.98	45.25
			5.00		68.94	231.06	77.02	39.28
5.7	150	120	1.00	1.48×10^{-3}	225.18	74.82	24.94	63.60
			2.00		167.72	132.28	44.09	56.22
			3.00		117.75	182.25	60.75	51.64
			4.00		78.64	221.36	73.79	47.04
			5.00		65.16	234.84	78.28	39.92
5.7	150	150	1.00	1.48×10^{-3}	220.50	79.50	26.50	67.58
			2.00		159.80	140.20	46.73	59.58
			3.00		110.16	189.84	63.28	53.79
			4.00		75.46	224.54	74.85	47.72
			5.00		57.81	242.19	80.73	41.17
5.7	150	180	1.00	1.48×10^{-3}	216.21	83.79	27.93	71.22
			2.00		152.36	147.64	49.21	62.75
			3.00		106.38	193.62	64.54	54.86
			4.00		74.62	225.38	75.13	47.89
			5.00		49.32	250.68	83.56	42.62
5.7	150	210	1.00	1.48×10^{-3}	216.21	83.79	27.93	71.22
			2.00		152.33	147.67	49.22	62.76
			3.00		106.41	193.59	64.53	54.85
			4.00		74.68	225.32	75.11	47.88
			5.00		49.21	250.79	83.60	42.63

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	1.48×10^{-3}	216.06	83.94	27.98	71.35
			2.00		152.36	147.64	49.21	62.75
			3.00		106.41	193.59	64.53	54.85
			4.00		74.62	225.38	75.13	47.89
			5.00		49.17	250.83	83.61	42.64
5.7	150	270	1.00	1.48×10^{-3}	216.09	83.91	27.97	71.32
			2.00		152.39	147.61	49.20	62.73
			3.00		106.38	193.62	64.54	54.86
			4.00		74.68	225.32	75.11	47.88
			5.00		49.20	250.80	83.60	42.64
5.7	150	300	1.00	1.48×10^{-3}	216.21	83.79	27.93	71.22
			2.00		152.36	147.64	49.21	62.75
			3.00		106.38	193.62	64.54	54.86
			4.00		74.62	225.38	75.13	47.89
			5.00		49.20	250.80	83.60	42.64
5.7	150	330	1.00	1.48×10^{-3}	216.12	83.88	27.96	71.30
			2.00		152.39	147.61	49.20	62.73
			3.00		106.38	193.62	64.54	54.86
			4.00		74.62	225.38	75.13	47.89
			5.00		49.20	250.80	83.60	42.64
5.7	150	360	1.00	1.48×10^{-3}	216.21	83.79	27.93	71.22
			2.00		152.39	147.61	49.20	62.73
			3.00		106.41	193.59	64.53	54.85
			4.00		74.65	225.35	75.12	47.89
			5.00		49.26	250.74	83.58	42.63

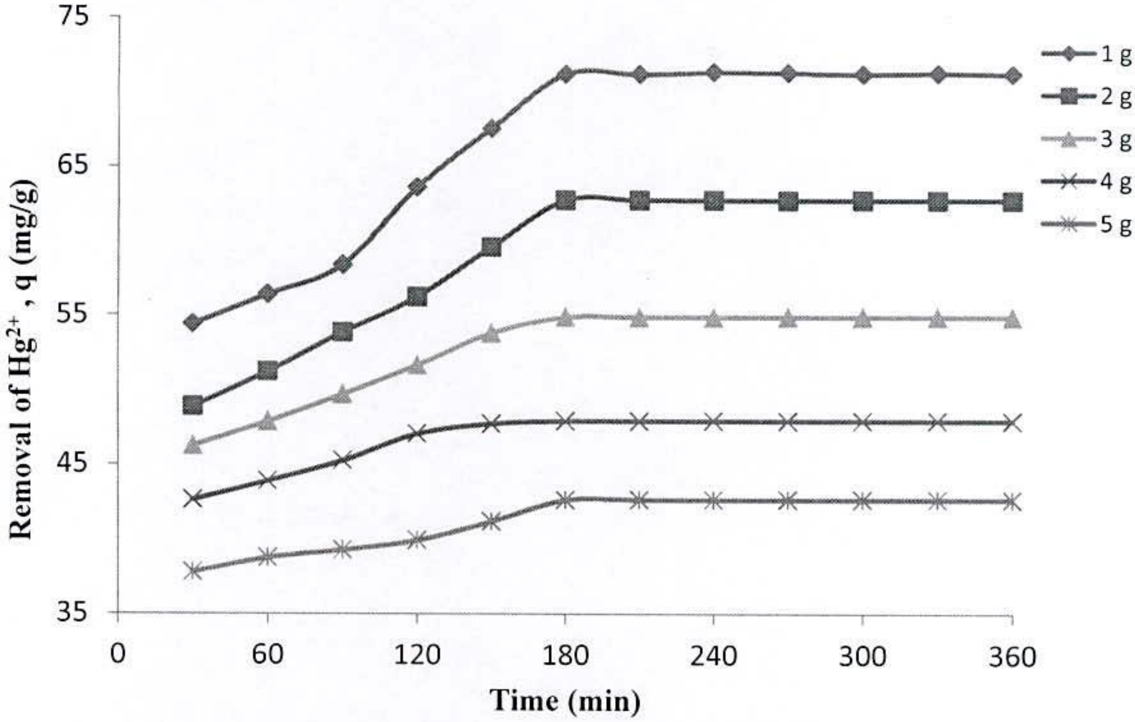


Figure 4.5.3 : Plot of removal of Hg^{2+} against time to determine equilibrium time of adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 1.48×10^{-3} M and 30 °C.

