

**ADSORPTION OF AMMONIUM PURPURATE USING COCONUT
SHELLCHARCOL AS BIOCHAR**

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BY
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PAVANATMA COLLEGE, MURICKASSERY**

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This is to certify that the project entitled “**ADSORPTION OF AMMONIUM PURPURATE USING COCONUT SHELL CHARCOL AS BIOCHAR**” is a bonafide work carried out by Miss **Ms. AKHILA SUKUMARAN** (Reg. NO: **190011010495**) under guidance of Dr. **ANEESH MATHEW**, PG Department of Chemistry, Pavanatma College, Murickassery, for partial fulfillment of their requirement for the award of Degree of Master of Science in Chemistry of Mahatma Gandhi University during the year 2019-2021.

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DECLARATION

I, **AKHILA SUKUMARAN**, do hereby declare that this dissertation entitled “**ADSORPTION OF AMMONIUM PURPURATE USING COCONUT SHELL CHARCOL AS BIOCHAR**” is a bonafide work carried out by me during **2019-2021** at Pavanatma College, Murickassery under the supervision and guidance of **Prof. Dr. Surendran Parambadath** and **Dr. Aneesh Mathew**, and no part therefore has been submitted for the award of any degree, diploma or recognition of university.

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CHAPTER - 1

INTRODUCTION

Biochar is the char coproduct from the thermochemical processing of biomass utilized as a soil amendment and/or carbon sequestration agent. Processes that produce biochar include pyrolysis (heating without oxygen) and gasification; other coproducts include heat, electricity, bio-oil, and syngas. Interest in biochar soil applications originally stemmed from the long-term fertility of terra preta anthropogenic soils in the Brazilian Amazon. More recently, the recalcitrance of biochar carbon has attracted international attention as an inexpensive and effective way to sequester atmospheric carbon for centuries to millennia while simultaneously producing carbon-negative energy and improving soil quality. Current research focuses on relationships between feedstock, reaction conditions, biochar properties, soil and crop responses to biochar applications, and biochar economics.

Biochar has drawn remarkable consideration for soil improvements due to their enhanced soil fertility, nutrient immobilization and slow-release, carbon storage, which are some of the handful of benefits for soil amendments made with biochar. These enhanced properties are a result of the cumulative effects arising from the changes in the microbial activities in soils along with the agrochemical application. The community structure of the soil biota alters, nutrients transformation rates changes, and so is the release of nutrients from the applied agrochemicals. This chapter reviews the linkages between the biochar applications in soils and the available nutrients for plant growth arising from the agrochemical applications to soils. Furthermore, the alteration in mechanisms of nutrient uptake for food crops growth has also been reviewed upon biochar amendments in soils. Biochar significantly affects the soil conditions and plant growth, which needs further understanding. The chapter gives insights to future directions with regard to the implications, drawbacks of using biochar as soil amendments for an effective biochar–plant nutrient interaction.

Process of biochar production had three stages: pre-pyrolysis; main-pyrolysis and formation of carbonaceous soil products. Evaporation of moisture and light volatiles is responsible for the first stage (from ambient temperature to 200 °C). The evaporation of moisture produces bond breaks and the creation

of hydroperoxide, $-\text{COOH}$, and $-\text{CO}$ groups. The second stage (from 200 to 500 °C) involved rapid devolatilization and decomposition of hemicelluloses and cellulose. Degradation of lignin and other organic materials with stronger chemical bonds occurs at a higher temperature (over 500 °C). Changes in the structure and physicochemical properties of biochar are significantly associated with pyrolysis temperature. Biochar is usually produced as a cost-effective and environmentally friendly sorbent. The ability of biochar in stabilizing organic and mineral compounds is due to its physicochemical properties such as porous structure, expanded specific surface area, high organic carbon content, active functional groups, and also high cation-exchange capacity (CEC). Pyrolysis is usually the method of choice to produce biochar, though biomass gasification produces a smaller char yield compared with pyrolysis. The production of biochar using pyrolysis process is influenced by the source of biomass, the properties (particle size and moisture content) and composition (contents of cellulose, lignin, and ash) of biomass, and also the process parameters (temperature, heating rate, pressure, and residence time). In order to produce a high char yield, a low-temperature and low-heating-rate process is more preferred. The characteristics of biochar are mainly determined by the type of biomass used and the parameters of pyrolysis process. By knowing the effect of each parameter on the biochar properties, the production of biochar with a desired performance can be tailored. As reported by Angin, temperature is the only pyrolysis parameter that can control the elemental compositions of biochar and their atomic ratios. The type of biomass chosen to undergo pyrolysis process plays an important role in determining the status of biochar. Biomass chemical compositions and its size, shape, and structure can significantly affect the sorption capacity of biochar. Agricultural wastes that are locally available can be considered as a low-cost and renewable pyrolysis feedstock. By converting this biomass into the biochar, the residues are able to be utilized in a sustainable way.

