

**COMPARATIVE STUDY ON ACIDIC AND BASIC ACTIVATING AGENT IN THE
ADSORPTION OF CRYSTAL VIOLET FROM TEXTILE WASTE WATER USING
CARBONIZED SAWDUST.**



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JULY, 2021

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SUBMITTED TO

**DEPARTMENT OF CHEMICAL ENGINEERING
FACULTY OF ENGINEERING
UNIVERSITY OF BENIN
BENIN CITY**

**IN PARTIAL FULFILLMENT OF THE REQUIREMENT FOR THE AWARD OF
BACHELOR DEGREE IN CHEMICAL ENGINEERING (B.ENG)**

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CERTIFICATION

This is to certify that this research project submitted to the Department of Chemical Engineering was carried out by **OSAH OHWOFASA** of the Department of Chemical Engineering University of Benin, Benin City, Edo State Nigeria, under the supervision of **ENGR. (MRS.) O. EDOKPAYI**

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Date

DEDICATION

This report is dedicated to God almighty the giver of knowledge, wisdom and inspiration. Also, to the entire body of Chemical Engineering, Students, researchers, to whom the above work is related to in different facets of life and human endeavor.

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My profound gratitude goes to the maker of the universe, the Lord God almighty for his infinite wisdom, grace and mercy towards me.

To my wonderful project supervisor, Engr. Mrs. O. Edokpayi, I say thank you ma for all the sacrifices made, I am grateful.

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ABSTRACT

The adsorption of crystal violet from textile waste water onto carbon produced from sawdust that was activated by phosphoric acid and potassium hydroxide was an experiment that was carried out under room temperature. Adsorption is a separation process where the molecules of a solute in an aqueous solution are adsorbed to the surface of another molecule.

The materials used for this experiment are phosphoric acid, potassium hydroxide, crystal violet dye, distilled water and activated carbon made from sawdust. In order to obtain the aim of the experiment, different experiments were performed which are; effect of initial concentration, effect of adsorbent dosage, effect of contact time, effect of temperature and adsorption isotherms were studied in order to find out the activating agent which best fit for the removal of the crystal violet. The percentage removal of the crystal violet was calculated to be 86.03% and 86.50% for the basic and acidic activating agents respectively for the adsorbent dosage experiment. Also, the determination coefficient value, R^2 for the acid treated sawdust activated carbon for Langmuir isotherms was 0.9992, maximum adsorption capacity, Q_0 was 29.1545mg/g and the dimensionless separation parameters, R_L was found to be favorable with value 0.0448.

In conclusion, the acid activated carbon was found to be more effective in the removal of crystal violet when compared to the alkaline activated carbon since its R^2 value is higher.

CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF STUDY

Industrial development has increased the amount of effluent discharged into the environment. Water as an essential constituent of the planet has been affected by various pollutants such as dyes, heavy metals, organic substances, pesticides, etc. [Kalavathy *et al* 2009]. The word textile comes from the Latin word 'texere' which means to weave. Textiles can be woven by both hand and machines. The textile industries are classified on the basis of the types of textile fiber they use. The raw materials for textiles are natural and synthetic fibres [Elliott *et al.*, 1954]. The textile raw materials can be classified into three main categories: cellulose fibres (cotton, rayon, linen), protein fibres (wool, silk) and synthetic fibres (polyester, nylon, acrylic) [Mostafa 2015]. Industries like the textile industry had produced contaminated waste water that is rich in harmful organic substances since they make use of dyes to colour their products [Mohan *et al* 2007]. The release of this contaminated water containing dyes without treatment affects the people who may use this water for living purposes such as drinking, bathing, cooking [Sharma *et al* 2000]. Therefore, it is very important to verify the water quality, especially when even just 1.0mg/L of dye concentration in drinking water could impart a significant color, making it unfit for human consumption [Malik *et al* 2007]. Furthermore, dyes can affect aquatic plants because they reduce sunlight transmission through water. Also, dyes may impart toxicity to aquatic life and may be muta-genic, carcinogenic and may cause severe damage to human beings, such as dysfunction of the kidneys, reproductive system, liver, brain and central nervous system [Kadirvelu *et al* 2003]. Furthermore, the treatment of this contaminated water is very paramount since a little content of these dyes in water is toxic to the human life and it is therefore of great need to look for efficient means to remove these dyes [Chiou *et al* 2004].

Dyes are colorful substances which have been utilized by humans since 3500 BC in various applications using natural extracts of flowers, fruits, certain insects, etc. natural dyes constitute a very limited range of colors and are produced in low quantities. However, after the discovery of synthetic colors by W. H. Perkins in 1856, a wide range of dyes are used in various fields to color their product such as paper, leather, rubber, textile, plastics, etc. [Kant 2012]. The dyes are

one important part of the pollution problem as it is estimated that 50% of their amount is not fixed on fibers and remain finally in wastewater. Dyes such as crystal violet (CV) are composed of many functional groups that are stable and difficult to decompose because of their aromatic nature [Alsenani 2013]. CV is used for making black and blue inks in ball-point pens and in printer ink jet manufacturing industrials [Alizadeh *et al* 2017]. Color paints, pharmaceuticals, leather, detergents, fertilizers, varnish, and waxes are also manufactured from CV [Nguyen *et al* 2013]. However, CV, similar to most dyes, is a toxic carcinogenic with a recalcitrant classification because of its non-biodegradability, persistence in various environments, and poor microbial metabolization [Parab *et al* 2009]. Furthermore, CV causes undesirable colorations of water bodies, resulting in low light penetration for photosynthetic activities, which affect aquatic life such as the growth of tumors in fish [Mohanty 2006]. Consequently, the removal of CV from waste water before discharge must be implemented for the survival of both humans and aquatic organisms.

Therefore, the use of agricultural by-products in adsorption process has been recently proven to be very efficient and economical method for the removal of the different pollutants such as dyes, heavy metals, phenol, COD and gases [Kayode *et al* 2015]

1.2 JUSTIFICATION

i Due to the fact that industries that make use of dyes will keep multiplying themselves in our country there is need to sort for ways in treating these pollutants present in the waste water produce from these industries since it poses treats to humans and aquatic creatures.

ii Having known that the conventional methods such as ion exchange resins, precipitation, chemical coagulation, sedimentation and biological of waste water treatment require alot of capital, it therefore needful to find alternative ways for the treatment of the effluents from textile industry

1.3 AIM AND OBJECTIVES

The aim of this study is to adsorb crystal violet dye ions from textile waste water using activated carbon produced from sawdust.

The objectives of this study are;

- To produce carbon from sawdust
- To activate the produced carbon using acid (H_3PO_4) and alkali (KOH)
- To determine the effectiveness of this activated carbon in the removal of crystal violet
- To determine the removal of this dye via adsorbent dosage experiment
- To determine the removal of this dye via contact time experiment
- To determine the removal of this dye via varying temperature experiment

1.4 SCOPE OF STUDY

This project work entails the production of carbon from sawdust, activation of the carbon using an acid (phosphoric acid, H_3PO_4) and an alkali (potassium hydroxide, KOH), preparation of the stock solution containing crystal violet and to study the effectiveness of these activated carbon in the removal of the dye via different experimental methods.

