ADSORPTION STUDIES OF METHYLENE BLUE WITH VARIATION OF PH USING CARBONIZED AND ACTIVATED PALM KERNEL AND COCONUT SHELLS

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CERTIFICATION

This is to certify that this research work was carried out by EBHOJIE PHILOMENA OBEHI with Matriculation Number AST/2382051229 in the Department of Physical Science Laboratory Technology, School of Applied Science.

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DEDICATION

This project work is dedicated to Almighty God in whom we live and have our being for His infinite kindness and love which was manifested all through the duration of this research work, to him be honour and glory now and forever. Amen.

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ABSTRACT

The ability of Palm kernel and coconut shells from both carbonized and activated to remove methylene blue (MB) from aqueous solution was investigated. The carbonized and activated palm kernel and coconut shells were done by physical process. Variation of PH was carried *out using 10ppm concentration. The absorption capacity was determined as a function of variation of PH of PH3,PH6 and PH8. The variation in pH shows that, the methylene blue was removed at alkaline medium than the acidic medium and near to neutral, with the absorbance value of 0.216,0.033,0.35 for carbonized palm kernel shell and -0.833,-0.139 and -0.677 for activated palm kernel shell,-0.012,-0.815,-0.537 for carbonized coconut shell and -0.806,0.249 and -0.477 for activated coconut shell, all at the PH of 3,6 and 8 respectively. The study also shows that the absorption of MB by palm kernel and coconut shells for both activated and carbonized depend on the initial concentration of MB. The use of palm kernel and coconut shells as activated carbon is not only effective for MB removal from wastewater but also in solving the problem of over abundance of palm kernel and coconut shell as agricultural waste products.*

KEY WORDS: Carbonization, adsorption,activation, adsorbent,adsorbate.

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CHAPTER ONE

INTRODUCTION

1.1 BACKGROUND OF THE STUDY

The problems of ecosystem are increasing with development technology. Heavy metal pollution is one of the main problems due to their toxicity to human life, Organic pollutants and its derivatives found in industrial waste waters and as a main components in plastics are considered to have toxic effects on human health even when present in small concentrations (Badmus et al, 2007). Through the advancement of technology has given us, comforts but at the same industrialization have contributed greatly to the environmental pollution e.g. Water pollution, air pollution and soil pollution. The water and air pollution directly affect the human and soil pollution can contribute to the agricultural and food poisoning, which ultimately contribute to adverse effects to human and other living creatures (Baby Shaikh et al, 2018),

Water contamination is a major hazard for living things and human beings and there may be different kinds of contaminants such as bacteria, viruses, organic molecules, dyes and heavy metal ions e.g. Cr^{6+}, Pb^{2+}, Cd^{2+}, Zn^{2+}, Ni^{2+}, As^{3+} and Hg^{2+} etc. Among all of these water contaminants, heavy metal ions are non-biodegradable in nature and can accumulate in the human body continuously and may results severe adverse effects such as brain damage, skin diseases, liver damage, kidney failure, anaemia, hepatitis, ulcers and are also carcinogenic (Yang et al, 2018). Heavy metal ions enter the water and environment from different sources, namely industrial wastes, batteries, fertilizers, pesticides, petrochemicals, pharmaceutical, paper and pulp industries etc. The water pollution is making the lives of millions of people at great risks of diseases, illness and even deaths. In addition, water pollution is continuously shortening the availability of drinking water (Wu, Y. et al., 2019).

1.2. SIGNIFICANCE OF THE STUDY

With a positive result of this study, this result can be applied in:

1. Textile industries
2. Tannery industries for the decontamination of their effluents.

1.3 SCOPE OF THE STUDY

This study covers Adsorption studies of methylene blue with variation of PH using palm kernel and coconut shell.

1.4 LIMITATIONS OF THE STUDY

Limitations faced in this study include

1. Difficulty in carbonization process of the palm kernel shell
2. Financial constraints
3. Irregular power supply

1.5 AIMS AND OBJECTIVES

The aim of this study is to determine the Adsorption of methylene blue with variation of PH using activated and carbonized palm kernel and coconut shell.