Benefits of making Biochar

They also produce large quantities of smog precursors such as ammonia and the SO_x (sulphur oxides) that react with sunlight to form smog. Finally, that smoke that blocks the sun is $\text{PM}_{2.5}$ – particulate matter so small that it passes through the walls of the lungs into the bloodstream to wreak havoc throughout the body. Crop waste eliminated through the process of making biochar produces no smoke. Every year, farmers in the developing world burn more than 10 billion tonnes of crop wastes in their fields. This releases

16.6 billion tonnes of CO₂, 11.2 billion tonnes of CO₂, 1.1 billion tonnes of smog precursors and 65.7 million tonnes of PM_{2.5} into the atmosphere. The combined annual CO₂ and CO_{2e} emissions from crop waste burning are equivalent to the annual emissions of 714 coal fired power plants. The alternative – converting the waste into biochar instead of burning it removes three tons CO₂ from the atmosphere for every ton produced; when added to fields as a soil amendment, that carbon is permanently sequestered. The long term benefits of making biochar is a huge reduction of greenhouse gases that contribute to global warming.

The use of Biochar

While it may be invaluable for farmers, it has many other practical uses too. The absorption qualities of biochar make it a perfect solution for odor control, useful for eliminating unwanted odors: in cars, homes, compost piles, pet odors, closets, bathrooms, even in stinky old sneakers. The powerful moisture adsorption quality makes it extremely helpful in cutting down mildew in damp areas. Look for ways to enrich your life with biochar, and help build a market to encourage widespread manufacturing of this simple, yet amazing natural product. It could help save the world.

Cation Exchange

Biochar can exchange its own cations (e.g., H⁺, K⁺, Na⁺, and Ca²⁺) with heavy metals and therefore form a chemical bond to retain the heavy metals on its surface. The exchangeable cations of biochar come from two fractions: H⁺ from its acidic functional groups such as carboxyl and phenolic groups; and alkaline minerals (e.g., K₂CO₃/K₂O) in biochar, which is formed during production.

The heavy metals adsorbed on biochar through cation exchange are regarded as a readily bioavailable fraction, which can be easily uptaken by plants (Filgueiras et al., 2002). They may also desorb from biochar and pose environmental risks when pH significantly changes. Therefore, the adsorption of heavy metals on biochar through cation exchange may not be suitable for immobilizing heavy metals in contaminated soils in the long term. However, as the exchangeable heavy metals on biochar are relatively easily desorbed by changing pH or compulsive exchange (Gillman and Sumpter, 1986), this adsorption mechanism has the potential to be applied in water treatment due to reusability, as with physical adsorption.

Uses

Charcoal has been used since earliest times for a large range of purposes including art and medicine, but by far its most important use has been as a metallurgical fuel. Charcoal is the traditional fuel of a blacksmith's forge and other applications where an intense heat is required. Charcoal was also used historically as a source of black pigment by grinding it up. In this form charcoal was important to early chemists and was a constituent of formulas for mixtures such as black powder. Due to its high surface area charcoal can be used as a filter, and as a catalyst or as an adsorbent.

1.1 COCONUT SHELL CHARCOAL

Coconut is a fruit, obtained from the coconut palm, the most cultivated palm in the world. The leading producers are the Philippines, Indonesia and India. In its growing stage, it can reach a height of approximately 25 meters. Activated carbon is obtained from the coconut shell, in which it goes through physical or chemical activation processes.

