ADSORPTION OF PHENOLPHTHALEIN USING BIOCHAR

The project report submitted to

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In partial fulfillment of the requirement for the award of

Master of Science in Chemistry

By

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CERTIFICATE

This is to certify that the project entitled "Adsorption of phenolphthalein using biochar" is a bonafide work carried out by Miss MIDHU VARGHESE (Reg. no: 190011010508) under guidance of Dr. ANEESH MATHEW, PG Department of Chemistry, Pavanatma College, Murickassery, for partial fulfillment of the 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, MIDHU VARGHESE, do hereby declare that this dissertation entitled "ADSORPTION OF PHENOLPHTHALIEN USING 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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INTRODUCTION

1.1 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 adsorption in which a fluid (the adsorbate) is dissolved by or permeates liquid or solid (the absorbent), respectively. Adsorption is a surface phenomenon, while absorption involves the whole volume of the material. The term sorption encompasses both processes.

Increase in the concentration of a substance at the interface of a condensed and a liquid or gaseous layer owing to the operation of surface forces.

Note: 1. Adsorption of proteins is of great importance when a material is in contact with blood or body fluids. In the case of blood, albumin, which is largely predominant, is generally adsorbed first, and then rearrangements occur in favour of other minor proteins according to surface affinity against mass law selection (Vroman effect).

Note: 2: Adsorbed molecules are those that are resistant to washing with the same solvent medium in the case of adsorption from solutions. The washing conditions can thus modify the measurement results, particularly when the interaction energy is low.

Similar to 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 materials are filled 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 or carbidederived carbons and water purification. Adsorption, ion exchange and chromatography are sorption processes ion 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.

1.1.1 PHYSISORPTION

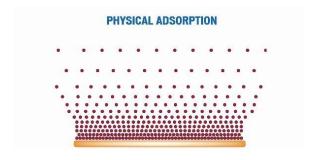


Figure 1 Physical adsorption

Physisorption also called physical adsorption is a process in which the electronic structure of the atom or molecule is barely perturbed upon adsorption.

The fundamental interacting force of physisorption is caused by van de Waals force. Even though the interaction energy is very weak. (10-100 meV), physisorption plays an important role in nature. For instance the van der Waals attraction between surfaces and foothairs of geckos provides the remarkable ability to climb up vertical walls. Van der Waals forces originate from the interactions between induced permanent or transient electric dipoles.

In comparison with chemisorptions, in which the electronic structure of bonding atoms or molecules is changed and covalent or ionic bonds form, physisorption, generally speaking, can only be observed in the environment of low temperature (thermal energy at room temperature 26meV) and the absence of the relatively strong chemisorption. In practice, the categorization of a particular adsorption as physisorption or chemisorptions depends principally on the binding energy of the adsorbate to the substrate.

1.1.2 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 adsorbent surface. Examples include macroscopic phenomena that can be very obvious, like corrosion, and subtler effects associated with heterogeneous catalysis. The strong interaction between the adorable and the substrate surface creates new types of electronic bonds.

In contrast with chemisorptions is physisorption, which leaves the chemical species of the adorable and surface intact. It is conventionally accepted that the energetic threshold separating the binding energy of physisorption from that of chemisorptions is about 0.5eV per adsorbed species. Due to specialty, the nature of chemisorptions can greatly differ, depending on the chemical identity and the surface structure. Activated carbon is used as an adsorbent.

Adsorbents are used usually in the form of spherical pellets, rods, moldings, or monoliths with a hydrodynamic radius between 0.25 and 5 mm. They must have high abrasion resistance, high thermal stability and small pore diameters, which results in higher exposed surface area and hence high capacity for adsorption. The adsorbents must also have a distinct pore structure that enables fast transport of the gaseous vapours.

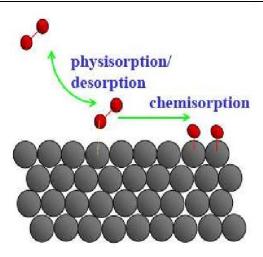


Figure 2 Schematic representation of different adsorption process

Industrial adsorbents fall into one of three classes:

- Oxygen- containing compounds are typically hydrophilic and polar, including materials such as silica gel and zeolites.
- Carbon- based compounds- are most typically hydrophobic and non-polar, including materials such as activated carbon and graphite.
- Polymer- based compounds- are polar or non-polar functional groups in a porous polymer matrix.