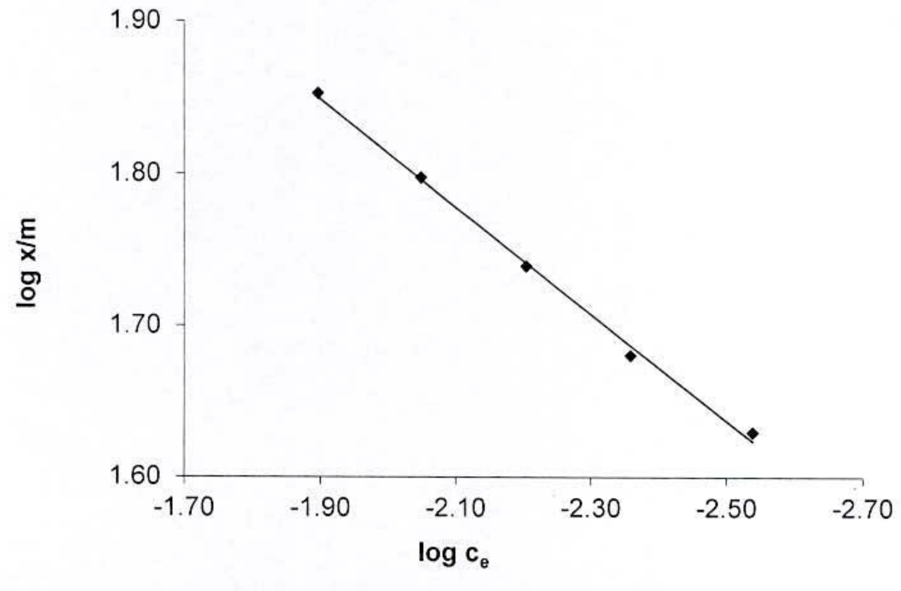


Figure 4.5.4 : Freundlich isotherm for the adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 1.48×10^{-3} M and 30 °C.

4.5.4 Estimation of equilibrium time for adsorption of aqueous Hg^{2+} ions onto prepared activated carbon (initial concentration of Hg^{2+}: 1.93×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 1.93×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Hg^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.5.4 and in Figure 4.5.5. It is observed from Table 4.5.4 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 50 to 70 mg/g while the initial concentration of Hg^{2+} is 1.93×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Hg^{2+} ions onto prepared activated carbon is found to be 80.59% within 3 hours and that for minimum is 19.02%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Hg^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.5.6). The value of n is 2.43. It is also seen from the results the isotherm that chemisorption of Hg^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Hg^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Hg^{2+} solution	: 1.93×10^{-3} M

Table 4.5.4 : **Determination of equilibrium time for the adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+} ions: 1.93×10^{-3} M and 30 °C.**

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	1.93×10^{-3}	323.92	76.08	19.02	64.67
			2.00		260.96	139.04	34.76	59.09
			3.00		210.59	189.41	47.35	53.67
			4.00		164.78	235.22	58.81	49.98
			5.00		116.04	283.96	70.99	48.27
5.7	150	60	1.00	1.93×10^{-3}	321.60	78.40	19.60	66.64
			2.00		252.88	147.12	36.78	62.53
			3.00		202.19	197.81	49.45	56.05
			4.00		164.82	235.18	58.80	49.98
			5.00		108.08	291.92	72.98	49.63
5.7	150	90	1.00	1.93×10^{-3}	317.16	82.84	20.71	70.41
			2.00		246.88	153.12	38.28	65.08
			3.00		191.41	208.59	52.15	59.10
			4.00		153.50	246.50	61.63	52.38
			5.00		103.28	296.72	74.18	50.44
5.7	150	120	1.00	1.93×10^{-3}	311.00	89.00	22.25	75.65
			2.00		238.64	161.36	40.34	68.58
			3.00		182.85	217.15	54.29	61.53
			4.00		140.01	259.99	65.00	55.25
			5.00		93.68	306.32	76.58	52.07
5.7	150	150	1.00	1.93×10^{-3}	306.75	93.25	23.31	79.26
			2.00		232.60	167.40	41.85	71.15
			3.00		170.45	229.55	57.39	65.04
			4.00		129.73	270.27	67.57	57.43
			5.00		85.20	314.80	78.70	53.52
5.7	150	180	1.00	1.93×10^{-3}	303.39	96.61	24.15	82.12
			2.00		225.49	174.51	43.63	74.17
			3.00		160.33	239.67	59.92	67.91
			4.00		117.69	282.31	70.58	59.99
			5.00		77.68	322.32	80.58	54.79
5.7	150	210	1.00	1.93×10^{-3}	303.43	96.57	24.14	82.09
			2.00		225.49	174.51	43.63	74.17
			3.00		160.33	239.67	59.92	67.91
			4.00		117.54	282.46	70.62	60.02
			5.00		77.68	322.32	80.58	54.79

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	1.93×10^{-3}	303.39	96.61	24.15	82.12
			2.00		225.49	174.51	43.63	74.17
			3.00		160.29	239.71	59.93	67.92
			4.00		117.50	282.50	70.63	60.03
			5.00		77.64	322.36	80.59	54.80
5.7	150	270	1.00	1.93×10^{-3}	303.39	96.61	24.15	82.12
			2.00		225.49	174.51	43.63	74.17
			3.00		160.33	239.67	59.92	67.91
			4.00		117.54	282.46	70.62	60.02
			5.00		77.68	322.32	80.58	54.80
5.7	150	300	1.00	1.93×10^{-3}	303.39	96.61	24.15	82.12
			2.00		225.49	174.51	43.63	74.17
			3.00		160.33	239.67	59.92	67.91
			4.00		117.54	282.46	70.62	60.02
			5.00		77.68	322.32	80.58	54.80
5.7	150	330	1.00	1.93×10^{-3}	303.43	96.57	24.14	82.09
			2.00		225.49	174.51	43.63	74.17
			3.00		160.29	239.71	59.93	67.92
			4.00		117.54	282.46	70.62	60.02
			5.00		77.68	322.32	80.58	54.80
5.7	150	360	1.00	1.93×10^{-3}	303.43	96.57	24.14	82.09
			2.00		225.49	174.51	43.63	74.17
			3.00		160.29	239.71	59.93	67.92
			4.00		117.61	282.39	70.60	60.01
			5.00		77.64	322.36	80.59	54.80