Coconut shell activated charcoal tends to be microporous and adsorbs more efficiently the low molecular weight organic pollutants that are most present in well water. All activated carbon of vegetable or mineral origin contains inorganic salts and elements, some of which are soluble in water

What is Coconut Shell Activated Charcoal?

Coconut is a fruit, obtained from the coconut palm, the most cultivated palm in the world. The leading producers are The Philippines, Indonesia and India. In its growing stage, it can reach a height of approximately 25 meters. Activated carbon is obtained from the coconut shell, in which it goes through physical or chemical activation processes.

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How activated coconut shell charcoal is prepared?

Coconut shell charcoal comes from the coconut shells. It is ecological since the husks of the small coconuts are used that would otherwise go to waste. To activate it, the coconut shell is heated to more than 1000 degrees Celsius without the presence of oxygen. Activated means that it has its 100% adsorption characteristic and maximum porosity.

Activated carbon works by using the adsorption process. Do not confuse absorption with adsorption. Adsorption is a process whereby atoms, ions, gas molecules, liquids or dissolved solids are trapped or retained on a surface while absorption is a phenomenon of volume.

Composition:

Coconut activated carbon is composed of 70 to 80% carbon, is practically pure, and the ash content varies between 5 to 10%.

Use:

Activated coconut shell charcoal has multiple applications. It is used to treat acute poisoning in people and for gastrointestinal problems. It is also used for water purification, deodorization and purification of air and water, for the removal of organic substances, solvent recovery and as a catalyst.

Benefits of Using Activated Coconut Shell Charcoal:

1. Natural Detoxificant

Coconut shell charcoal helps in detoxification processes. It is used as one of the ingredients in colon cleansing therapies and detoxifying products. It can absorb toxins and eliminate them intestinally so that it will be an essential cleaning system.

2. Treats Poisoning

Activated charcoal is used as an adsorbent agent to treat poisoning and overdose by oral ingestion. Prevents from absorbing poison in the stomach. Coal absorbs the toxins from the stomach and intestine.

3. Helps Reduce Cholesterol Levels

Activated carbon reduces the levels of cholesterol, lipids and triglycerides in the blood.

4. Stomach Relief

Relieves the stomach by removing excess gas from the digestive tract.

5. Rejuvenating Effect

It is also used to counteract the results of the passage of time in the body since it assists in the processes of the liver, kidneys and adrenal glands avoiding excessive cell aging.

6. Skin Care

Activated coconut charcoal has been lately used in many beauty products. This is because it is effective in eliminating bacteria, chemicals and impurities. It is regulatory sebum and very useful in cases with acne and skin blemishes. But they also use it to control LDL cholesterol, reduce flatulence and promote renal function by reducing the number of waste products that the kidneys must filter.

Like everything, they have their precautions. It should not be taken within two hours after administration of vitamins, medications or supplements, as it will prevent the body from absorbing them.

Activated Coconut Charcoal Is Odorless, Tasteless and Non-Toxic.

It is an ingredient that we love and use in almost all our beauty and personal care products are activated carbon. In recent years it has become a trend and controversy at the same time. However, Ayurvedic medicine used this black powder for thousands of years. Its first registered use dates back to 1550 BC. It is also well established in medical literature as a powerful antidote that adsorbs most toxins.

Activated carbon is a substance, usually bamboo, wood, coal, or coconut shell that is activated with high temperatures and an inevitable process. It is essential to know that activated coconut shell charcoal is not the same coal used for the grill or charred wood of fire. Please do not try to replace or do it yourself.



Figure 1. Schematic representation of charcoal from coconut shell

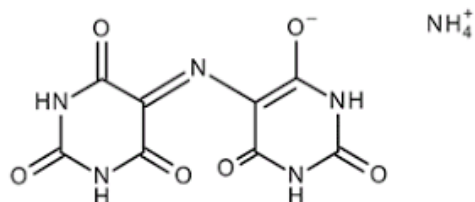
1.2 DYE

A dye is a colored substance that chemically bonds to the substrate to which it is being applied. This distinguishes dyes from pigments which do not chemically bind to the material they color. The dye is generally applied in an aqueous solution, and may require a mordant to improve the fastness of the dye on the fiber.