CHAPTER TWO

LITERATURE REVIEW

2.1. DESCRIPTION OF COCONUT SHELL

Coconuts are the seeds of a species of palm called *Cocos nucifera* which belongs to the arecaceae family and found throughout the tropic and sub-tropic area where it is known for its versatility. The palms flower on a monthly basis and the fruit take one year to ripen. A mature tree may have fruit in every stage of maturity and can produce 50 to 100 coconuts per year. The fruit contains the fibrous layer, the hard shell and the inner edible whitish layer (endosperm). The fibrous layer can be separated from the hard shell manually. The hard shell is broken open to separate it from the nutritious edible endosperm (Predeepkumor et al, 2008). Large quantities of coconut shell are generated annually and only some fractions are used for fuel and some other applications, but greater percentage are dumped around the processing mill constituting environmental and economic liability for the people (Woolley et al, 1997).. Coconut shell is composed of 53.06 % cellulose, 36.51 % lignin and 29.27 % pentosans. Coconut shell powder is used to produce shell charcoal and applied widely as domestic and industrial fuel. Shell charcoal is used to produce activated carbon. Activated carbon produced from coconut shell has certain specific advantages as the raw material can adsorb certain molecular species. Coconut shell powder is also used as fillers in the manufacture of thermosets.

2.2. DESCRIPTION OF PALM KERNEL

Elaeis guineensis (oil palm) originated in Guinea and was later introduced to Java (Indonesia), Nigeria and Malaysia. Though the oil palm originated in West Africa, it has since been planted successfully in tropical regions within 20 degrees of the equator (Obahiagbon, 2012). Oil extracted from both the pulp of the fruit (palm oil) and the kernel (palm kernel oil) is used for foods and soap manufacture (Paterson, 2007). The increasing use of oil in the commercial food industry all over the world is because of its cheap pricing, high oxidative stability and high levels of natural antioxidants.

Palm kernel is a virgin biomass with high calorific value, low ash, moisture and sulphur contents. It is a lignocellulose biomass. Carbonized palm kernel shells are used as charcoal which can be pressed into bio-fuel briquette. It can also be processed into activated carbon which is used in liquid and gas phase filtration or adsorption (Mathaus, 2007). In 2010/2011, the total world production of palm kernels was 12.6 million tons.

2.3. USES OF PALM KERNEL SHELL (PKS) AND COCONUT SHELL (CS)

Palm kernel shell (PKS) and coconut shell (CS) are not commonly used in the construction industry but are often dumped as agricultural wastes. However, with the quest for affordable housing system for both the rural and urban population of Nigeria and other developing countries, various proposals focussing on cutting down conventional building material costs have been put forward. One of the suggestions in the forefront has been the sourcing, development and use of alternative, non-conventional local construction materials including the possibility of using some agricultural wastes and residues as construction materials (Olanipekun et al, 2005).

PKS and CS, both of which belong to the family of palm shells, are agricultural waste products obtained in the processing of palm oil and coconut oil, respectively, and are available in large quantities in the tropical regions of the world, most especially in Africa, Asia and America. In Nigeria, both are available in large quantities in the southern part of the country. Previous studies have shown that PKS is suitable as granular filter for water treatment, as a suitable aggregate in plain, light and dense concretes and as a road building material. Apart from its use in production of fibre-roofing material, the other possibility of using CS as an aggregate in concrete production has not been given any serious attention. However, Adeyemi investigated, for one mix ratio (1:2:4) the suitability of CSs as substitute for either fine or coarse aggregate in concrete production. It was concluded that the CSs were more suitable as low strength-giving lightweight aggregate when used to replace common coarse aggregate in concrete production (Olanipekun et al, 2005).

2.4. Adsorption studies

Adsorption is a surface process that leads to transfer of a molecule from a fluid bulk to solid surface. This can occur because of physical forces or by chemical bonds. Usually it is reversible (the reverse process is called desorption); then it is responsible not only for a subtraction of substances but also for release. In most of the cases, this process is described at the equilibrium by means of some equations that quantify the amount of substance attached on the surface given the concentration in the fluid. These equations are called isotherms (the most famous are the Langmuir and the Freundlich equations) because of the dependence of their parameters on the temperature, which is one of the most important environmental factors affecting adsorption. Adsorption has a fundamental role in ecology: it regulates the exchanges between geosphere and hydrosphere and atmosphere, accounts for the transport of substances in the ecosystems, and triggers other important processes like ionic exchange and enzymatic processes.