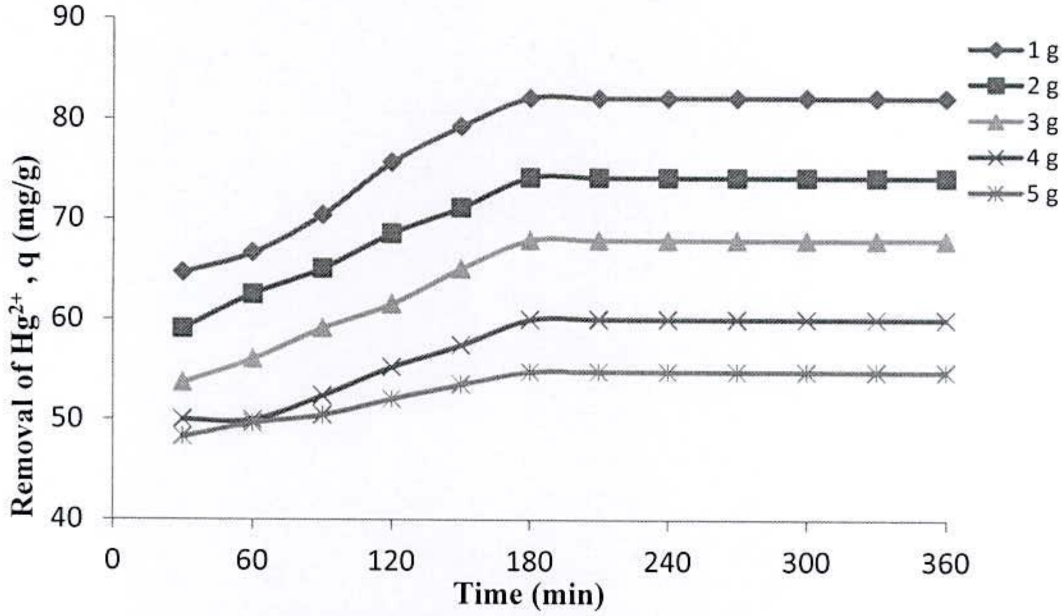


Figure 4.5.5 : **Plot of removal of Hg^{2+} against time to determine equilibrium time of adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 1.93×10^{-3} M and 30 °C.**

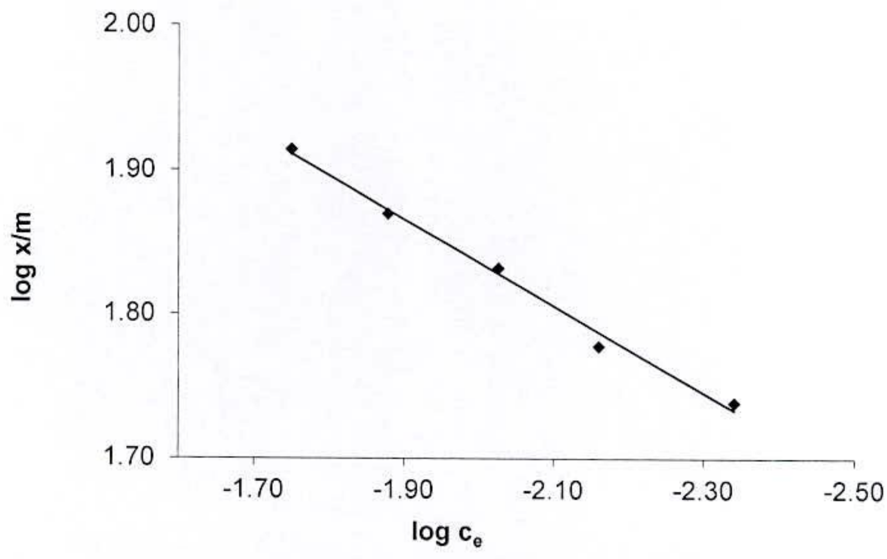


Figure 4.5.6 : **Freundlich isotherm for the adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 1.93×10^{-3} M and 30 °C.**

4.5.5 Estimation of equilibrium time for adsorption of aqueous Hg^{2+} ions onto prepared activated carbon (initial concentration of Hg^{2+}: 2.42×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 2.42×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Hg^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.5.5 and in Figure 4.5.7. It is observed from Table 4.5.5 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 62 to 88 mg/g while the initial concentration of Hg^{2+} is 2.42×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Hg^{2+} ions onto prepared activated carbon is found to be 81.99% within 3 hours and that for minimum is 17.74%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Hg^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.5.8). The value of n is 4.87. It is also seen from the results the isotherm that chemisorption of Hg^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Hg^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Hg^{2+} solution	: 2.42×10^{-3} M

Table 4.5.5: **Determination of equilibrium time for the adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+} ions: 2.42×10^{-3} M and 30 °C.**

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	2.42×10^{-3}	411.30	88.70	17.74	75.40
			2.00		330.90	169.10	33.82	71.87
			3.00		268.61	231.39	46.28	65.56
			4.00		205.65	294.35	58.87	62.55
			5.00		152.90	347.10	69.42	59.01
5.7	150	60	1.00	2.42×10^{-3}	406.25	93.75	18.75	79.69
			2.00		327.40	172.60	34.52	73.36
			3.00		258.61	241.39	48.28	68.39
			4.00		196.30	303.70	60.74	64.54
			5.00		142.90	357.10	71.42	60.71
5.7	150	90	1.00	2.42×10^{-3}	401.80	98.20	19.64	83.47
			2.00		321.10	178.90	35.78	76.03
			3.00		245.31	254.69	50.94	72.16
			4.00		184.75	315.25	63.05	66.99
			5.00		129.60	370.40	74.08	62.97
5.7	150	120	1.00	2.42×10^{-3}	396.60	103.40	20.68	87.89
			2.00		308.61	191.39	38.28	81.34
			3.00		226.16	273.84	54.77	77.59
			4.00		163.45	336.55	67.31	71.52
			5.00		115.50	384.50	76.90	65.37
5.7	150	150	1.00	2.42×10^{-3}	391.65	108.35	21.67	92.10
			2.00		298.66	201.34	40.27	85.57
			3.00		211.16	288.84	57.77	81.84
			4.00		148.45	351.55	70.31	74.71
			5.00		99.95	400.05	80.01	68.01
5.7	150	180	1.00	2.42×10^{-3}	387.25	112.75	22.55	95.84
			2.00		292.51	207.49	41.50	88.18
			3.00		204.86	295.14	59.03	83.62
			4.00		133.40	366.60	73.32	77.90
			5.00		90.05	409.95	81.99	69.69
5.7	150	210	1.00	2.42×10^{-3}	387.25	112.75	22.55	95.84
			2.00		292.46	207.54	41.51	88.20
			3.00		204.86	295.14	59.03	83.62
			4.00		133.45	366.55	73.31	77.89
			5.00		90.05	409.95	81.99	69.69