Both dyes and pigments are colored, because they absorb only some wavelengths of visible light. Dyes are usually soluble in water whereas pigments are insoluble. Some dyes can be rendered insoluble with the addition of salt to produce a lake pigment. The color of a dye is dependent upon the ability of the substance to absorb light within the visible region of the electromagnetic spectrum (380-750 nm). An earlier theory known as Witt theory stated that a colored dye had two components, a chromosphere which imparts color by absorbing light in the visible region (some examples are nitro, azo, quinoid groups) and an auxochrome which serves to deepen the color. This theory has been superseded by modern electronic structure theory which states that the color in dyes is due to excitation of valence π -electrons by visible light.

MUREXIDE

Murexide ($\text{NH}_4\text{C}_8\text{H}_4\text{N}_5\text{O}_6$, or $\text{C}_8\text{H}_5\text{N}_5\text{O}_6 \cdot \text{NH}_3$), also called ammonium purpurate or MX, is the ammonium salt of purpuric acid. It is a purple solid that is soluble in water. The compound was once used as an indicator reagent.^[1] Aqueous solution is yellow at low pH, reddish-purple in weakly acidic solutions, and blue-purple in alkaline solutions.



Molecular Formula	C ₈ H ₈ N ₆ O ₆
Synonyms	Murexide Ammonium purpurate 3051-09-0 MFCD00012777 Murexide, p.a., ACS reagent Molecular Weight -284.1

Computed Properties

Property Name	Property Value
Molecular Weight	284.19
Hydrogen Bond Donor Count	6
Hydrogen Bond Acceptor Count	8
Hydrogen Bond Acceptor Count	1
Rotatable Bond Count	284.05053200
Exact Mass	284.05053200
Monoisotopic Mass	167 Å ²
Topological Polar Surface Area	20
Heavy Atom Count	0
Formal Charge	581
Complexity	0
Isotope Atom Count	0
Defined Atom Stereocenter Count	0
Undefined Atom Stereocenter Count	0
Defined Bond Stereocenter Count	0
Undefined Bond Stereocenter Count	2

PREPARATION

Murexide is prepared by treating alloxantin with ammonia to 100 °C, or by treating uramil (5-aminobarbituric acid) with mercuryoxide. It may also be prepared by digesting alloxan with alcoholic ammonia.

USES

Murexide is used in analytical chemistry as a complexometric indicator for complexometric titrations, most often of calcium ions, but also for Cu, Ni, Co, Th and rare-earth metals. It functions as a tridentate ligand.

1.3 ADSORPTION

Adsorption is the adhesion of atoms, ions or molecules from a gas, liquid or dissolved solid to a surface. This process creates a film of the adsorbate on the surface of the adsorbent. This process differs from absorption, in which a fluid (the absorbate) is dissolved by or permeates a liquid or solid (the *absorbent*). Adsorption is a *surface phenomenon*, while absorption involves the whole volume of the material, although adsorption does often precede absorption. The term sorption encompasses both processes, while desorption is the reverse of it.

Like surface tension, adsorption is a consequence of surface energy. In a bulk material, all the bonding requirements (be they ionic, covalent or metallic) of the constituent atoms of the material are fulfilled by other atoms in the material. However, atoms on the surface of the adsorbent are not wholly surrounded by other adsorbent atoms and therefore can attract adsorbates. The exact nature of the bonding depends on the details of the species involved, but the adsorption process is generally classified as physisorption (characteristic of weak van der Waals forces) or chemisorptions (characteristic of covalent bonding). It may also occur due to electrostatic attraction.

Adsorption is present in many natural, physical, biological and chemical systems and is widely used in industrial applications such as heterogeneous catalysts, activated charcoal, capturing and using waste heat to provide cold water for air conditioning and other process requirements (adsorption chillers), synthetic resins, increasing storage capacity of carbide-derived carbons and water purification. Adsorption, ion exchange and chromatography are sorption processes in which certain adsorbates are selectively transferred from the fluid phase to the surface of insoluble, rigid particles suspended in a vessel or packed in a column. Pharmaceutical industry applications, which use adsorption as a means to prolong neurological exposure to specific drugs or parts thereof, are lesser known.