1.5 RELEVANCE OF STUDY

The relevance of this study is to know the efficiency of acid and basic activated carbon produced from sawdust in the removal of crystal violet in textile waste water.

CHAPTER TWO

LITERATURE REVIEW

2.1 POLLUTION

Many of the major problems that humanity is facing in the twenty-first century are related to water quantity and/or water quality issues [Schwarzenbach *et al* 2010]. These problems are going to be more aggravated in the future by climate change, resulting in higher water temperatures, melting of glaciers, and an intensification of the water cycle [Huntington 2006], with potentially more floods and droughts [Oki 2006]. With respect to human health, the most direct and most severe impact is the lack of improved sanitation, and related to it is the lack of safe drinking water, which currently affects more than a third of the people in the world.

Mining activities worldwide mobilize more than 50×10^9 metric tons of geological material per year, which is similar to the flux of particles transported by rivers from the continents to the sea [Douglas *et al* 2000]. Most mining operations trigger significant environmental and social problems as they result in large waste deposits, which are exposed to oxidation by air and weathering by precipitation, and subsequent pollution of water resources [Bridge 2004]. Mining for coal, lignite, building materials, and iron involves the largest mass movements with a significant yield of end products. The extraction of rare metals, such as copper, nickel or gold, however, produces up to 1,000 tons of waste materials per kilogram of pure metal.

Municipal wastewater contributes significantly to the micro pollutant load into the aquatic environment [Kolpin *et al* 2002]. The main concerns are pharmaceutical compounds and personal care products. Approximately 3,000 pharmaceuticals are used in Europe and the United States today, including painkillers, antibiotics, beta blockers, contraceptives, lipid regulators, antidepressants, and others [Ternes *et al* 2006]. Pharmaceuticals are launched on the market every year with 8% of the worldwide research and development [R&D] expenditure. On the basis of the world wide R&D expenditure of about US\$83 billion in 2007 [Verband 2009], it can be extrapolated that on average more than 300 new pharmaceutical compounds are launched every year. The worldwide market of pharmaceuticals [100,000 tons per year was US\$773 billion, with the highest per capital sales of US\$676 in the United States [Kummerer 2004].

2.2 TEXTILE WASTE WATER

The Textile Industrial Wastewater is largely yielded by the Textile industry all day long to night, might be called as a Manufacturer through some operations related to the industrial production. However, all the textile industries consume a large quantity of water for several ways in order to different purposes. Although a good number of chemicals of variable textile processing such as bleaching, scouring, dyeing, finishing & so forth, most obviously dyeing & finishing are the processes in which a variety of chemicals is widely used. Nonetheless, a large quantity of discharged wastewater turned to colored wastewater by the manufacturing of dyes in industry. But it now concerned of few causes like toxicity, accumulation & a few like them. If the toxicity increased & perfectly accumulated in nature, this process may be seemed like as a terrible view in environmental balance [Fazlur 2016]. Textile dyes are ionized and organic, shows a strong affinity to the aqua solution and a bit on industry water, mostly used to be coloring during the manufacture of final product. Moreover, a lot of industries use finishing in the manufacturing process in leather, plastic, tanning & textile industry [Karthik *et al* 2014]. Furthermore, both of dye manufacturing and textile finishing become a source of presumptive but only dye-manufacturing can discharge industrial waste-water without any disturbance [Yasemin 2012].

2.3 DYES

Dyes as colorful substances have been utilized by humans since 3500 BC in various applications using natural extracts of flowers, fruits, certain insects, etc. These natural dyes constitute a very limited range of colors and are produced in low quantities. However, after the discovery of synthetic colors by W. H. Perkins in 1856, a wide range of dyes are used in various fields to color their product such as paper, leather, rubber, textile, plastics, etc. [Kant 2012]. Synthetic dyes are developed and have replaced natural dyes gradually in different industries because their molecules are stable and can resist degradation upon contact with water, detergents, or any other washing agents [Al-Alwani *et al* 2018]. As shown in Table 1, dyes can be classified based on their chemical structure into azo, anthraquinone, indigoid, nitroso, nitro, and triarylmethane dyes [Yagub *et al* 2014]. Sometimes, they are classified by their application or by their solubility in water; vat, azoic, sulfur, and disperse dyes are in soluble, while reactive, direct, basic, and acid dyes are highly soluble [Hassan *et al* 2018]. Figure 1 presents types of dyes used in different

industries. As we all know, dyes are indispensable to modern life. However, their discharge as industrial waste into water body causes threat to living things in the environment [Gholami-Borujeni *et al* 2018]. Dyes may affect the photosynthesis of aquatic life due to decreased sunlight transmission [Lambert *et al* 2011]. Furthermore, most of these dyes are harmful and potentially carcinogenic, mutagenic, or teratogenic for aquatic organisms. They can also cause severe damage to human beings such as dysfunction of the kidney, reproductive system, liver, brain, and central nervous system [Zhou *et al* 2019]. Additionally, synthetic dyes are chemically stable and resistant to degradation even when exposed to extreme heat sources, oxidizing agent, or strong light [Forgacs *et al* 2004]. For these reasons, highlighting efficient treatment methods of dye wastewater is necessary.

2.3.1 CATEGORIES OF TEXTILE DYES

By consisting data collections, it narrates every dye, consisting properties of physical & chemical. Gathering of information of some dyes mentioned as much as possible for environmental factor. Afterwards, the dyes are selected & have effect to the nature & health factor by developing activities. These dyes are propagated to the textile industry of dyeing process [George *et al* 2013]. They are;

Sulphur dyes: These are applicable in alkaline solution. They widely used for cotton, viscous & staple fibers& so forth.

Disperse dyes: They are insoluble in water. They have worldwide usage in textile industry, basically for synthetic fibers like polyester & cellulose acetate such as Di-acetate, Tri-acetate & others. This dye also used for nylon. They are applied on the dye bath at high temperature range around 120°C - 140°C.

Direct dyes: They are the dyes, that can be called as “substantive dyeing”. Cotton used as natural fiber & Viscous used as synthetic fiber. They also used for aqueous solution. In solution, electrolysis & salts are available. Predominantly, direct dyes are used as the second dyes in worldwide. Direct dyes used for cotton, viscous.

Azoic dyes: These are used for natural fibers, viscous, cellulosic fibers. They consist two soluble components to be formed colored molecules, are insoluble. The synthesis of azo dyes operated by two stages such as Diazotization and Azo Coupling

2.4 CRYSTAL VIOLET

Crystal violet (CV) have a Molecular formula, $C_{25}H_{30}N_3Cl$ and Molecular weight, 407.979 []. They are composed of many functional groups that are stable and difficult to decompose because of their aromatic nature. Crystal violet is a basic dye and it's used to colourize diverse products such as paper, leather, fertilizers, anti-freezes, detergents, and also as a component of inks for ball-point pens. It is also used in veterinary medicine, such as a dermatological drug, and for expelling intestinal parasites and fungi from the body (Tolga *et al* 2011). However, like other common dyes, CV is highly toxic to living organisms. Therefore, it should be removed from the wastewater.