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	2.42×10^{-3}	387.35	112.65	22.53	95.75
			2.00		292.86	207.14	41.43	88.03
			3.00		204.86	295.14	59.03	83.62
			4.00		133.34	366.67	73.33	77.92
			5.00		90.05	409.95	81.99	69.69
5.7	150	270	1.00	2.42×10^{-3}	387.25	112.75	22.55	95.84
			2.00		292.61	207.39	41.48	88.14
			3.00		204.81	295.19	59.04	83.64
			4.00		133.34	366.67	73.33	77.92
			5.00		90.05	409.95	81.99	69.69
5.7	150	300	1.00	2.42×10^{-3}	387.20	112.80	22.56	95.88
			2.00		292.71	207.29	41.46	88.10
			3.00		204.86	295.14	59.03	83.62
			4.00		133.34	366.67	73.33	77.92
			5.00		90.00	410.01	82.00	69.70
5.7	150	330	1.00	2.42×10^{-3}	387.30	112.70	22.54	95.80
			2.00		292.51	207.49	41.50	88.18
			3.00		204.81	295.19	59.04	83.64
			4.00		133.34	366.67	73.33	77.92
			5.00		90.10	409.91	81.98	69.68
5.7	150	360	1.00	2.42×10^{-3}	387.25	112.75	22.55	95.84
			2.00		292.46	207.54	41.51	88.20
			3.00		204.86	295.14	59.03	83.62
			4.00		133.33	366.67	73.33	77.92
			5.00		90.10	409.91	81.98	69.68

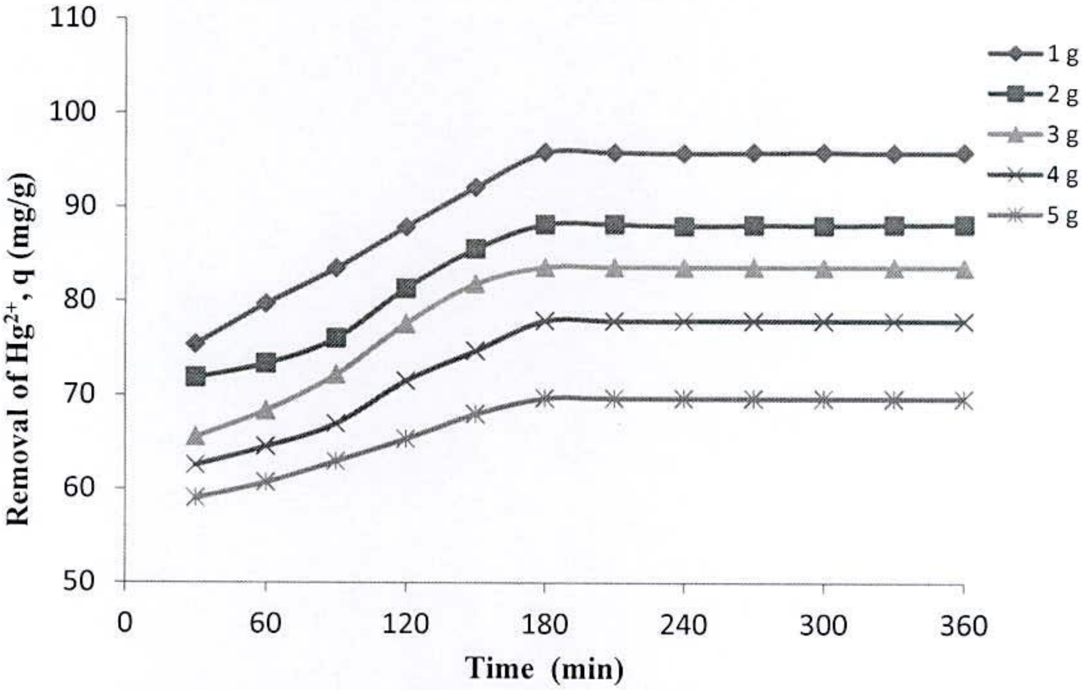


Figure 4.5.7 : **Plot of removal of Hg^{2+} against time to determine equilibrium time of adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 2.42×10^{-3} M and 30 °C.**

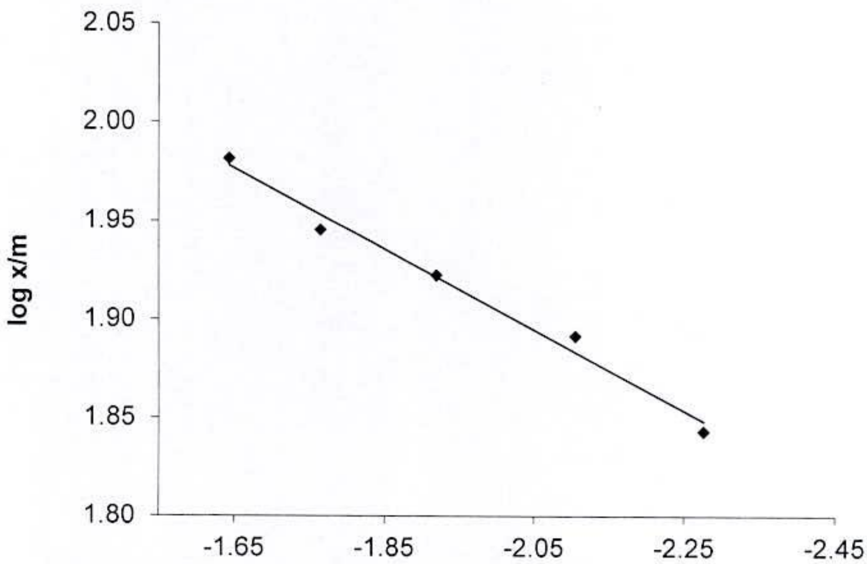


Figure 4.5.8 : **Freundlich isotherm for the adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 2.42×10^{-3} M and 30 °C.**

4.5.6 Estimation of equilibrium time for adsorption of aqueous Hg^{2+} ions onto prepared activated carbon (initial concentration of Hg^{2+}: 2.90×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 2.90×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Hg^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.5.6 and in Figure 4.5.9. It is observed from Table 4.5.6 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 80 to 98 mg/g while the initial concentration of Hg^{2+} is 2.90×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Hg^{2+} ions onto prepared activated carbon is found to be 81.97% within 3 hours and that for minimum is 16.29%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Hg^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.5.10). The value of n is 6.76. It is also seen from the results the isotherm that chemisorption of Hg^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Hg^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Hg^{2+} solution	: 2.90×10^{-3} M

Table 4.5.6 : Determination of equilibrium time for the adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+} ions: 2.90×10^{-3} M and 30 °C.