Adsorption process is highly efficient, useful, simple and insensitive to toxic substances, effective, less environmentally degrading and economical tool (Loubna et al., 2007). The major advantages of adsorption technologies are its effectiveness in reducing the concentration of heavy metal ions to very low levels and the use of inexpensive adsorbent materials. However, most conventional waste water treatment techniques for removal of metals such as filtration, activated charcoal, reverse osmosis, chemical precipitation, solvent extraction, ion exchange and membrane separation. These are not economically viable for small scale industries due to huge capital investment. It is therefore necessary to search for low-cost techniques that may be effective, less environmentally degrading and economical for industrial usage. Adsorption has been shown to be an economically feasible alternative method for removing heavy metals from waste water and water supplies (Horsfall and Spiff,. 2004)). In the purification process, low cost adsorbent are preferred

The use of agricultural by-products for the sorption of valuable metals from waste water has continued to attract considerable attention because they are cheap source of biosorbents, sludge free and involve small initial cost and land investment (Horsfall et al,. 2004).These includes the use of numbers of agricultural materials such as sugar cane cabon (Krishnan,et al,. 2003), rice husk (Naiya, et al., 2009), Cassava waste (Horsfall, et al 2004.), wild cocoyam biomass (Horsfall and Spiff,. 2004), wheat bran (Loubna et al,.2007).

Palm kernel shell (PKS) of oil palm is useful material to be applied as an adsorbent for the removal of heavy metal ions, as the good quality of organic compounds in it capable of adsorption of metal ions through biosorption mechanisms. PKS is sustainable agricultural waste produced in millions of tons every year. The disposal of large quantity PKS causes adverse effects to the environment as it disposed-off by burning causing a lot smoke (. In this study, we utilized the PKS and coconut shell as an adsorbent for the treatment of heavy metal contaminated water and the effect of the parameter pH of the solution

Types of Adsorption

On the basis of interaction forces between adsorbate and adsorbent, adsorption is of two types.

1. **Physical adsorption:**

This type of adsorption is also known as physisorption. It is due to weak Van der Waals forces between adsorbate and adsorbent.

For example, H_2 and N_2 gases adsorb on coconut charcoal.

2. **Chemical adsorption:**

This type of adsorption is also known as chemisorption. It is due to strong chemical forces of bonding type between adsorbate and adsorbent. We can take the example involving the formation of iron nitride on the surface when the iron is heated in N2 gas at 623 K.

Adsorption of gas on a solid is a spontaneous exothermic reaction. The amount of heat liberated when a unit mass of a gas is adsorbed on the surface is called heat of adsorption.

Physisorption and Chemisorption Adsorption Characteristics

Characteristics of physical adsorption:

1. This type of adsorption is caused by physical forces.
2. Physisorption is a weak phenomenon.
3. This adsorption is a multi-layered process.
4. Physical adsorption is not specific and takes place all over the adsorbant.
5. Surface area, temperature, pressure, nature of adsorbate effects physisorption.
6. Energy for activation is low (20 – 40 kg/mol).

Characteristics of chemical adsorption:

1. This type of adsorption is caused by chemical forces.

2. It is a very strong process.
3. This type of adsorption is almost a single-layered phenomenon.
4. Chemisorption is highly specific and takes place at reaction centres on the adsorbant.
5. Surface area, temperature, nature of adsorbate effects chemisorption.
6. Energy of activation is very high 40 – 400 kJ/mol.

Adsorption Isotherm

Adsorption is usually described by isotherms. It is due to the fact that temperature plays an important role or that it has a great effect on the whole process. Moreover, there are several isotherm models that are used to describe the adsorption technique.

Applications of Adsorption

1) Air pollution masks:

These consist of silica gel or activated charcoal powder, when dust or smoke are paused through them, those particles get adsorbed on the surface of these materials.

2) Separation of noble gases by Dewar's flask process:

A mixture of noble gases of Ne, Ar, Kr is passed through Dewar's flask in presence of heated coconut charcoal. Argon and Krypton gels adsorbed leaving Neon.

3) Purification of water:

By the addition of alum stone to the water, impurities get adsorbed on the alum and water gets purified.

4) Removal of moisture and humidity:

Moisture in the air is removed by placing silica gel on which water molecular gets adsorbed.