2.5 REMOVAL OF TEXTILE DYES BY TECHNIQUES OF TREATMENT

Numerous methods are obviously relevant to the dye removal of industrial waste water treatment. Among of them, it is feasible to identify the proper techniques that are best and they can be described below [Karthik *et al* 2014]:

- i. Chemical technique
- ii. Physical technique
- iii. Biological technique

Physical technique

Physical technique is consisted of several methods such as sedimentation (clarification), screening, Nano filtration, reverse osmosis, electro dialysis and so forth. During the process of clarification, physical is a phenomenon that is allowed to be operated, relating to settling of solid by gravity. Sedimentation is one of the most physical treatment methods, used to attain treatment. In addition, another treatment method is aeration, by summing air to provide oxygen (O_2) to the wastewater.

Chemical technique

Chemical technique contained some methods like ozonation, neutralization, chlorination, & so on. Chemical treatment reposes of chemical reactions to develop the quality of water. Perhaps, Chlorination is the most common chemical method. A strong oxidizing chemical named chlorine

(Cl₂) used to kill bacteria & also the dissipation of wastewater. Neutralization is one of the most common methods, used industrial wastewater treatment. Neutralization consists of the addition of acid or base to modify pH levels back to neutrality. Ozonation is also one of the regular methods as chemical technique for using wastewater treatment

Biological technique

Biological technique is one of the most cost-effective treatment methods than others treatment methods. Biological treatment is awkward to the regular variation & toxoids whereas it has good affability in design & activity. It is overviewed to be proven that biological treatment is the way of eradication of color with tolerable. Some metals which are heavy are involved of the wastewater of textile industry.

2.6 HISTORY OF ACTIVATED CARBON AND PRESENT - DAY APPLICATIONS

The useful properties of activated carbon have been known since ancient times. This traces back to 1500 BC when Egyptians used charcoal as an adsorbent for medicinal purposes and a purifying agent. Around 420 BC it was observed that Hippocrates dusted wounds with powdered charcoal to remove their odor. Ancient Hindu societies purified their water by filtration through charcoal [Bansal *et al* 2005]. In 1773, the Swedish chemist Karl Wilhelm Scheele was the first to observe adsorption of gases on charcoal. A few years later activated carbons began being used in the sugar industry as a decolorizing agent for syrup.

In the early 20th century the first plant to produce activated carbon industrially was built for use in sugar refining industry in Germany. Many other plants emerged in the early 1900's to make activated carbons primarily for decolorization. During World War I, activated carbon was used in gas masks for protection against hazardous gases and vapors. Today, activated carbons are used to remove color from pharmaceutical and food products, as air pollution control devices for industrial and automobile exhaust, for chemical purification, and as electrodes in batteries. 500,000 tons per year of activated carbon are produced globally [Jankowska *et al* 1991]. 80% of this is used for liquid phase applications, and 20% is used for solid phase applications

[Bansal *et al* 2005]. This paper will focus on activated carbons applications as a natural gas adsorbent with the intentions of using the stored natural gas as an alternative fuel to petroleum.

2.6.1 TYPES OF ACTIVATED CARBON

Relating to the particle size, activated carbon can be classified in dust carbons or granular carbon. Dust activated carbons: Those carbons are characterized for having a size lower than 100 μm , being the common ones between 15 and 25 μm . The most important physical properties are the filterability and global density. That kind of carbons show some advantages, first of all is approximately twice cheaper than granular carbon and the adsorption kinetics is quick due to its surface is easily accessible. Otherwise, show some inconvenient in the recovery process.

Granular activated carbon: Those carbons show an average particle size between 1 to 5 mm can be divided into two categories: chopped carbon (formless) and formed carbon with a specific form as cylindrical. The first type is obtained by milling and sieving while the second ones are a mixture of carbon and a binder. Furthermore, there also other forms of carbon adsorbents as activated carbon fibres, filters of activated carbon, monolithic structures, carbon membranes, etc. Those carbons are valuable for their hardness and particle size. Much of the operation cost is caused for the attrition during the regeneration and normal work.

2.7 SAWDUST

Sawdust Nowadays, sawdust plays an important role in the adsorption of wastewater pollutants, because it contains numerous functional groups such as carboxyl, hydroxyl, phenolic, and amide groups in its structure, which may be favorable for adsorbing a large variety of dyes [Sciban *et al* 2007]. Furthermore, sawdust can be modified by acid and alkali to increase its adsorption properties. A considerable amount of literature has been published on the use of wood sawdust: its composition, its preparation methods, and its capacity to remove dyes from effluents [Tarr'es *et al* 2020].

Chemical Composition of Sawdust: Sawdust as a lignocellulosic material can be utilized as a sustainable precursor to produce activated carbon. In fact, the major chemical constituents of sawdust are hemicellulose, cellulose, and lignin [Danish *et al* 2018]. On average, the quantitative percentages of hemicellulose, cellulose, and lignin in the sawdust are observed in the range of 15–35, 35–60, and 15–30%, respectively [Rangabhashiyam *et al* 2019].

2.7.1 PREPARATION METHODS OF SAWDUST

Generally, there are two different methods for activated carbon preparation: they are chemical and physical activation methods [Yang *et al* 2015].

Physical activation treatments consist of two steps: in the first, precursors will be carbonized in presence of inert atmosphere such as nitrogen or helium, whereas in the second step, the carbonized substance is placed in air, oxygen (O₂), carbon dioxide (CO₂), steam, or the mixed atmosphere at much elevated temperature. On the other hand, in chemical treatments, precursors are impregnated by an activating reagent, followed by heating process under an inert atmosphere [59–63]. In physical activation, the purpose of the carbonization is to eliminate the volatile matters at relatively low temperature (400–700°C) from the raw material and then to convert the resulting char with higher content of fixed carbon for activation purpose. At high temperature (800–1200°C), activation treatment with oxidizing gas takes place to form carbon oxides, and pores are created inside the carbon material by releasing gas [Li *et al* 2020].

Chemical activation method refers to the impregnation of the raw material with a dehydrating chemical agent and heating at a high temperature under inert atmosphere. A variety of chemical activating agents such as potassium hydroxide (KOH), zinc chloride (ZnCl₂), phosphoric acid (H₃PO₄), sulphuric acid (H₂SO₄), nitric acid (HNO₃), potassium carbonate (K₂CO₃), etc. can be used [Chikri *et al* 2020].

2.8 ACTIVATING AGENTS

There are many different activation agents, but the most studied ones are phosphoric acid, zinc chloride and potassium hydroxide.

Phosphoric acid

Phosphoric acid also known as orthophosphoric acid is a mineral acid having the chemical formula H₃PO₄. Phosphoric acid works in two ways (Robert *et al* 2001): as an acidic catalyst in promoting bond cleavage reactions and formation of crosslink; and by being able to combine with organic species to form phosphate linkages, such as phosphate and polyphosphate esters, that can serve to connect biopolymer fragments.

Potassium hydroxide

This agent is an inorganic compound with the formula KOH. It is a strong base and is used in many industrial applications. Some of its characteristics are its high reactivity toward acids and its corrosive nature.

Zinc chloride

Zinc chloride is the name of a chemical compound with the formula $ZnCl_2$. This product was employed in the 1970's especially for wood wastes. However, it is no longer used due to the environmental problems that generate. Impregnation with $ZnCl_2$ produces a degradation of the cellulosic material, during the carbonization results in charring and aromatization of the carbon skeleton and creation of the pore structure (Figueiredo *et al* 1999).