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	2.90×10^{-3}	502.26	97.74	16.29	83.08
			2.00		418.26	181.74	30.29	77.24
			3.00		334.14	265.86	44.31	75.33
			4.00		256.26	343.74	57.29	73.04
			5.00		189.72	410.28	68.38	69.75
5.7	150	60	1.00	2.90×10^{-3}	496.26	103.74	17.29	88.18
			2.00		409.79	190.21	31.70	80.84
			3.00		324.55	275.45	45.91	78.04
			4.00		236.03	363.97	60.66	77.34
			5.00		175.68	424.32	70.72	72.13
5.7	150	90	1.00	2.98×10^{-3}	491.94	108.06	18.01	91.85
			2.00		398.99	201.01	33.50	85.43
			3.00		310.21	289.79	48.30	82.11
			4.00		220.61	379.39	63.23	80.62
			5.00		155.28	444.72	74.12	75.60
5.7	150	120	1.00	2.90×10^{-3}	484.98	115.02	19.17	97.77
			2.00		387.29	212.71	35.45	90.40
			3.00		297.43	302.57	50.43	85.73
			4.00		203.65	396.35	66.06	84.22
			5.00		131.10	468.90	78.15	79.71
5.7	150	150	1.00	2.90×10^{-3}	482.34	117.66	19.61	100.01
			2.00		377.33	222.67	37.11	94.63
			3.00		278.00	322.00	53.67	91.23
			4.00		184.24	415.76	69.29	88.35
			5.00		119.46	480.54	80.09	81.69
5.7	150	180	1.00	2.90×10^{-3}	476.46	123.54	20.59	105.01
			2.00		367.37	232.63	38.77	98.87
			3.00		263.12	336.88	56.15	95.45
			4.00		176.38	423.62	70.60	90.02
			5.00		108.18	491.82	81.97	83.61
5.7	150	210	1.00	2.90×10^{-3}	476.46	123.54	20.59	105.01
			2.00		367.37	232.63	38.77	98.87
			3.00		263.06	336.94	56.16	95.47
			4.00		175.78	424.22	70.70	90.15
			5.00		108.12	491.88	81.98	83.62

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	2.90×10^{-3}	476.53	123.47	20.58	104.95
			2.00		367.31	232.69	38.78	98.89
			3.00		263.12	336.88	56.15	95.45
			4.00		176.38	423.62	70.60	90.02
			5.00		108.05	491.95	81.99	83.63
5.7	150	270	1.00	2.90×10^{-3}	476.53	123.47	20.58	104.95
			2.00		367.37	232.63	38.77	98.87
			3.00		263.12	336.88	56.15	95.45
			4.00		176.38	423.62	70.60	90.02
			5.00		108.17	491.83	81.97	83.61
5.7	150	300	1.00	2.90×10^{-3}	476.53	123.47	20.58	104.95
			2.00		367.37	232.63	38.77	98.87
			3.00		263.06	336.94	56.16	95.47
			4.00		175.54	424.46	70.74	90.20
			5.00		108.17	491.83	81.97	83.61
5.7	150	330	1.00	2.90×10^{-3}	476.53	123.47	20.58	104.95
			2.00		367.31	232.69	38.78	98.89
			3.00		263.12	336.88	56.15	95.45
			4.00		176.38	423.62	70.60	90.02
			5.00		108.17	491.83	81.97	83.61
5.7	150	360	1.00	2.90×10^{-3}	476.47	123.53	20.59	105.00
			2.00		367.31	232.69	38.78	98.89
			3.00		263.12	336.88	56.15	95.45
			4.00		176.38	423.62	70.60	90.02
			5.00		108.26	491.74	81.96	83.60

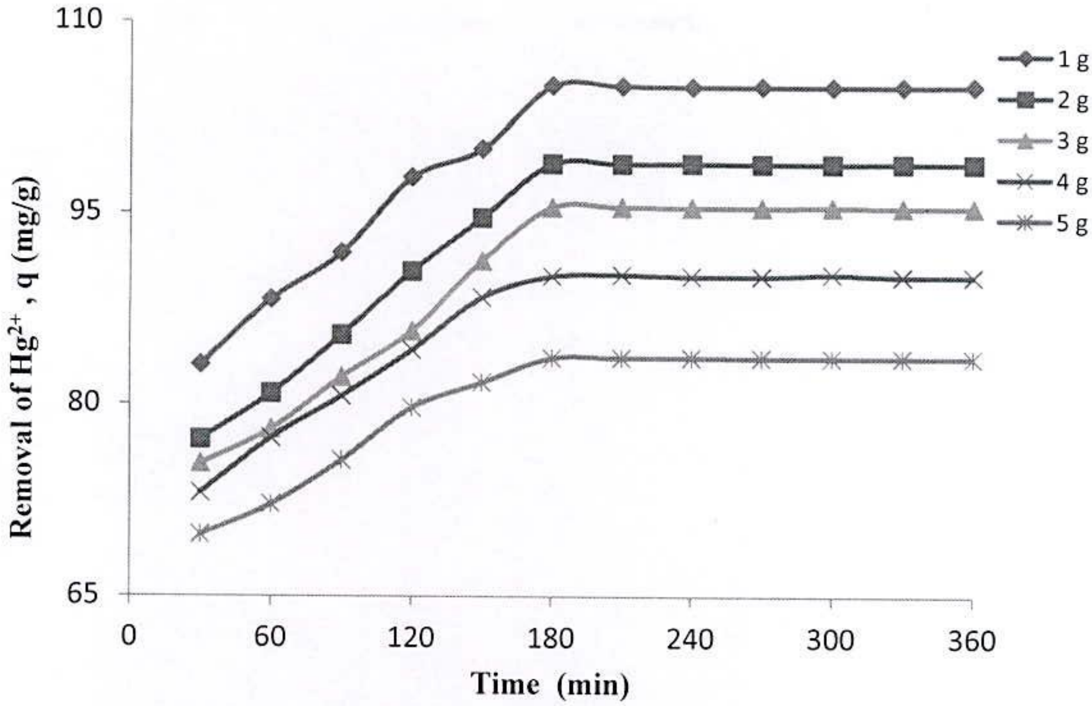


Figure 4.5.9 : Plot of removal of Hg^{2+} against time to determine equilibrium time of adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 2.90×10^{-3} M and 30 °C.