PHYSISORPTION

Physical adsorption results from interactions between subcritical fluid species and nearly any solid surface. The measurements are made by a variety of well-developed techniques and interpreted by using ever more sophisticated models. Physical adsorption experiments probe thermodynamic phase equilibria between bulk fluid phases and adsorbed phases, which progress from single, isolated molecules to a single

layer of molecules on the surface (a monolayer) to multilayers to condensation (or sublimation). Analyses of equilibrium data characterizing the adsorption of physisorbing gases are commonly employed to estimate the morphology of the sample, including the total surface area, the distribution of the dimensions of any pores (ranging in diameter from about 0.1 to 50 nm), and the total pore volume/void fraction. These analyses are employed to guide understanding of the influence of morphology on sorption, separations, and catalysis. Considerable progress has been made in the last several decades in investigations of physical adsorption on high-surface-area solids, both experimentally and theoretically (1–8), such that we now understand the phenomena associated with sorption far better than we ever have. Furthermore, materials synthesis has developed to such an extent that we can now produce materials possessing very high surface areas ($> 1000 \text{ m}^2 \text{ g}^{-1}$ of solid) or with uniform pores in the range 1–20 nm, or even solids with multiscale porosity comprising a network of pores of one size embedded within a network of pores of another dimension and/or connectivity. Physical adsorption (physisorption) is then employed to characterize, design, and optimize the morphology of the material for specific applications.

Chemisorption

Chemisorption is a kind of adsorption which involves a chemical reaction between the surface and the adsorbate. New chemical bonds are generated at the adsorbant surface. Examples include macroscopic phenomena that can be very obvious, like corrosion, and subtler effects associated with heterogeneous catalysis, where the catalyst and reactants are in different phases. The strong interaction between the adsorbate and the substrate surface creates new types of electronic bonds.

In contrast with chemisorption is physisorption, which leaves the chemical species of the adsorbate and surface intact. It is conventionally accepted that the energetic threshold separating the binding energy of "physisorption" from that of "chemisorption" is about 0.5 eV per adsorbed species.

Due to specificity, the nature of chemisorption can greatly differ, depending on the chemical identity and the surface structural properties. The bond between the adsorbate and adsorbent in chemisorption is either ionic or covalent.

1.4 Factors Affecting Adsorption

Adsorption occurs on the surface of almost all solids. However, the extent of adsorption of a gas on the surface of a solid depends upon the following factors:

(i) Nature and surface area of the adsorbent

(ii) Nature of the adsorbed gas

(iii) Temperature

(iv) Pressure of the gas

Let us now discuss these factors briefly.

(i) Nature and Surface Area of the Adsorbent

Different solids would adsorb different amounts of the same gas even under similar conditions. Substances like charcoal and silica gel are excellent adsorbents. The substances that are porous in nature and have rough surfaces are better adsorbents.

The extent of adsorption also depends upon the surface area of the solid. Greater the surface area, more is the surface available for adsorption and greater is the adsorption. The surface area depends upon the particle size of the substance. A cube of each side equal to 1cm has six faces. Each of them is a square with surface area of 1cm^2 . Thus, the total surface area of this cube is 6 cm^2 . If its each side is divided into two equal halves, $\frac{1}{2}\text{ cm}$ long, and the cube is divided into two equal halves, $\frac{1}{2}\text{ cm}$ long, and the cube is cut along the lines indicated in the Fig (b), the cube would be divided into 8 smaller cubes with each side 0.5 cm long. Surface area of each small cube would be $(6 \times 0.5 \times 0.5) = 1.5\text{ cm}^2$ and the total surface area of all the 8 smaller cubes would be 12 cm^2 which is double the surface area of the original cube. If it is subdivided into smaller cubes, each of side equal to $1 \times 10^{-6}\text{ cm}$ the surface area will increase to $6 \times 10^6\text{ cm}^2$ or 600 m^2 . The increase in surface area would result in greater adsorption.