2.9 ADSORPTION PROCESS

Adsorption is a physicochemical phenomenon in which a solid, called adsorbent, retains in its walls a certain kind of molecules, called adsorbates, that are contained in a gas or liquid. Hence it is a separation and concentration process of one or more compounds of a system on a solid or liquid surface. The most employed adsorbents are silica, some synthetic resins and activated carbons. Factors to be taken into consideration in an adsorption process regarding to the adsorbent-adsorbate are the following:

1. Specific surface and porosity of the solid
2. Particle size
3. Pore size, structure and distribution.
4. Affinity of the adsorbate, depending on the chemical properties of the adsorbent surface.
5. Partial pressure or concentration of the adsorbate in the fluid phase.

There can be three types of adsorption attributed to the adsorbent-adsorbate attraction: electrical, Van der Waals or chemical. The first type is commonly called ion exchange because the ions of a substance are concentrated on the surface as a result of an electrostatic attraction. The Van der Waals adsorption, also known as physical adsorption, takes place when the molecule is bound into the interphase usually at low temperatures. Most of organic compounds that are in water are adsorbed with activated carbons for this type of adsorption. The chemical adsorption is a reaction between the adsorbate and the adsorbent due to their chemistry and is characterized for being strong in the active point of the adsorbent.

CHAPTER THREE
MATERIALS AND METHODS

3.1 MATERIALS

Table 3.1 Materials used during experiment

S/N	Materials	Source	Uses
1	Sawdust	Local vendor along uselu road	For the production of activated carbon
2	Crystal Violet dye	LUCO scientific company Ltd	To prepare the stock solution that is to be purified
3	Phosphoric acid	LUCO scientific company Ltd	To activate the carbon
4	Distilled water	LUCO scientific company Ltd	To prepare solutions and wash equipments
5	Potassium hydroxide	LUCO scientific company Ltd	To activate the carbon
6	Sulphuric acid	LUCO scientific company Ltd	To neutralize the alkali activated carbon

Table 3.2 Equipment /Apparatus

S/N	Equipment/Apparatus	Uses
1	Crucible	To place the coconut shells into the furnace
2	Electric muffle furnace	To carbonize and activate the coconut shells
3	Mortar and pestle	To reduce the size of the carbon
4	Sieve	To sieve the carbon after reduction of size
5	Funnels	To hold the filter papers during filtration
6	Beakers	To hold solutions
7	Conical flasks	To hold the funnels straight while filtering the adsorbent from the purified solution
8	Measuring cylinders	To measure solutions
9	Filter papers	To filter the adsorbent from the purified solution
10	Pan	For drying the adsorbent in the oven
11	Oven	To dry the adsorbent
12	Electric vibrating stirrer	To stir the mixture of the crystal violet dye solution and the adsorbent
13	pH paper	To check the washed activated carbon
14	Spectrophotometer	To measure the absorbance of the dye solution
15	Masking tape	To label the containers
16	Stirring rod	To stir solutions
17	Magnetic stirrer	To heat up the sample to a desired temperature

3.2 METHODS

3.2.1 PRODUCTION OF CARBON FROM SAWDUST

The sawdust was obtained from a local wood milling factory in Benin City, Edo State. The saw dust was first sun dried to remove all moisture content. Thereafter the saw dust was placed in the furnace in order to be carbonize at the temperature of 620°C. The carbon was crushed with mortar and pestle to obtain a finer particle, the resulting material was sieved with a mesh size of 500 microns particle size. The carbon was divided into two portions and each was impregnated.

A portion was impregnated with 14.02g in 500ml (0.5M) of potassium hydroxide for 24 hours while the other was impregnated with 5.65ml of phosphoric acid for 24hours. Both samples were then washed with deionized water until a pH of 7 was achieved and the samples were dried to remove moisture at 105°C in the oven. The material was placed in an airtight container for further use.

The yield of the activated carbon can be calculated as follows:

$$Yield = \frac{W_1}{W_2} \times 100\%$$

W1 is the mass of the unwashed carbon after activation

W2 is the original mass of the precursor on dry basis.

3.2.2 PREPARATION OF AQUEOUS CRYSTAL VIOLET SOLUTION.

1g of crystal violet dye was measured with help of the weighing balance and transferred into 1000ml (1L) of distilled water in measuring cylinder. This solution was allowed to mix properly before using it for the other experiments

3.2.3 EFFECT OF ADSORPTION FACTORS ON DYE UPTAKE

3.2.3.1 EFFECT OF INITIAL DYE CONCENTRATION

1ml, 2ml, 3ml, 4ml, 5ml, and 6ml of the stock solution were collected into a 250ml measuring cylinder each and made up to 100ml. 100ml of the solution was measured into six (6) 200ml Erlenmeyer flasks and the mass of 0.4g was added to the different flasks containing different concentration, thereafter they were placed in acyclone vibrator to stir for 5 minutes. The mixtures were filtered and their absorbance were taken with the aid of the spectrophotometer.

3.2.3.2 EFFECT OF ADSORBENT DOSAGE

Different adsorbent dosage was taken ranging from 0.05g to 0.5g of the adsorbent and added into 50mg/l for acidic and alkaline samples respectively and were placed in the cyclone vibrator for proper agitation of the samples for 5 minutes before filtration and their respective absorbance were taken with the aid of the spectrophotometer.

3.2.3.3 EFFECT OF CONTACT TIME

0.4g of the adsorbent were taken and added into different 50mg of the stock solution for acidic and alkaline samples respectively and were placed in the cyclone vibrator for proper agitation of the samples for varying time from 1 minute to 6 minutes before filtration and their respective absorbance were taken with the aid of the spectrophotometer.

3.2.3.4 EFFECT OF TEMPERATURE

0.4g of the adsorbent were taken and added into different 50mg of the stock solution for acidic and alkaline samples respectively and were placed in the magnetic stirrer for proper agitation of the samples under varying temperature from 30°C (room temperature) to 45°C before filtration and their respective absorbance were taken with the aid of the spectrophotometer.

CHAPTER FOUR
RESULTS AND DISCUSSIONS

The batch adsorption experiments were carried out at room temperature as outlined below; and percentage (%) removal of the dye by each adsorbent was calculated from the relation:

$$\frac{C_0 - C_e}{C_0} \times 100\%$$

Where C_0 and C_e are the concentrations (mg/L) of the dye initially and at equilibrium time [Muhammad *et al* 2012].

4.1 EFFECT OF INITIAL DYE CONCENTRATION

Adsorption experiments for the dye crystal violet were carried out by selecting a concentration range of 10 to 60mg/l with an adsorbent dosage of 0.4g.