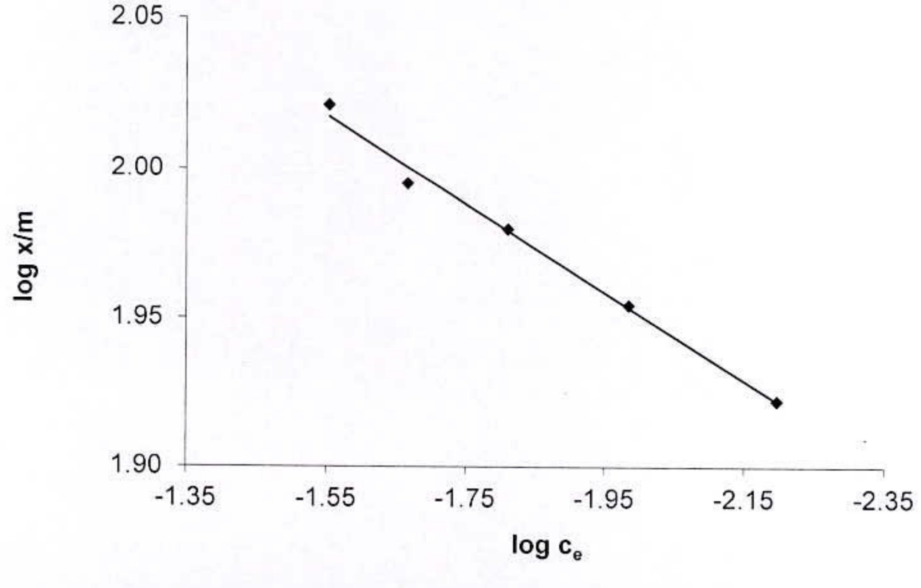


Figure 4.5.10 : Freundlich isotherm for the adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 2.90×10^{-3} M and 30 °C.

4.5.7 Estimation of equilibrium time for adsorption of aqueous Hg^{2+} ions onto prepared activated carbon (initial concentration of Hg^{2+}: 3.34×10^{-3} M, shaking frequency: 150 rev/min and pH 5.7)

For the estimation of equilibrium time, the adsorption experiment was carried out at pH 5.7, shaking frequency: 150 rev/min and concentration of solution was 3.34×10^{-3} M at 30 °C. The amounts of prepared activated carbon taken for the experiments were: 1.0, 2.0, 3.0, 4.0 and 5.0 g Dimensions of the particles size were (particles diameter ≤ 200 mesh). Under this condition to accomplish the equilibrium time for the adsorption of Hg^{2+} ions onto prepared activated carbon were found to be about 3 hours. Results of equilibrium time were presented in Table 4.5.7 and in Figure 4.5.11. It is observed from Table 4.5.7 that the percentage of adsorption increased with increasing the amount of adsorbent but the adsorption capacity decreased with increasing the amount of adsorbent. In most cases the adsorption capacity retain within 87 to 115 mg/g while the initial concentration of Hg^{2+} is 3.34×10^{-3} M at 30 °C. During estimating the equilibrium time, it is also observed from the table that maximum removal of Hg^{2+} ions onto prepared activated carbon is found to be 72.58% within 3 hours and that for minimum is 14.35%. The fast rate of removal and the high percentage removal at equilibrium found proves prepared activated carbon is an efficient adsorbent to remove Hg^{2+} from aqueous solution. The experimental data has been fitted into adsorption isotherms and found to be well fit into Freundlich isotherm (Figure 4.5.12). The value of n is 3.12. It is also seen from the results the isotherm that chemisorption of Hg^{2+} occurred on prepared activated carbon.

The experiments have been carried out under the following experimental condition:

pH of the solutions	: 5.7
Temperature	: 30 °C
Amount of adsorbents	: 1.0, 2.0, 3.0, 4.0 and 5.0 g
Particle size	: particles diameter ≤ 200 mesh
Volume of aqueous Hg^{2+} solution	: 850 mL
Rate of shaking	: 150 rev/min
Time of adsorption	: 6.0 hours
Initial concentration of Hg^{2+} solution	: 3.34×10^{-3} M

Table 4.5.7 : Determination of equilibrium time for the adsorption of aqueous Hg^{2+} ions onto prepared activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+} ions: 3.34×10^{-3} M and 30 °C.

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	30	1.00	3.34×10^{-3}	599.55	100.45	14.35	85.38
			2.00		501.55	198.45	28.35	84.34
			3.00		403.27	296.73	42.39	84.07
			4.00		313.95	386.05	55.15	82.04
			5.00		259.00	441.00	63.00	74.97
5.7	150	60	1.00	3.34×10^{-3}	591.85	108.15	15.45	91.93
			2.00		494.41	205.59	29.37	87.38
			3.00		392.63	307.37	43.91	87.09
			4.00		302.75	397.25	56.75	84.42
			5.00		245.00	455.00	65.00	77.35
5.7	150	90	1.00	3.34×10^{-3}	583.80	116.20	16.60	98.77
			2.00		480.55	219.45	31.35	93.27
			3.00		381.71	318.29	45.47	90.18
			4.00		291.83	408.17	58.31	86.74
			5.00		232.40	467.60	66.80	79.49
5.7	150	120	1.00	3.34×10^{-3}	573.92	126.08	18.01	107.17
			2.00		467.25	232.75	33.25	98.92
			3.00		370.58	329.42	47.06	93.34
			4.00		280.70	419.30	59.90	89.10
			5.00		218.40	481.60	68.80	81.87
5.7	150	150	1.00	3.34×10^{-3}	565.59	134.41	19.20	114.25
			2.00		451.85	248.15	35.45	105.46
			3.00		358.89	341.11	48.73	96.65
			4.00		269.01	430.99	61.57	91.59
			5.00		204.40	495.60	70.80	84.25
5.7	150	180	1.00	3.34×10^{-3}	556.77	143.23	20.46	121.74
			2.00		437.15	262.85	37.55	111.71
			3.00		347.20	352.80	50.40	99.96
			4.00		257.32	442.68	63.24	94.07
			5.00		191.94	508.06	72.58	86.37
5.7	150	210	1.00	3.34×10^{-3}	556.77	143.23	20.46	121.74
			2.00		437.15	262.85	37.55	111.71
			3.00		347.48	352.52	50.36	99.88
			4.00		257.32	442.68	63.24	94.07
			5.00		192.01	507.99	72.57	86.36