Table 1 Comparison between physisorption and chemisorptions

Physisorption	Chemisorptions		
Low heat of adsorption usually in the range	High heat of adsorption in the range		
of 20-40 KJ mol-1	of 40-400kJ mol-1		
Force of attraction are van der wall's forces	Forces of attraction are chemical		
	bond forces		
It usually takes place at low temperature	It takes place at high temperature		
and decreases with increasing temperature			
It is reversible	It irreversible		

It is related to the ease of liquefaction of the	The extent of adsorption of				
gas	adsorption is generally not related to				
	liquefaction of the gas				
It is not very specific	It is highly specific				
It forms multi –molecular layers	It forms monomolecular layers				
It does not require any activation energy	It requires activation energy				

1.2 FACTORS AFFECTING ADSORPTION

The extent of adsorption depends upon the following factors:

- 1. Nature of adsorbate and adsorbent.
- 2. The surface area of adsorbent.
- 3. Activation of adsorbent.
- 4. Experimental conditions. E.g. Temperature, pressure etc.

Adsorption Process



Figure 3 Schematic representation of Adsorption process

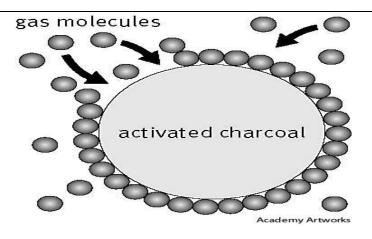


Figure 4 Adsorption process on charcoal

The following are the factors which affect the adsorption,

Nature of the adsorbate; (Adsorption process is usually studied through graphs known as adsorption isotherm. That is the amount of adsorbate on the adsorbent as a function if its pressure or concentration at constant temperature. The quantity adsorbed is nearly always normalized by the mass of the adsorbent to allow comparison of different materials.

- 1) Adsorbate (gas) and adsorbent (solid)
 - In general, easily liquefiable gases e.g., Co₂, NH₃, Cl₂ and SO₂ etc. are adsorbed to a greater extent than the elemental gases eg.H₂, O₂, N₂, He etc. (While chemisorptions is specific in nature) Porous and finely powdered solid e.g. Charcoal, fuller's earth, adsorbs more as compared to the hard-porous materials. Due to this property powdered charcoal is used in gasmasks.
- 2) Surface area of the solid adsorbent
 - The extent of adsorption depends directly upon the surface area of the adsorbent, i.e., larger the surface area of the adsorbent, greater is the extent of adsorption.
 - Surface area of powdered solid adsorbent depends upon its particle size.

 Smaller the particle size, greater is its surface area.
- 3) Effect of pressure on the adsorbate gas

- An increase in the pressure of the adsorbate gas increases the extent of adsorption
- At low temperature, the extent of adsorption increases rapidly with pressure.
- Small range of pressure, the extent of adsorption is found to be directly proportional to the pressure.
- At high pressure (closer to the saturation vapour pressure of the gas), the adsorption tends to achieve a limiting value.

4) Effect of Temperature

As adsorption is accompanied by evolution of heat, so according to the LeChatelier's Principle, the magnitude of adsorption should Porous and finely
powdered solid e.g. Charcoal, fuller's earth, adsorb more as compared to
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6) Effect of pressure on the adsorbate gas

- An increase in the pressure of the adsorbate gas increases the extent of adsorption
- At low temperature, the extent of adsorption increases rapidly with pressure.

- Small range of pressure, the extent of adsorption is found to be directly proportional to the pressure.
- At high pressure (closer to the saturation vapour pressure of the gas), the adsorption tends to achieve a limiting value.

7) Effect of Temperature

 As adsorption is accompanied by evolution of heat, so according to the Le-Chatelier's Principle, the magnitude of adsorption should decrease with rise in temperature.