Table 4.1: Effect of Initial Dye Concentration Values

Initial Dye Concentration (mg/l)	Absorbance (nm)
10	0.031
20	0.084
30	0.102
40	0.158
50	0.208
60	0.244

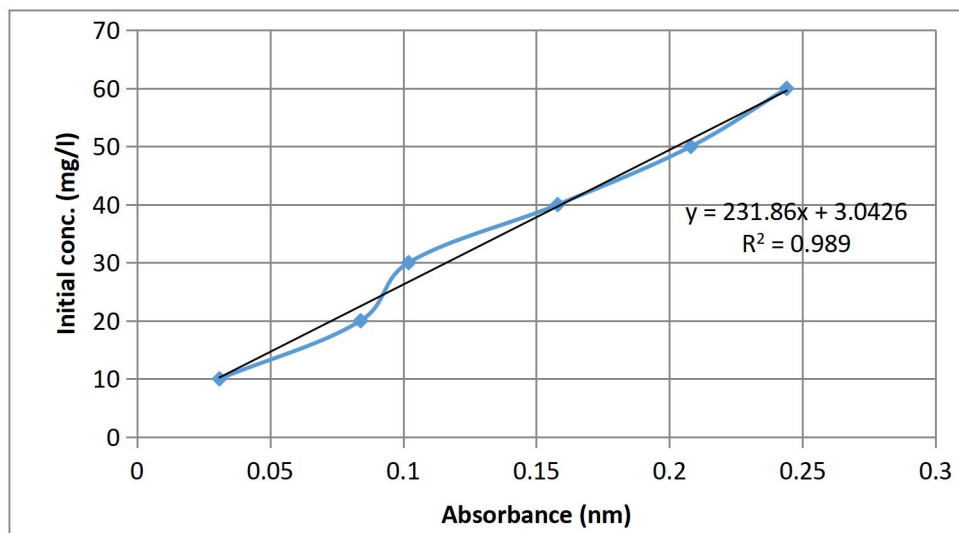


Figure 4.1: Plot of Effect of Initial Dye Concentration

The experimental results show that an increase in the concentration led to an increase in the amount of the dye adsorbed. An optimum concentration of 40mg/l and 60mg/l for both acid and alkaline adsorbents respectively of the dye solution were considered to provide the best result.

Table 4.2: Effect of Initial Concentration on Dye Removal Values

Initial concentration (mg/l)	Absorbance (nm) (Alkaline Treated SDAC)	Absorbance (nm) Acid Treated SDAC)	Dye Conc. (mg/l) (Alkaline Treated SDAC)	Alkaline Treated SDAC Dye Removal %	Dye Conc. (mg/l) Acid Treated SDAC)	Acid Treated SDAC Dye Removal %
10	0.028	0.013	9.53468	4.6532	6.05678	39.4322
20	0.035	0.011	11.1577	44.2115	5.59306	72.0347
30	0.025	0.020	8.8391	70.53633	7.6798	74.40066667
40	0.033	0.010	10.69398	73.26505	5.3612	86.597
50	0.036	0.016	11.38956	77.22088	6.75236	86.49528
60	0.026	0.044	9.07096	84.88173	13.24444	77.92593333

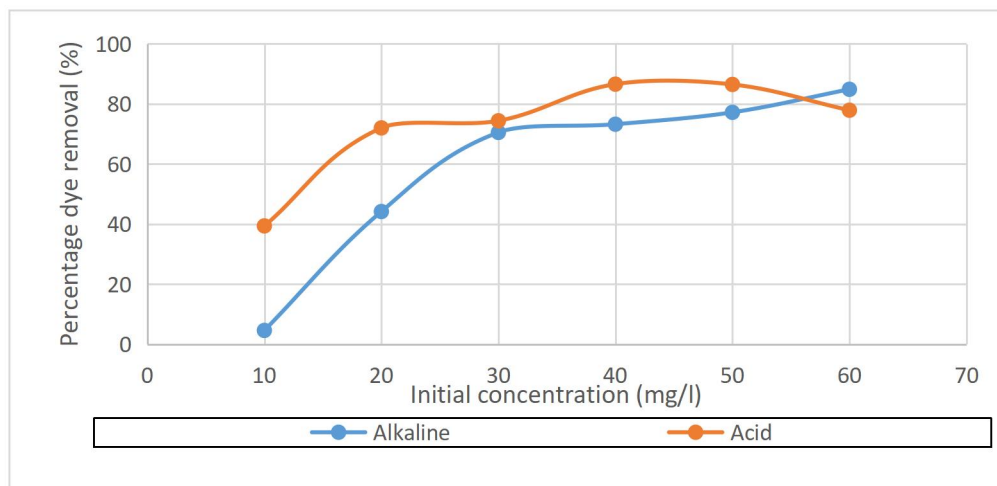


Figure 4.2: Plot of Effect of Initial Concentration on Dye Removal

4.2 EFFECT OF ADSORBENT DOSAGE

The study of the effect of amount of the adsorbents was necessary in order to observe the minimum possible amount, which shows maximum adsorption. The amounts of the adsorbents were varied from 0.05 to 0.5g. In this experiment, the adsorptive ability of the adsorbents at a definite concentration of g/100ml dye solution was considered. An increase in the amount of the adsorbents leads to an increase in the amount of the dye adsorbed. The quantitative results have been shown in the table below while the graphical presentation is provided in the figure below.

Table 4.3 Effect of adsorbent dosage values

Initial conc. (mg/l)	Adsorbent dosage (g/100ml)	Absorbance (nm) (Alkaline Treated SDAC)	Absorbance (nm) (Acid Treated SDAC)	Dye Conc. (mg/l) (Alkaline Treated SDAC)	Alkaline Treated SDAC Dye Removal %	Dye Conc. (mg/l) (Acid Treated SDAC)	Acid Treated SDAC Dye Removal %
50	0.05	0.05	0.056	14.6356	70.7288	16.02676	67.94648
50	0.1	0.043	0.054	13.01258	73.97484	15.56304	68.87392
50	0.2	0.017	0.024	6.98422	86.03156	8.60724	82.78552
50	0.3	0.034	0.016	10.92584	78.14832	6.75236	86.49528
50	0.4	0.035	0.018	11.1577	77.6846	7.21608	85.56784

50	0.5	0.048	0.02	14.17188	71.65624	7.6798	84.6404
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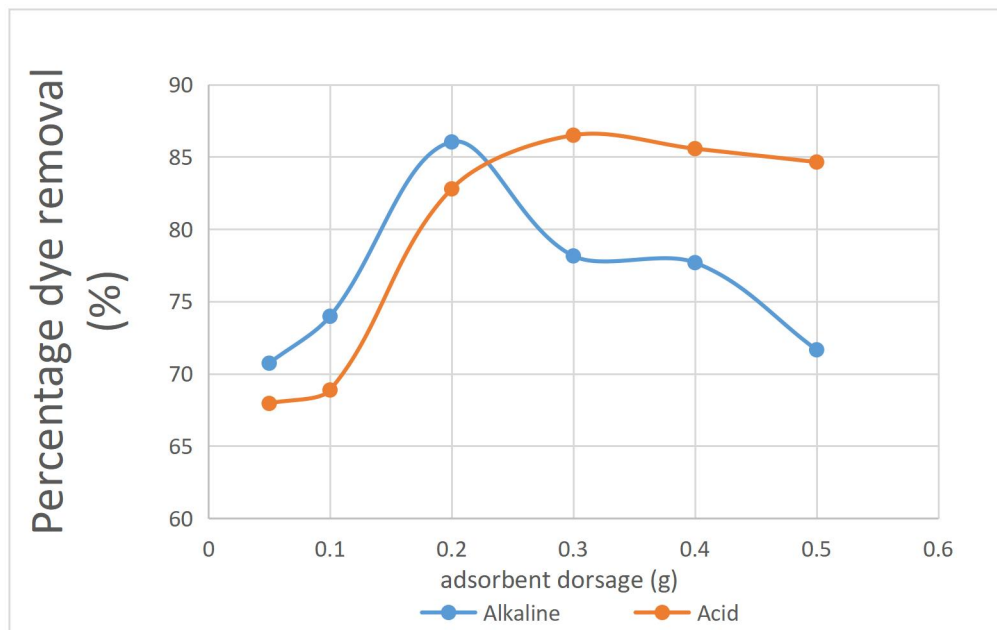