pH	Shacking Frequency (rev/min)	Time (min)	Amount of carbon (g)	Initial Conc. (mol/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)
5.7	150	240	1.00	3.34×10^{-3}	556.69	143.31	20.47	121.81
			2.00		437.01	262.99	37.57	111.77
			3.00		346.78	353.22	50.46	100.08
			4.00		257.18	442.82	63.26	94.10
			5.00		192.01	507.99	72.57	86.36
5.7	150	270	1.00	3.34×10^{-3}	556.76	143.24	20.46	121.75
			2.00		437.08	262.92	37.56	111.74
			3.00		346.78	353.22	50.46	100.08
			4.00		257.25	442.75	63.25	94.08
			5.00		191.94	508.06	72.58	86.37
5.7	150	300	1.00	3.34×10^{-3}	556.76	143.24	20.46	121.75
			2.00		437.15	262.85	37.55	111.71
			3.00		347.48	352.52	50.36	99.88
			4.00		257.32	442.68	63.24	94.07
			5.00		191.94	508.06	72.58	86.37
5.7	150	330	1.00	3.34×10^{-3}	556.76	143.24	20.46	121.75
			2.00		437.15	262.85	37.55	111.71
			3.00		346.78	353.22	50.46	100.08
			4.00		257.32	442.68	63.24	94.07
			5.00		191.94	508.06	72.58	86.37
5.7	150	360	1.00	3.34×10^{-3}	556.83	143.17	20.45	121.69
			2.00		437.15	262.85	37.55	111.71
			3.00		347.48	352.52	50.36	99.88
			4.00		257.32	442.68	63.24	94.07
			5.00		191.98	508.02	72.57	86.36

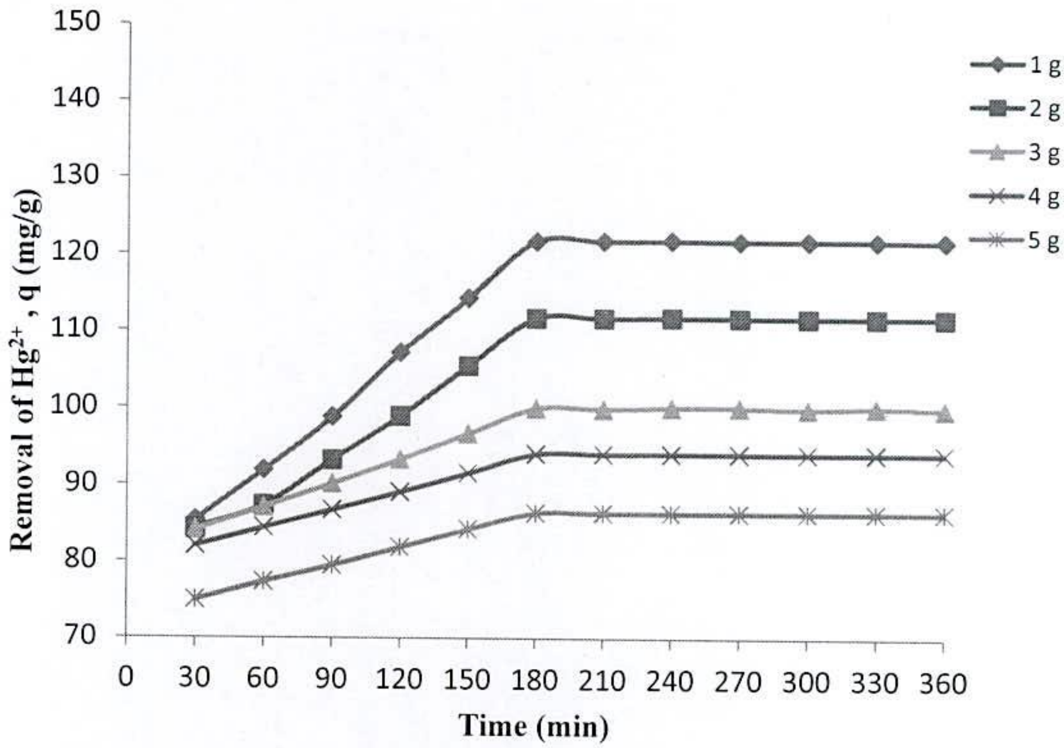


Figure 4.5.11 : **Plot of removal of Hg^{2+} against time to determine equilibrium time of adsorption of Hg^{2+} on activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 3.34×10^{-3} M and 30 °C.**

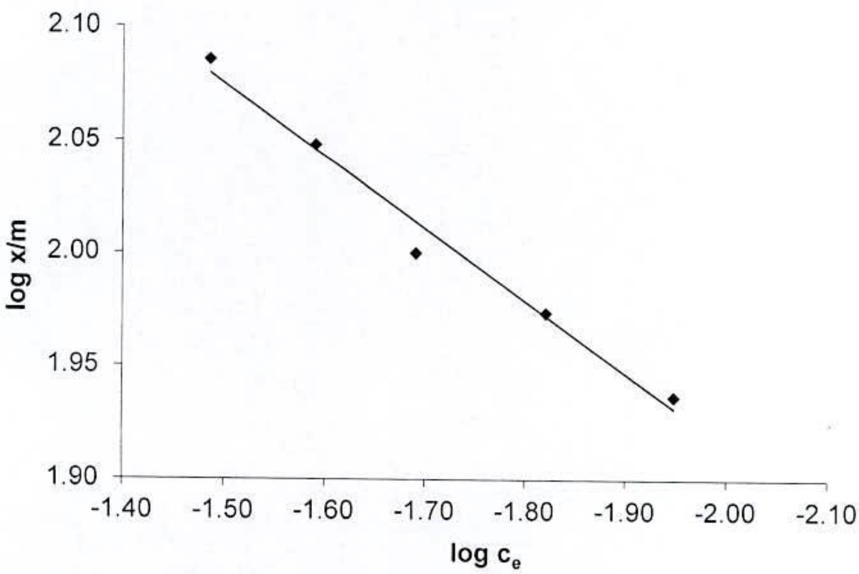


Figure 4.5.12 : **Freundlich isotherm for the adsorption of Hg^{2+} onto activated carbon at pH: 5.7, shaking frequency: 150 rev/min, initial concentration of Hg^{2+}: 2.90×10^{-3} M and 30 °C.**