1.3 MECHANISM OF ADSORPTION

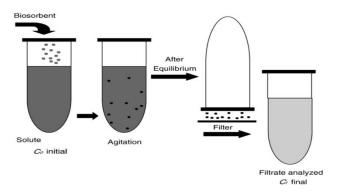


Figure 5: Schematic representation of adsorption process

Adsorption, Adsorbent and Adsorbate

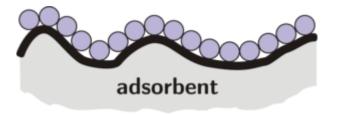


Figure 6: Unimolecular adsorption

It is an exothermic process which means that energy is liberated during this process. The amount of heat that gets evolved when one mole of the adsorbate is adsorbed on adsorbent is known as enthalpy. The change in enthalpy is devoted to be negative. The reason behind this is that when adsorbate molecules are adsorbed on the surface, freedom of

movement of molecules become restricted and this result in a decrease in entropy. At constant temperature and pressure, adsorption occurs spontaneously.

Adsorption is the deposition of molecular species on to the surface. The molecular species that gets adsorbed on the surface is known as the adsorbate. Common examples of adsorbents are clay, silica gel, colloids, metals etc. Thus, adsorption is a surface phenomenon. The process of removal of adsorbent from the surface of adsorbate is known as desorption.

1.4 APPLICATIONS

The phenomenon of adsorption finds a number of applications. Important applications are given as follows:

1) Production of high vacuum

In gas masks: This apparatus is used to adsorb poisonous gases e.g. O₂, CO₂, oxide of sulphur etc.) And thus purify the air for breathing.

For desiccation or dehumidification: These substances can be used to reduce/remove water vapours or moisture present in the air. Silica gel and alumina are used for dehumidification in electronic equipment.

Removal of colouring matter from solution: (i) Animal charcoal remove colors of solutions by adsorbing coloured impurities. (ii) Animal charcoal is used as decolorize in the manufacture of cane sugar.

Heterogeneous catalysis: Mostly heterogeneous catalytic reactions proceed through the adsorption of gaseous reactants on solid catalyst. For example, finely powdered nickel is used for the hydrogenation of oils. Finely divided vanadium pentaoxide is used in the contact process for the manufacture of sulphuric acid. Separation of inert gases: Due to the difference in degree of adsorption of gases by charcoal, a mixture of inert gases can be separated by adsorption on coconut charcoal at different low temperatures.

1.5 DYES

Dyes are a coloured substance that has an affinity to the substrate to which it is being applied. 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 in soluble with the addition of salt to produce a lake pigment.

Dyes are classified according to their solubility and chemical properties

- 1) Acid dye
- 2) Basic Dyes.

Acid dyes are water-soluble anionic dyes that are applied to fibers such as silk, wool, nylon and modified acrylic fibers using neutral to acid dye baths. Attachment to the fiber is attributed, at least partly, to salt formation between anionic groups in the dyes and cationic groups in the fiber. Acid dyes are not substantive to cellulosic fibers. Most synthetic food colors fall in this category. Examples of acid dye are Alizarin Pure Blue B, Acid red 88 etc.Basic dyes are water-soluble cationic dyes that are mainly applied to acrylic fibers, but find some use for wool and silk. Usually acetic acid is added to the dye bath to help the uptake of the dye onto the fiber. Basic dyes are also used in the coloration of paper.

PHENOLPHTHALEIN

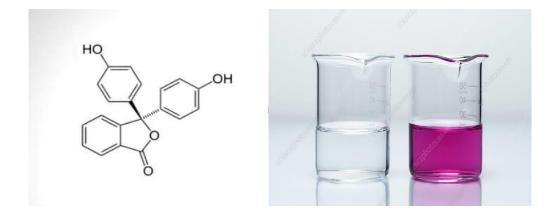


Figure 7 Structure of phenolphthalein

Figure 8 Phenolphthalein colorless and coloured

Phenolphthalein is a chemical compound with formula $C_{20}H_{14}O_4$ and is often written as 'HIn' or 'phph' in shorthand notation. Phenolphthalein is often used as an indicator in acid- base titrations. For this application, it turns colorless in acidic solutions and observed as pink in basic solutions. It belongs to the class of dyes known as phenolphthlein dyes.