Fig 4.3 Plot of Effect of Adsorbent Dosage on Dye Removal

The results follow the expected pattern, in which the percentage adsorption of the dye varies from 67.9% to 86.5% for the acid treated SDAC and that of alkaline treated SDAC increases from 70.7% to 86.0% with increased adsorbent dosage. The percentage increase in CV dye removal is due to the increase in the number of adsorption site (surface area) or due to conglomeration of carbons at higher doses [Kannan 1991 and Phyto *et al* 2020]

4.3 EFFECT OF TEMPERATURE

The effect of temperature and the dye (CV) concentration on adsorption properties of the activated carbon is illustrated in table 4.4 and fig. 4.4.

Table 4.4 Varying temperature experimental table

Initial conc. (mg/l)	Temperature (°c)	Absorbance (nm) (Alkaline Treated SDAC)	Absorbance (nm) (Acid Treated SDAC)	Dye Conc. (mg/l) (Alkaline Treated SDAC)	Alkaline Treated SDAC Dye Removal %	Dye Conc. (mg/l) (Acid Treated SDAC)	Acid Treated SDAC Dye Removal %
50	30	0.032	0.027	10.46212	79.07576	9.30282	81.39436
50	35	0.096	0.016	25.30116	49.39768	6.75236	86.49528
50	40	0.058	0.015	16.49048	67.01904	6.52052	86.959
50	45	0.071	0.017	19.50466	60.99068	6.98422	86.03156

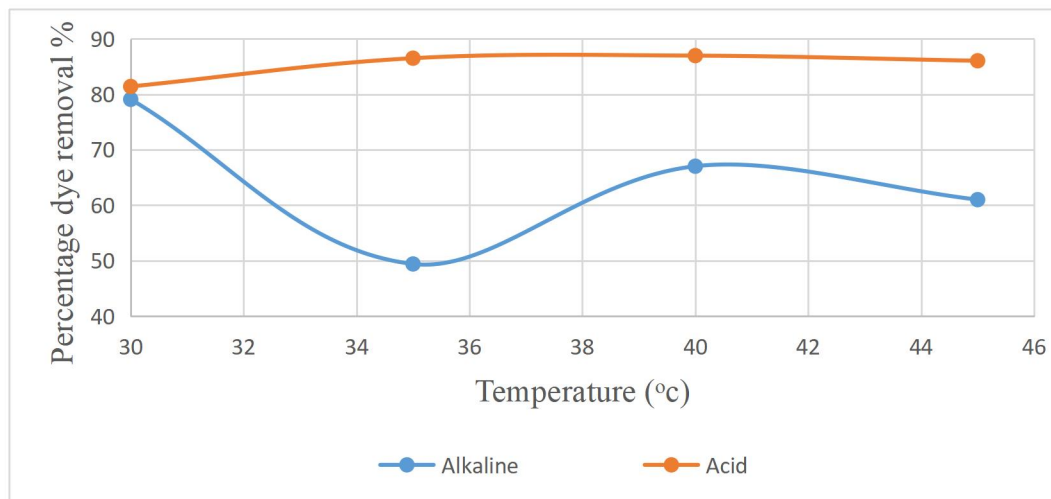


Figure 4.4: Plot of effect of temperature on dye removal

It can be seen from the above figure that the percentage of dye removal at adsorption is a temperature-dependent character. It increases and as well decreases with the temperature raise

which means that the adsorption process is endothermic. The number of binding sites for the dye molecules on the adsorbent surface may be increased by the temperature rise. In addition, the increase of the dye removal depending on the temperature can be explained by the increase of the mobility of the dye molecules [Tolga *et al* 2011].

4.4 EFFECT OF CONTACT TIME

The amount of the dye adsorbed at definite intervals of time (1 minute) was monitored for a fixed amount of adsorbent (0.4g) at a particular concentration 50mg/l for acid and alkaline treated SDAC.

Table 4.5: Values of contact time experiment

Initial conc. (mg/l)	Time (minutes)	Absorbance (nm) (Alkaline Treated SDAC)	Absorbance (nm) (Acid Treated SDAC)	Dye Conc. (mg/l) (Alkaline Treated SDAC)	Alkaline Treated SDAC Dye Removal %	Dye Conc. (mg/l) (Acid Treated SDAC)	Acid Treated SDAC Dye Removal %
50	1	0.083	0.03	22.28698	55.42604	9.9984	80.0032
50	2	0.019	0.039	7.44794	85.10412	4	75.82972
50	3	0.018	0.048	7.21608	85.56784	8	71.65624
50	4	0.022	0.011	8.14352	83.71296	5.59306	88.81388
50	5	0.016	0.024	6.75236	86.49528	8.60724	82.78552
50	6	0.017	0.024	6.98422	86.03156	8.60724	82.78552

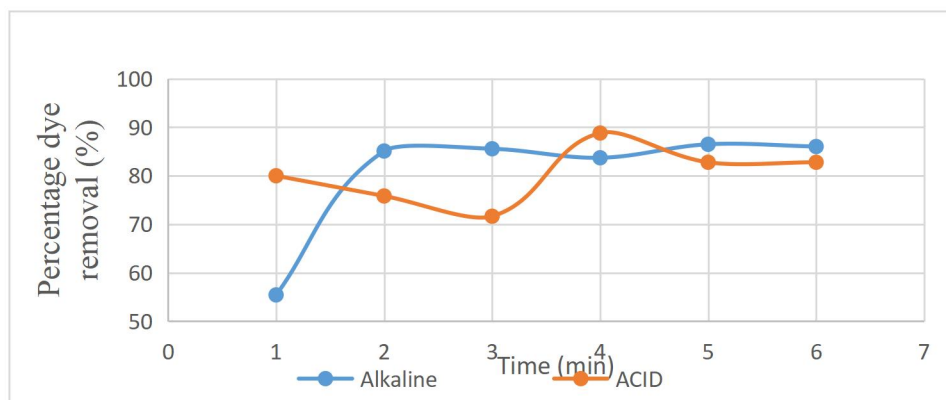


Fig. 4.5 Graph of effect of contact time on dye removal

From the plot of effect of contact time on CV dye removal, the increase in time leads to a corresponding increase in the adsorption rate of the dye. This is due to the fact that more molecules of the CV are adsorbed by the activated carbon. The percentage of CV dye removal increases from 55.4% to 86.5% for the alkaline treated SDAC while that of acid treated SDAC increases from 71.7% to 88.8%.

4.5 ADSORPTION ISOTHERMS

In general, experimental isotherm is useful for describing adsorption capacity to facilitate evolution of the feasibility of the process for a given application, for selection of most appropriate adsorbent and for preliminary determination of adsorbent dose requirements.