5) Adsorption chromatography:

It is used to separate pigments and hormones.

6) Ion exchange method:

In this method of removing the hardness of water, calcium and magnesium ions get adsorbed on the surface of ion exchange resin

7) In metallurgy:

In the froth floatation process of concentration of ore, the particle gets adsorbed on the froth.

2.5. Methylene Blue

Methylene Blue is a cationic stain. In simple terms, it is a blue dye that is charged positively and binds the negatively charged components of the cells such as the nucleus, DNA or RNA present in the cytoplasm. These cells usually have a low affinity. It is a popular stain used for a variety of activities, including bacterial identification and cellular structure in both plant cells and animal cells. Methylene Blue solution is used to stain the cells. The purpose of staining with methylene blue is to determine cell mortality. When methylene blue stain is given to a sample, a healthy cell causes the stain to become colourless.

Methylene blue is used for several purposes, including as an antidepressant, a treatment for cyanide poisoning, and a titration indicator in a laboratory. It turns dark blue when it is dissolved in water. It is utilized in a number of scientific investigations for a variety of procedures. It is an efficient redox indicator in chemistry because it turns clear when it comes into touch with reducing substances. It is used as a titration aid in solutions. It’s also employed as a pigment in microbiology to analyze nucleic acid chains. It can be added to a solution to colour RNA and DNA for visual analysis.

Methylene Blue can be used as a counterstain. For using counterstain you have to decolorize the primary stain using a decolorizing agent (an acid-alcohol solution)

What does methylene blue stain

One of the most popular uses of methylene blue today is in chemistry, biology, medicine and microbiology for staining or highlighting the sections specimens for the animal, bacterial, and blood tissues. It can be used to examine the forms, structures, and locations of dead cells, cell structures and other tissues in plants as well as animals in great detail.

Methylene blue solution contains a blue dye that stains acidic animal cell components (such as the nucleus) blue, allowing to readily examine and analyze them during microscopy, histology, hematology, morphology, and other biological research. Methylene blue works well as a counter-stain for Eosin Y, a red stain used to stain connective tissue and cytoplasm. It is also used in Gram staining to identify gram-positive and gram-negative bacteria.

Uses of Methylene Blue as a stain

- For determining cell damage: When methylene blue stain is introduced to a sample of cells or tissue, a healthy cell causes the stain to become colourless. This is because the enzymes in the cell degrade the methylene blue, causing it to lose its colouration. Because the enzymes in the cell have been inactivated by the dye, there will be no response if the cell is dead. Refer here for more details about this use.
- Identification of Microorganisms: Methylene blue is a dye frequently employed by biologists to aid in the identification of microorganisms. As the bacteria are nearly colourless, a biologist can detect bacterial forms and structures by adding a drop or two of methylene blue to a microscope slide. In biology, a stain is a dye such as

methylene blue. It acts by attracting chemicals to biological tissues and binding to them.

- Identifying Nucleic Acids: When methylene blue comes into contact with acids, it turns a vivid shade of blue. This characteristic makes it extremely valuable for identifying nucleic acids such as DNA and RNA. It can also be used in cellular structure, gram's staining techniques as an alternative to chemical crystal violet.
- Identifying RNA Sequences: Because of its affinity for nucleic acids, methylene blue has been employed in specialized procedures such as "northern blotting" to identify RNA sequences (also called "northern hybridization"). Furthermore, methylene blue is a safer alternative to another chemical known as ethidium bromide, which is frequently employed in the viewing of DNA on gels in procedures known as "western blots.
- Calculating viable cells in yeast sample: Methylene blue has also been used to calculate the proportion of viable cells in a yeast sample quickly. Dead yeast cells lack an enzyme that decolorizes methylene blue, but living yeast cells do. As a result, when yeast cells are suspended in a dye-containing solution, the dye colours the dead cells blue while leaving the living cells uncolored. Refer here for more details about this use.
- Identifying distinctions between bacterial, viral and fungal diseases: A technique used to identify and distinguish between bacterial, viral, and fungal diseases is the basic staining approach used by Löffler in alkaline methylene blue staining. Because the positively charged methylene blue dye is attracted to negatively charged particles such as DNAs, RNAs and polyphosphates, it is a cationic dye that stains cells blue. Swabbed specimens from patients are spread onto microscope slides, and the

methylene blue solution is placed on the surface. The slide is viewed under a microscope after being covered with a glass coverslip.