Phenolphthalein is slightly soluble in water and usually is dissolved in alcohols for use in experiments. It is a weak acid, which can lose H+ ions in solution. The phenolphthalein molecule is colorless, and the phenolphthalein is pink. When a base is added to the phenolphthalein, the equilibrium shifts, leading to more ionization as H+ ions are removed. This is predicted by Le Hotelier's principle

PH INDICATOR

Phenolphthalein's common use is as an indicator in acid-base titrations. Indicators are the substance that changes colour of the solution in response to a chemical change. A drop of indicator is added in the start of the titration, the end point has been appeared when colour of the solution is changes. It also serves as a component of universal indicator, together with methyl red, bromothymol blue, and thymol blue.

Phenolphthalein adopts at least four different states in aqueous solution as a result of				
PH changes, under strongly acidic conditions, it exists in protonated form (HIn+),providing				
an orange coloration. Between strongly acidic and slightly basic conditions, the lactone form				
(HIn) is colorless. The doubly deprotonated (In ²⁻) phenolate form (the anion form of phenol)				
gives the familiar pink colour. In strongly basic solutions, phenolphthalein is converted to its				
In (OH) 3- form, and its pink colour undergoes a rather slow fading reaction and becomes				
completely colorless above 13.0 pH				

LITERATURE SURVEY

There are many processes for the treatment of metal- contaminated water including chemical precipitation, membrane filtration, reverse osmosis, ion exchange, and adsorption. However, their use is limited due to various disadvantages such as incomplete metal removal, high capital operational cost and the disposal of residual metal sludge which are not suitable for small scale industries. Adsorption has been proved as one of the most efficient methods for the removal of heavy metals from aqueous media. Adsorption has advantages over other methods for the removal of heavy metals from water because (a) its design is simple (b) it is sludge- free and (c) adsorbent used is low cost. There are many reports showing the development of low cost activated carbon prepared from cheaper and readily available materials such as apricot seed, olive seed, date seed, peanut shell, coconut shell, palm shell, rice husk etc. for the production of activated carbon. In present study adsorption isotherms of (Cd) II adsorption onto PAALSSC (Phosphoric acid activated Lapsi Seed Stone carbon) studied through Langmuir isotherm and Freundlich isotherm. Adsorption isotherm usually describes the equilibrium state between the amount of adsorbed metal ion onto the adsorbent surface and the concentration of metal ions in solution. The kinetics of (Cd)) II adsorption onto activated carbon has been studied through a pseudo-first-order and a pseudo- pseudosecond-order.

Biochar is organic matter that has undergone combustion under low to no oxygen conditions (i.e., pyrolysis) resulting in a recalcitrant, high carbon material specifically for use as a soil amendment. Recently, fervent interest in the production of biochar to address issues of fertility, water holding capacity, remediation, climate change mitigation, etc., led to much greater understanding of the complexities of this potential amendment in altering soil biological, chemical, and physical properties. Rather than assume benefit of any biochar

created from any feedstock added to any soil ecosystem, concepts of matching appropriate feedstock and pyrolysis condition to soil type to achieve specific goals associated with remediation, increasing yields, decreasing greenhouse gas emission, and/or climate change mitigation emerged. The potential for biochar to improve both environmental and economic viability remains but should be explored fully in a integrative analysis of waste-stream management, local biochar production capacity, realized improvement in soil function, etc., with an economic benefit-cost and net greenhouse warming potential analysis of the system.

The control of water pollution has become of increasing importance in recent years. The release of dyes into the environment constitutes only a small proportion of water pollution, but dyes are visible in small quantities due to their brilliance. Tightening government legislation is forcing textile industries to treat their waste effluent to an increasingly high standard. Currently, removal of dyes from effluents is by physio-chemical means. Such methods are often very costly and although the dyes are removed, accumulation of concentrated sludge creates a disposal problem. There is a need to find alternative treatments that are effective in removing dyes from large volumes of effluents and are low in cost, such as biological or combination systems. This article reviews the current available technologies and suggests an effective, cheaper alternative for dye removal and decolourisation applicable on large scale.