4.5.7 Quantification of the adsorption isotherm data

The linear form of Freundlich equation, which was also applied for the adsorption of Hg^{2+} is given as:

$$log\frac{x}{m} = logk + \frac{1}{n}logC_e$$

Where, k is roughly an indicator of the adsorption capacity and 1/n the adsorption intensity. In general, as the k value increases the adsorption capacity of the adsorbent for a given dye increases. The magnitude of the exponent 1/n gives an indication of the favorability of adsorption. Values, n>1 represent favorable adsorption condition. Linear plots of $log\frac{x}{m}$ vs. log Ce (Figure 4.5.4, 4.5.6, 4.5.8, 4.5.10 and 4.5.12) show that the adsorption of activated carbon onto also follow the Freundlich isotherm. Data for the Freundlich isotherm are shown in Table 4.5.7. Values of k and n were calculated from the intercepts and slopes of the plots and are listed in Table 4.5.8. The results suggest that the activated carbon is favorably adsorbed by activated carbons prepared from rice husk.

Table 4.5.7 : Freundlich constants for the adsorption of aqueous Hg^{2+} ions onto prepared activated carbon

Amount of carbon (g)	Initial Conc. (mg/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)	Equilibrium Conc., C_e (M)	log C_e	log x/m
1.00	3.34×10^{-3}	556.77	143.23	20.46	121.74	0.0327	-1.49	2.09
2.00		437.15	262.85	37.55	111.71	0.0256	-1.59	2.05
3.00		347.20	352.80	50.40	99.96	0.0204	-1.69	2.00
4.00		257.32	442.68	63.24	94.07	0.0151	-1.82	1.97
5.00		191.94	508.06	72.58	86.37	0.0113	-1.95	1.94
1.00	2.90×10^{-3}	476.46	123.54	20.59	105.01	0.0279	-1.55	2.02
2.00		367.37	232.63	38.77	98.87	0.0215	-1.67	2.00
3.00		263.12	336.88	56.15	95.45	0.0154	-1.81	1.98
4.00		176.38	423.62	70.60	90.02	0.0103	-1.99	1.95
5.00		108.18	491.82	81.97	83.61	0.0063	-2.20	1.92
1.00	2.42×10^{-3}	387.25	112.75	22.55	95.84	0.0227	-1.64	1.98
2.00		292.51	207.49	41.50	88.18	0.0172	-1.77	1.95
3.00		204.86	295.14	59.03	83.62	0.0120	-1.92	1.92
4.00		133.40	366.60	73.32	77.90	0.0078	-2.11	1.89
5.00		90.05	409.95	81.99	69.69	0.0053	-2.28	1.84

Amount of carbon (g)	Initial Conc. (mg/L)	After Adsorption (mg/L)	Total Adsorption (mg/L)	Percentage of Adsorption (%)	Adsorption Capacity (mg/g)	Equilibrium Conc., C_e (M)	log C_e	log x/m
1.00	1.93×10^{-3}	303.39	96.61	24.15	82.12	0.0178	-1.75	1.91
2.00		225.49	174.51	43.63	74.17	0.0132	-1.88	1.87
3.00		160.33	239.67	59.92	67.91	0.0094	-2.03	1.83
4.00		117.69	282.31	70.58	59.99	0.0069	-2.16	1.78
5.00		77.68	322.32	80.58	54.79	0.0046	-2.34	1.74
1.00	1.48×10^{-3}	216.21	83.79	27.93	71.22	0.0127	-1.90	1.85
2.00		152.36	147.64	49.21	62.75	0.0089	-2.05	1.80
3.00		106.38	193.62	64.54	54.86	0.0062	-2.20	1.74
4.00		74.62	225.38	75.13	47.89	0.0044	-2.36	1.68
5.00		49.32	250.68	83.56	42.62	0.0029	-2.54	1.63

Table 4.5.8 : Freundlich constants for the adsorption of aqueous Hg^{2+} ions onto prepared activated carbon

Initial Conc. of Hg^{2+} (mol/L)	Value of k	Value of n
1.48×10^{-3}	2.51	2.83
1.93×10^{-3}	2.43	2.43
2.42×10^{-3}	2.31	4.87
2.90×10^{-3}	2.47	6.76
3.34×10^{-3}	2.55	3.12

Conclusion

Activated carbon has been prepared from rice husk in a locally designed furnace and very slow burning method in presence of insufficient oxygen was adopted. The carbonized temperature and time was 350–550 ◦C and about 3.0 hours respectively. The percentage of the yield in this method is about 25 (wt) %. The prepared activated carbons contain 39.47% ash and 35.98% fixed carbon. SEM photographs exhibit that the prepared activated carbon possesses significant number of micropores, mesopores and macropores. XRD analysis provides information that the prepared samples are amorphous in nature. SEM photograph before and after adsorption clearly showed the evidence in favor adsorption. Prepared activated carbon was used for the removal of methylene blue, Pb^{2+} and Hg^{2+} in aqueous solution. Following observations were perceived in the adsorption process:

- The removal of methylene blue, Pb^{2+} and Hg^{2+} at equilibrium were increased upon increasing the initial concentration of methylene blue, Pb^{2+} and Hg^{2+}.
- Methylene blue showed maximum adsorption at pH 6.0 while both Pb^{2+} and Hg^{2+} showed maximum adsorption at pH 5.7.
- Both Pb^{2+} and Hg^{2+} showed maximum adsorption at 150 rev/min shaking frequency
- Methylene blue adsorption experiments done at 30 °C and 40 °C and in both cases chemisorption occurred established by Freundlich adsorption isotherm.
- Both Pb^{2+} and Hg^{2+} were adsorbed onto prepared activated carbon at 30 °C and in all cases chemisorption occurred recognized by Freundlich adsorption isotherm.

XRD As, Cd

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