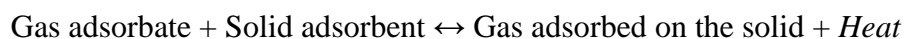
(ii) The Nature of the Adsorbed Gas

The extent of adsorption also depends upon the nature of the gas. The gases which are more easily liquifiable or are more soluble in water are more readily adsorbed than others. For example, under similar conditions, the amount of SO₂ or NH₃ adsorbed by charcoal is much more than that of H₂ or O₂ gases. It is because the intermolecular forces are stronger in more easily liquifiable gases, therefore, they get adsorbed more strongly.

(iii) Temperature

The extent of adsorption decreases with rise in temperature. For example, under one atmosphere pressure, one gram of charcoal adsorbs about 10 cm³ of N₂ gas at 272 K, 20 cm³ at 248 K and 45 cm³ at 195 K.

Adsorption is an exothermic process. The change in enthalpy when one mole of a substance is adsorbed, is called enthalpy of adsorption. The adsorption process is similar to the condensation process. The reverse process is called desorption and is endothermic in nature. It is similar to the evaporation process. When a gas is kept in contact with a solid adsorbent in a closed container, a dynamic equilibrium is established in due course of time.



Since the forward process (adsorption) is exothermic in nature, according to the Le Chatelier's principle, it would be favoured at low temperature. Therefore, the extent of adsorption would increase on decreasing the temperature and would decrease on increasing the temperature.

(iv) Pressure of the gas

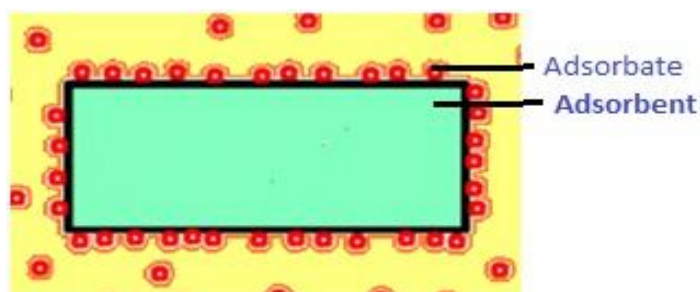
At a constant temperature the extent of adsorption increases with increase in the pressure of the gas (adsorbate). We shall study the relation between the two in detail a little later

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1.5 Mechanism of Adsorption

- Adsorption occurs because the particle on the surface and the particle in the bulk of the adsorbent are not in the same environment. That is, the net force acting on them is not the same.
- The particle on the surface has unbalanced forces acting on it which are also called residual attractive forces
- Due to these forces, the surface particles of the adsorbent attract the adsorbate particles



- During adsorption, there is always a decrease in the residual attractive forces of the surface. That is, the energy of the surface decreases and this appears as heat. This is called the heat of adsorption
- The amount of heat evolved when one mole of adsorbate is adsorbed on the adsorbent surface is called enthalpy of adsorption
- Adsorption is always exothermic and the enthalpy change, ΔH is always negative
- When the adsorbate molecules are adsorbed on the surface of the adsorbent, their freedom of movement becomes restricted and hence ΔS the entropy decreases
- We know that Gibbs free energy, $\Delta G = \Delta H - T\Delta S$.

For adsorption to be spontaneous, ΔG must be negative. This can happen if ΔH has a significantly high negative value as $-T\Delta S$ is positive.

- As the adsorption continues, ΔH becomes less and less negative till it becomes equal to $T\Delta S$ and ΔG becomes zero. At this point, equilibrium is attained.

1.6 Applications of Adsorption

1. For production of high vacuum.
2. Gas masks containing activated charcoal is used for breathing in coalmines. They adsorb poisonous gases.
3. Silica and aluminium gels are used as adsorbents for controlling humidity.
4. Removal of colouring matter from solutions.
5. It is used in heterogeneous catalysis.
6. In separation of inert gas.
7. As adsorption indicators.
8. In chromatographic analysis.
9. Qualitative analysis, e.g., lake test for Al^{3+} .

OBJECTIVE OF THE STUDY

- To study the dependence of variable parameters on the adsorption of **Murexide** on biochar.
- To confirm the activity of adsorbant.