${\bf OBJECTIVE_OF\ STUDY}$

In this project the main objective of study is to remove the phenolphthalein dye from the aqueous solution by adsorption technique using eco-friendly coconut shell bio-char and also discuss about,

- i) Effect of amount on adsorption.
- ii) Effect of concentration on adsorption

MATERIALS

4.1 COCONUT SHELL BIOCHAR

The coconut shells are good raw materials to produce coconut shell charcoal. The process of converting shells to charcoal includes removing all the moisture from the raw material in a limited supply of air. It requires a special charcoal drum to carbonize the coconut shells and produce high quality charcoal.



Figure 9 Carbonized coconut shell

4.2 CHEMICALS

- ❖ Phenolphthalein solution- NICE CHEMICALS (P) LTD.
- ❖ Potassium hydroxide pellets- NICE CHEMICALS (P) LTD.

Here potassium hydroxide is used to make the solution alkaline which makes the solution to appear pink in color.

A 2 ADDADATUS				
4.3 APPARATUS				
*	250 ml beaker			
*	Conical flask			
*	Glass rod			
*	20ml pipette			
*	Funnel			
*	Whatmann filter paper			
*	Spatula			
*	Test tube			

INSTRUMENTATION

5.1 HOT OVEN

Laboratory ovens are ovens for high-forced volume thermal convection applications. Those ovens generally provide uniform temperatures throughout. Process applications for laboratory ovens can be for annealing, die-bond curing, drying, polyimide baking, sterilizing, and other industrial laboratory functions. Typical sizes are form one cubic foot to 0.9 cubic meters (32 cu ft) with temperatures that can be over 340 degrees Celsius. Laboratory ovens can be used in numerous different applications and configurations, including clean rooms, forced convection, horizontal airflow, inert atmosphere, neutral convection, and pass through.



Figure 10 Hot air oven

5.2 MUFFLE FURNACE

A muffle furnace (sometimes report furnace in historical usage) is a furnace in which the subject material is isolated from the fuel and all of the products of combustion, including gases and flying ash. After the development of high-temperature electric heating elements and widespread electrification in developed countries, new muffle furnaces quickly moved to electric design. Today, a muffle furnace is (usually) a front- loading box-type oven

or kiln for high- temperature applications such as fusing glass, creating enamel coatings, ceramics, and soldering and brazing articles. They are also used in many research facilities, for example by chemists in order to determine what proportion of a sample is non-combustible and non-volatile (i.e., ash). Also advances in materials for heating elements, such as molybdenum disilicide, can now produce working temperatures up to 1,800 degrees Celsius, which facilitate more sophisticated metallurgical applications.



Figure 11 Muffle Furnace

The term muffle furnace may also be used to describe another oven constructed on many of the same principles as the box-type kiln mentioned above, but takes the form of a long, wide, and thin hollow tube used in roll- to-roll manufacturing processes. Both of the above mentioned furnaces are usually heated to desired temperatures by conduction, convection, or blackbody radiation from electrical resistance heating elements. Therefore, there is (usually) no combustion involved in the temperature control of the system, which allows for much greater control of temperature uniformity and assures isolation of the material being heated from the byproducts of fuel combustion.

5.3 COLORIMETER

A colorimeter is a device used in colorimetric process. In scientific fields the word generally refers to the device that measures the absorbance of particular wavelengths of light by a specific solution. This device is commonly used to determine the concentration of a known solute in a given solution by the application of the Beer-Lambert law, which states that the concentration of a solute is proportional to the absorbance.



Figure 12 Digital Colorimeter

Cuvettes: In a manual colorimeter the cuvettes are inserted and removed by hand and automated colorimeter (as used in an AutoAnalyzer) is fitted with a flow cell through which solution flows continuously.

Output: The output from a colorimeter may be displayed by an analogue or digital meter and may be shown as transmittance (a linear scale from 0-100%) or as absorbance (a logarithmic scale from zero to infinity). The useful range of the absorbance scale is from 0-2 but it is desirable to keep within the range 0-1, because above 1 the results become unreliable due to scattering of light. In addition, the output may be sent to a chart recorder, data logger, or computer.