The two isotherms that will be discussed in this study are;

- i. Langmuir Isotherm
- ii. Freundlich Isotherm

4.5.1 LANGMUIR ISOTHERM

The Langmuir isotherm is most frequently used to represent the data of adsorption from solution. The isotherm studied was carried out for optimum condition, which was obtained. The Langmuir isotherm assumes that the adsorption takes place at homogeneous sites, all sites are equivalent and there are no interactions between adsorbate molecule and adjacent sites. The adsorption data were analyzed according to the linear form of

$$\frac{C_e}{q_e} = \frac{1}{Q_0 b} + \frac{C_e}{Q_0}$$

Where, C_e is equilibrium constant of dye (mg/l), q_e is amount of dye adsorbed at equilibrium (mg/g), Q_0 is Langmuir constant related to adsorption capacity (mg/g), b is Langmuir constant related to energy of adsorption capacity (l/mg).

Table 4.5.1A Langmuir table of values with Alkaline Treated SDAC

Adsorbent dosage (g/100ml)	Initial Conc. (mg/l)	Final dye Conc. (mg/l), C_e	Dye adsorbed (mg/l), q_e	C_e/q_e
0.05	50	14.6356	35.3644	0.41385122
1	50	13.01258	36.9874	0.35181097
2	50	6.98422	43.0158	0.16236414
3	50	10.92584	39.0742	0.27961804
4	50	11.1577	38.8423	0.28725642
5	50	14.17188	35.8281	0.39555187

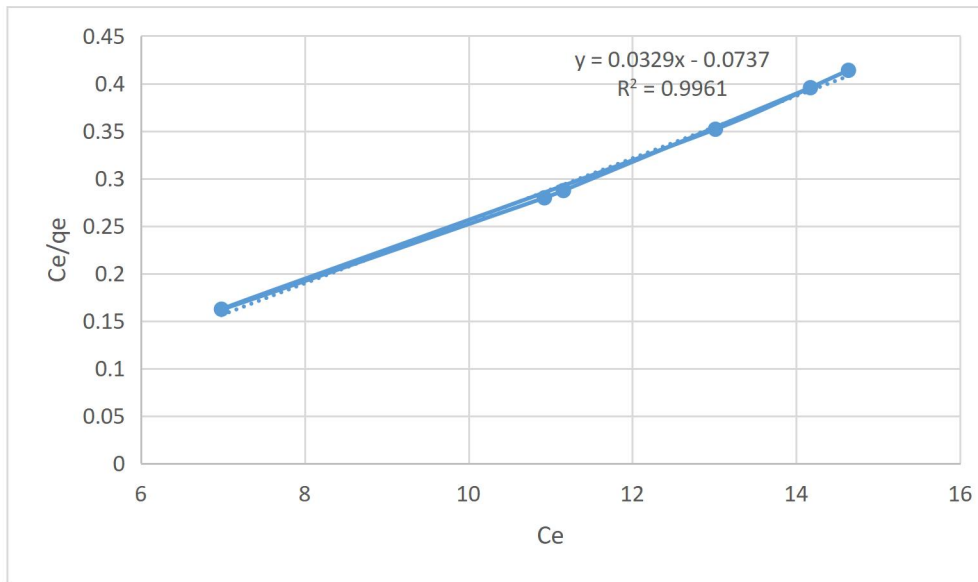


Fig 4.5.1A: Langmuir plot for the removal of CV onto Alkaline Treated SDAC

Table 4.5.1B: Langmuir table of values with Acid Treated SDAC

Adsorbent dosage (g/100ml)	Initial Conc. (mg/l)	Final dye Conc. (mg/l), C_e	Dye adsorbed (mg/l), q_e	C_e/q_e
0.05	50	16.02676	33.97324	0.47175
0.1	50	15.56304	34.43696	0.45193
0.2	50	8.60724	41.39276	0.20794
0.3	50	6.75236	43.24764	0.15613
0.4	50	7.21608	42.78392	0.16866
0.5	50	7.6798	42.3202	0.18147

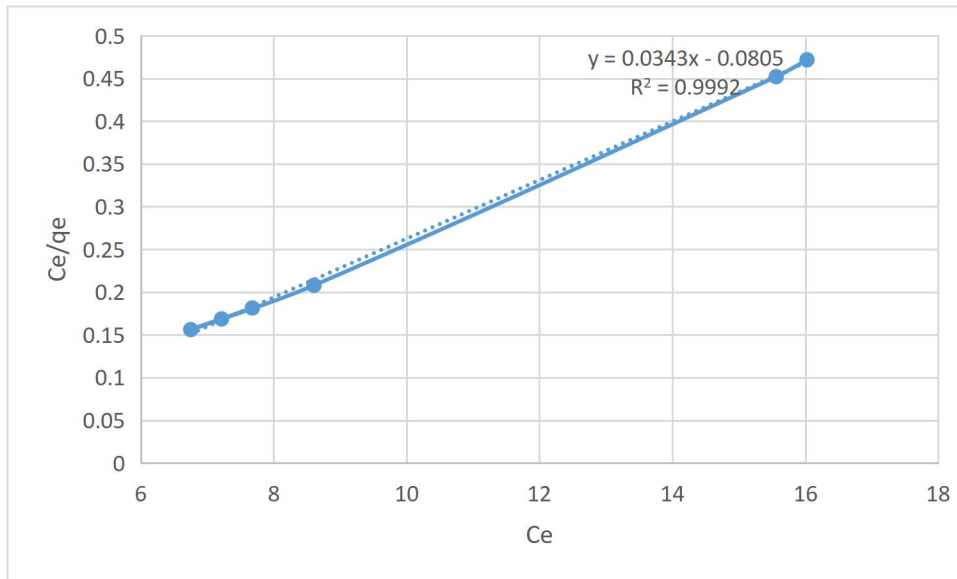


Fig 4.5.1B: Langmuir plot for the removal of CV onto Acid Treated SDAC

From the Langmuir plots of acid and alkaline treated SDAC, one can analyze the equilibrium data and hence calculate each parameter in the standard equations, the table below shows the values of each parameter.

Table 4.5.1C Langmuir table of values.

Alkaline Treated SDAC				Acid Treated SDAC			
R ²	R _L	K _L (l/mg)	Q _o (mg/g)	R ²	R _L	K _L (l/mg)	Q _o (mg/g)
0.9961	0.0429	0.4464	30.3951	0.9992	0.0448	0.4261	29.1545

The table above depicts that the determination coefficient value, R² for the acid treated SDAC best fit the Langmuir isotherms since its value is 0.9992, with maximum adsorption capacity, Q_o to be 29.1545mg/g and the dimensionless separation parameters, R_L was found to be more favorable with acid treated SDAC with value 0.0448 [Phyo *et al* 2020]

4.5.2 FREUNDLICH ISOTHERM

Freundlich isotherm model was chosen to estimate the adsorption intensity of the adsorbate on the adsorbent surface²⁴. Linear form of Freundlich model was expressed by

$$\text{Log } q_e = \text{Log } K_f + \frac{1}{n} \text{Log } C_e$$

where, q_e is dye concentration in solid at equilibrium (mg/g), C_e is dye concentration in solution at equilibrium (mg/L), K_f is the Freundlich isotherm constant related to adsorption capacity (L/mg) and n is the Freundlich isotherm constant related to adsorption intensity.