CHAPTER - 2

LITERATURE REVIEW

Removal of basic dyes from solution using coconut shell charcoal, in this research aims to use agricultural waste as an adsorbent for dye removal from textile wastewater. Coconut shell charcoal (CSC), an agricultural waste, was used as dye adsorbent whilst basic yellow13 (BY13) and basic red14 (BR14) were used as representative dye used in textile manufactory. The removal of BY13 and BR14 from aqueous solution by CSC was investigated in batch adsorption at room temperature. Two parameters investigated in this research were pH of dyes solution; pH 2, 7, and 11, and particle size of adsorbents; 510- 700 μm and 1000-2000 μm . The adsorption model, Langmuir and Freundlich were also examined. It was found that by CSC, BY13 and BR14 had maximum removal percentage of 23.6 and 55.7 at pH 11. Size of adsorbent had also shown the effect on dye removal, i.e.; increasing size with decreasing removal capability. The dye removal experimental data were fitted to Langmuir adsorption model for both dyes with maximum adsorption capacity (q_m) for BY13 and BR14 of 19.76 and 22.93 mg/g, respectively. It can be implied that the adsorption is monolayer. It can conclude that CSC can be used as adsorbent for basic dye removal from aqueous solution. 1 Introduction Textile and handicraft dyeing use large volumes.

Decolorization of dyes from textile waste water using biochar, the textile industry is one of the largest in many low and middle-income countries, especially in Asia, second only to agriculture. Textile wastewater is discharged into the environment due to the lack of affordable and sustainable solutions to adsorb or remove the dye from the water. Biochar is generated by pyrolysis of organic material from plant waste in low-oxygen conditions, and is considered carbon-negative. Biochar for dye adsorption in textile wastewater effluent was proven to be highly effective. However, adsorption efficiency varies with experimental parameters, therefore there is a gap in application especially in small dye houses. Efforts should be made to find innovative and affordable solution to make the textile industry more sustainable, by developing methods for collection and reuse, recycle and upcycle of textile waste, by reducing the consumption of water, energy and chemicals and by developing methods for treatment of the textile wastewater.

Removal of Murexide (Dye) from aqueous media using rice husk as an adsorbent, the use of low-cost and ecofriendly adsorbent was investigated as an ideal alternative to the current expensive methods of removing dyes from wastewater. Rice husk was used as an adsorbent for the removal of Murexide from aqueous solutions. The rate of adsorption was investigated under various parameters such as size of adsorbent, contact time of solution with adsorbent, temperature, pH, adsorbent dose and stirring speed for the removal of this dye. Langmuir isotherm was also applied to evaluate maximum adsorption capacity of rice husk for Murexide. On the basis of results obtained, it is proposed that rice husk can be effectively used for the elimination of Murexide from waste water.

CHAPTER - 3

MATERIAL AND METHOD

INSTRUMENTS REQUIRED

Colorimeter, Weighing machine, Heavy rotary shaker, Hot air oven.

CHEMICALS REQUIRED:

Ammonium Purpurate, Biochar



Figure 2 - Ammonium purpurate

PREPARATION OF THE ADSORBENT

Dye adsorption behaviour of Ammonium Purpurate

The adsorption experiment was performed using a 0.02mM solution of the dye material. The effects of important parameters, such as amount of adsorbent, volume of dye solution, time were also studied on the adsorption using Ammonium Purpurate solution.

Effect of initial adsorbent amount

The experiments were carried out using 20ml of 0.02mM concentrated dye solutions and different amounts of adsorbent, and the amount adsorbed was calculated by analyzing the initial and final concentrations of dye solution using a colorimetric analysis. The dyes adsorptions were linearly increased in

progressing the amount of adsorbent. The increase in the number of active site causes the rise in adsorption amount.

Effect of contacttime

The experiments were carried out using 30mg of wood charcoal and 20 ml of Ammonium Purpuratesolution in different 5 conical flask. Then we shake these 5 mixtures (6,12,24hrs) and the amount adsorbed was calculated by analysing the initial and final concentrations of the dye solutions using a colorimetric analysis. The dyes adsorptions were linearly increased in progressing the amount of adsorbent. The increasing number of active sites causes the rise in adsorption amount.