5.4 LABORATORY SHAKER

A Shaker is a piece of laboratory equipment used to mix, blend, or agitate substances in a tube or flask by shaking them. It is mainly used in the fields of Chemistry and Biology. A shaker contains an oscillating board that is used to place the flasks, beakers, or test tubes. Although the magnetic stirrer has lately come to replace the shaker, it is still the preferred choice of equipment when dealing with large volume substances or when simultaneous agitation is required.



Figure 13 Laboratory shaker

A platform shaker has a table board that oscillates horizontally. The liquids to be stirred are held in beaker, jars, or Erlenmeyer flasks that are placed over the table, or sometimes, in test tubes or vials that are nested into holes in the plate. Platform shakers can also be combined with other systems like rotating mixers for small systems and have been designed to be manufactured in laboratories themselves with open source scientific equipment.

EXPERIMENTAL ANALYSIS

6.1 Adsorption Experiment

Stock solution of potassium-phenolphthalein solution was prepared by dissolving two pellets of KOH in 250 ml of distilled water and two drops of phenolphthalein were added to KOH solution and pink color is obtained.



Figure 14 Stock solution of phenolphthalein-water solution

PROCEDURE-1

 $20 \; ml \; of \; phenolph thale in-potassium \; complex \; solution \; pipetted \; out \; into \; three \; different \; conical \; and \; control \; properties \; conical \; control \; properties \; proper$

flasks.



Figure 15 Phenolphthalein –potassium solution in three conical flasks

Added 100 mg, 200 mg, 300 mg of coconut shell biochar to each of the conical flask. Kept three conical flasks on mechanical shaker for 40 minutes to mix well.



Figure 16 Shaking for 40 minutes on mechanical shaker

After 40 minutes the solution is filtered with whatmann filter paper and transferred to test tubes.



Figure 17 Filtration process

To record transmittance, the solution is taken in cuvette and placed in calorimeter and noted the percentage of transmittance for different amount of biochar before and after adsorption.

PROCEDURE-2



Figure 18 Different concentration of phenolphthalein-potassium water solution.

Take three conical flasks. To the first conical flask add 20ml of stock solution of potassium-phenolphthalein solution alone. To the second conical flask add 15ml of potassium-phenolphthalein solution and 5 ml distilled water. To the third conical flask add 10 ml potassium-phenolphthalein solution and 10 ml distilled water. Add constant amount of coconut shell biochar (100mg) to each conical flask. Record transmittance of each mixture using colorimeter.

RESULTS AND DISCUSSIONS

7.1 EFFECT OF AMOUNT OF ADSORPTION

Table 2.Effect of amount of coconut shell biochar on phenolphthalein adsorption

SL.NO. Conce	КОН	Coconut Shell	Time	Phenolphthalein (drop)	Transmittance	
	Concentration (M)	Biochar (mg)			Before Adsorption (%)	After Adsorption (%)
1	1	100	40	2	50	55.8
2	1	200	40	2	50	57
3	1	300	40	2	50	59

Taking constant volume of potassium-phenolphthalein solution, varied amount of biochar from 100 to 300mg. As the amount increase the transmittance value increases. Transmittance measure the amount of light that passes through a material. So here adsorption takes place.

7.2 EFFECT OF CONCENTRATION

Table 3. Effect of concentration on adsorption.

SL.NO	Amount of coconut shell biochar(mg)	Volume of potassium-phenolphthalein solution(ml)	Volume of distilled water(ml)	Transmittance (%)
1	100	20	-	78
2	100	15	5	82
3	100	10	10	88

As the concentration of potassium-phenolphthalein solution decreases, transmittance increases.

CONCLUSION In this study we have analysed the adsorption of phenolphthalein from aqueous solution by exploring the colour formation chemistry of Phenolphthalein-Potassium complex in different amount of coconut shell biochar and difference in concentration of potassiumphenolphthalein solution. In both cases transmittance increased due to the adsorption phenomena. All the adsorption parameters discussed above has been supported the efficiency of activated charcoal towards Phenolphthalein adsorption from aqueous solution.

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