Table 4.5.2A: Freundlich table of values with Alkaline Treated SDAC

Initial Conc. (mg/l)	Final dye Conc. (mg/l), C _e	Dye adsorbed (mg/l), q _e	In C _e	In q _e
50	14.6356	35.3644	2.68346	3.56571
50	13.01258	36.9874	2.56592	3.61058
50	6.98422	43.0158	1.94365	3.65157
50	10.92584	39.0742	2.39113	3.66546
50	11.1577	38.8423	2.41213	3.65951
50	14.17188	35.8281	2.65126	3.57873

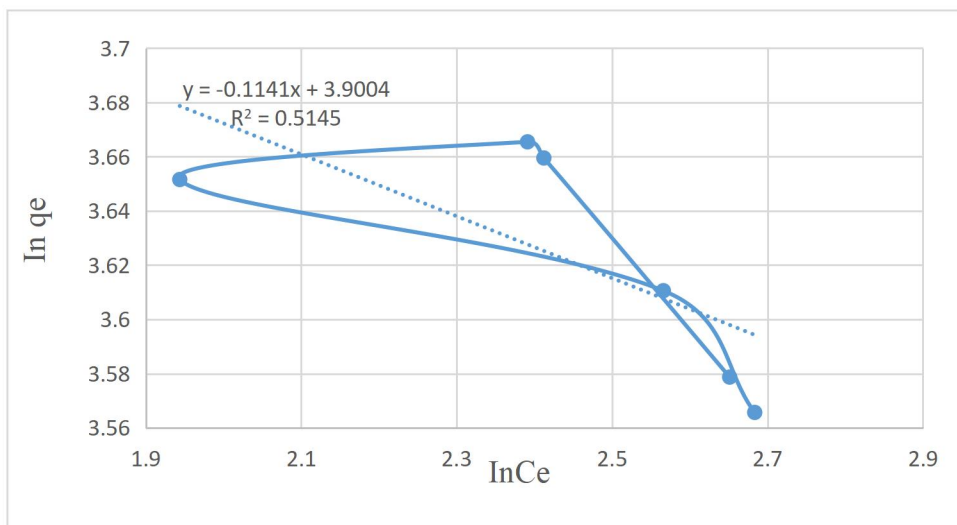


Fig 4.5.2A: Freundlich plot for the removal of CV onto Alkaline Treated SDAC

Table 4.5.2B: Freundlich table of values with Acid Treated SDAC

Initial Conc. (mg/l)	Final dye Conc. (mg/l), C_e	Dye adsorbed (mg/l), q_e	In C_e	In q_e
50	16.02676	33.9732	2.77426	3.52557
50	15.56304	34.437	2.7449	3.53913
50	8.60724	41.3928	2.1526	3.72311
50	6.75236	43.2476	1.90989	3.76694
50	7.21608	42.7839	1.97631	3.75616
50	7.6798	42.3202	2.03859	3.74526

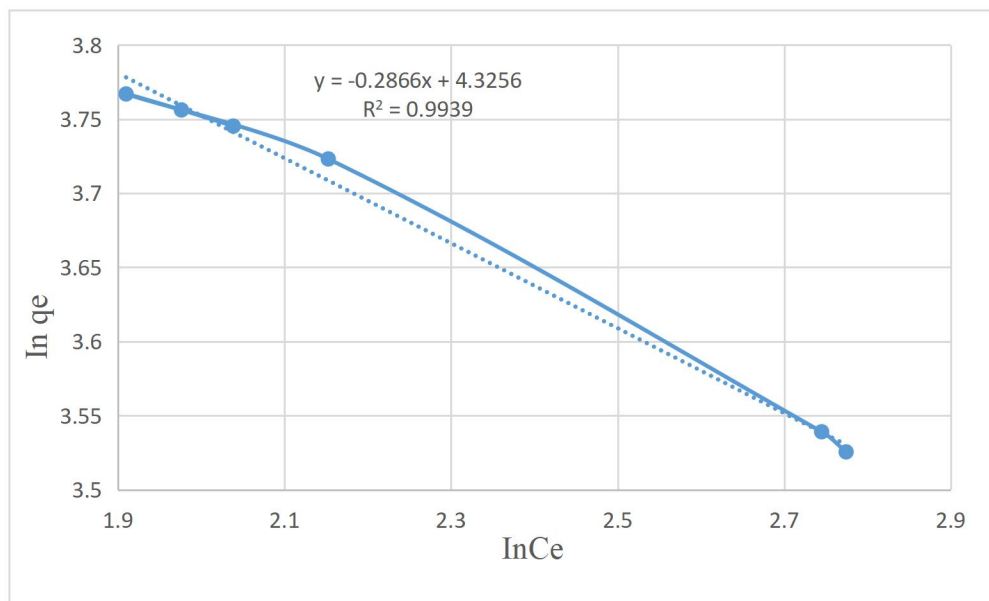


Fig 4.5.2B: Freundlich plot for the removal of CV onto Acid Treated SDAC

From the Freundlich plots, the following are the values of parameters obtained

Table 4.5.2C: Freundlich table of values.

Alkaline Treated SDAC			Acid Treated SDAC		
R ²	1/n	K _F	R ²	1/n	K _F
0.5145	0.1141	49.4222	0.9939	0.2866	75.6109

In general, the Langmuir isotherm is best fit for this equilibrium data since its values for the acid and alkaline SDAC are mostly approximately 1 when compared to the values obtained from the Freundlich isotherm under adsorption isotherms.

CHAPTER FIVE

CONCLUSSION AND RECOMMENDATIONS

5.1 CONCLUSIONS

- From the performed experiment, it was found that sawdust is an effective raw material in the production of activated carbon.
- Additionally, the rate of removal of this dye is higher when the carbon is activated with an acid when compared to that of alkali.
- The more the adsorbent dosage, the higher the adsorption of the crystal violet provided the concentration is constant.
- Increase in temperature of samples lead to a corresponding increase in the percentage removal of CV dye.
- The percentage removal of CV dye increases with increase in contact time the activated carbon had with the CV solution
- the result gotten shows that the Langmuir isotherm best fit the adsorption of CV onto the activated carbon since the determination coefficient value, R^2 is higher when compared to that gotten from the freundlich isotherm.

5.2 RECOMMENDATIONS

- It is advisable to use adsorption process in the removal of crystal violet due to efficacy, economy and ability to separate a wide range of chemical compounds and simple procedure.
- The use of sawdust in the production of carbon for the adsorption of CV should be greatly utilized by the textile industry.
- The use of acid activating agent should be use in treating carbon when the dye to be removed is a basic dye.
- Waste water should be properly treated before being discharged to the environment.

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APPENDIX

ABBREVIATIONS

CV

SDAC

MEANING

crystal violet

sawdust activated carbon

CALCULATION

1. Yield of activated carbon

$$Yield = \frac{W_1}{W_2} \times 100\%$$

Where W_1 is the mass of the unwashed carbon after activation = 135 and

W_2 is the original mass of the precursor (sawdust) on dry basis = 474.

$$Yield = \frac{135}{474} \times 100\%$$

Therefore, the Yield = 28.48%