Effect of concentration of dye

The experiments were carried out using 30g of wood charcoal and different concentrations of dye solutions (0.02,0.04,0.06,0.08mM) and the amount adsorbed was calculated by analysing the initial and final concentrations of dye solution using a colorimetric analysis. The dyes adsorptions were linearly increased in progressing the amount of adsorbent. The increase in the number of active site causes the rise in adsorption amount.

CHAPTER - 4

INSTRUMENTATION

The instruments used for making the biochar and batch adsorption include,

1) HOT AIR OVEN



Figure3 - Hot Air Oven

Hot air ovens are electrical devices which use dry heat to sterilize. They were originally developed by Pasteur.^[1] Generally, they use a thermostat to control the temperature. Their double walled insulation keeps the heat in and conserves energy, the inner layer being a poor conductor and outer layer being metallic. There is also an air-filled space in between to aid insulation. An air circulating fan helps in uniform distribution of the heat. These are fitted with the adjustable wire mesh plated trays or aluminium trays and may have an on/off rocker switch, as well as indicators and controls for temperature and holding time. The capacities of these ovens vary. Power supply needs vary from country to country, depending on the voltage and frequency (hertz) used. Temperature sensitive tapes or biological indicators using bacterial spores can be used as controls, to test for the efficacy of the device during use.

HEAVY ROTARY SHAKE



Figure 4 – Heavy Rotary Shake

These Shakers are ideal for mixing and development of cultures, chemicals, solvents, and assays etc. in Microbiological, Cell Culture & Life Science laboratories.

The Unit has base assembly fabricated from heavy mild steel sections. Brushless Induction drive motor with frequency drive makes the unit suitable for continuous non-stop operation. Step less electronic frequency control ensures gentle start and maintains preset speed. Compact counter balanced drive mechanism ensures high stability and reliability even in continuous operation & uneven load distribution.

WEIGHING MACHINE



Figure 5 – Weighing Machine

A scale or balance is a device to measure weight or mass. These are also known as mass scales, weight scales, mass balances, and weight balances. The traditional scale consists of two plates or bowls suspended at equal distances from a fulcrum. One plate holds an object of unknown mass (or weight), while known masses are added to the other plate until static equilibrium is achieved and the plates level off, which happens when the masses on the two plates are equal. The perfect scale rests at neutral. A spring scale will make use of a spring of known stiffness to determine mass (or weight). Suspending a certain mass will extend the spring by a certain amount depending on the spring's stiffness (or spring constant). The heavier the object, the more the spring stretches, as described in Hooke's law. Other types of scales making use of different physical principles also exist.

CHAPTER -5

RESULT AND DISCUSSION

About 0.014g **Murexide (ammonium purpurate)** of is weighed and transferred into 250 ML beaker, and it is diluted to 250 ML using distilled water. About 20 ML of made p solution is transferred into 3 different conical flask and then 0.30gm of charcoal is added to each of the conical flask. It is then placed on heavy rotary shaker for 10 minutes. It is then taken out and filtered using whatmann filter paper and then the absorbance is measured using calorimeter. This method is repeated for 20,30, 40 etc up to 80 minutes respectively.

Table 1. Colorimetric transmittance value of Murexide aqueous solution before and after adsorption by coconut shell biochar

TIME (Minutes)	TRANSMITTANCE (%)	
	Before adsorption	After adsorption
0	33	33
10	33	45
20	33	57
30	33	75
40	33	87
60	33	85
80	33	88

CHAPTER – 6

CONCLUSION

The goal of the investigation was to determine the adsorption of biochars on **Murexide**. Colorimetry was used to determine the absorbance. Initially, the concentration of dye solution was calculated, and the least amount was found to be 0.02 Mm for a reliable result. The multiple adsorption experiments with this concentration of and altering the amount of adsorbent **Murexide** resulted in 30mg being the minimal amount for obtaining a consistent outcome. The quick adsorption capacity **Murexide** of activated charcoal was curtailed by the kinetics of this adsorption. The effectiveness of activated charcoal has been validated by all of the adsorption metrics listed above.

CHAPTER - 7

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Temperatur zur Befreiung der porösen Körper von Gasen benutzte." ("Saussure already knew the two factors that determine the quantity of adsorption – [namely,] the pressure and temperature – since he used the lowering of the pressure or the raising of the temperature to free the porous substances of gases.")

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