- Methylene blue can also be used as a surgical and medical marking stain (albeit it can induce localized tissue inflammation), as a diagnostic agent in renal function testing, and as a vital nerve staining agent.
- Furthermore, methylene blue is employed to enhance the visibility of the interaction between Fehling’s solution and reducing sugars. It is also used as a reagent in volumetric redox titrations.
- methylene blue stain is used in the experiment to increase the visibility of biological components. It selectively stains certain tumor cells of the head, neck and mouth in cancer operations.

Adsorbent: This is the material that is used for adsorption process

Adsorbate: This is a material that is absorbed by an adsorbent.

CHAPTER THREE

MATERIALS AND METHODS

3.1 Materials

Apparatus/Reagents

- Distilled water
- HCl (Hydrochloric acid) 1M
- Ammonium chloride (NH_4CL_2)
- Beakers
- Kiln furnace
- Measuring cylinder
- Syringes
- Specimen bottles
- Heating Apparatus
- Analytical sieves
- Weighing balance
- Mechanical shaker
- Visible spectrophotometer
- Masking tape
- Mortar and pestle

- Naoh - 2M
- Tap water

3.2. Collection of Sample

The palm kernel shells were obtained from a local source, palm kernel mill at Jattu Ozairue, Etsako West Local Government Area, Edo state, Nigeria.

The coconut shells were gotten from a local market, Uchi market, Auchi Edo state, Nigeria.

The dye methylene blue was purchased from Medi Plus.

3.3. Preparation of adsorbent

The palm kernel and coconut shells were washed severally with tap water, and rinsed with distilled water.

The washed palm kernel and coconut shells were sun dried for 24 hours and were taken for carbonization. Carbonization is a process of heating in the absence of oxygen.The palm kernel and coconut shells were carbonized in a locally made kiln furnace at a varied temperature ranging from $300^{o}C$ to $800\,^{o}C$ for one hour, and finally crushed to gain smaller sizes. The crushed palm kernel and coconut shells were made to pass through set of sieve with the following particle size, 53 um, 125 um ,212 um, 300 um, 425 um and 600 um.

The sieves were arranged from the highest particle size 600 um to the lowest particle size, 53um and finally the receiver. The sieves were shaken manually using the hand. The essence of shaking is to separate the size particles that passed through the sieve in order to get the actual particle size. Particle size 212um was selected for analysis.

3.4. Preparation of activated adsorbent

The particle size 212 um of carbonized palm kernel and coconut shells were activated by soaking in 500ml of hot water and 32.2g of NH_4CL_2 in a beaker for 1 hour and was filtered using sieve size 125 um. The wet palm kernel and coconut shells were oven dried for 10 hours at 120 ^oC. In order to wash out the ammonium chloride, the palm kernel and coconut shells were soaked in 1M hydrochloric acid (HCl) solution.The soaked palm kernel and coconut shells were rinsed with hot distilled water several times, to wash out NH_4CL_2. The palm kernel and coconut shells activated carbon, were washed with distilled water and dried in the oven for 3hours at a temperature of 105 ^oC.

3.5. Preparation of dye stock solution

1g of methylene blue was dissolved in 1000 cm^3 of distilled water to give 1000ppm solution using standard flask and was stored in the container as stock solution and was labeled properly.

A serial dilution (5ppm, 10ppm, 15ppm, 20ppm and 25ppm) were made from the stock solution and were labeled appropriately. The 10ppm serial dilution was taken for visible spectrophotometric analysis, in order to know the absorbance. The visible spectrophotometer was calibrated using distilled water. The solution was placed inside the cuvette and the wavelength was set to 665nm, and it was zeroed for calibration. Selected pH was taken from the 10ppm serial dilution.The selected pH which are, pH3, pH6 and pH8 of 300ml of the 10ppm serial dilution were mixed with 1g of the carbonized and activated palm kernel and coconut shells each. The solution was stirred using a stirring rod. And the solution was placed in a cuvette and placed inside the visible spectrophotometer. And the absorbance of each pH was taken and recorded down appropriately.

CHAPTER FOUR

RESULTS AND DISCUSSION

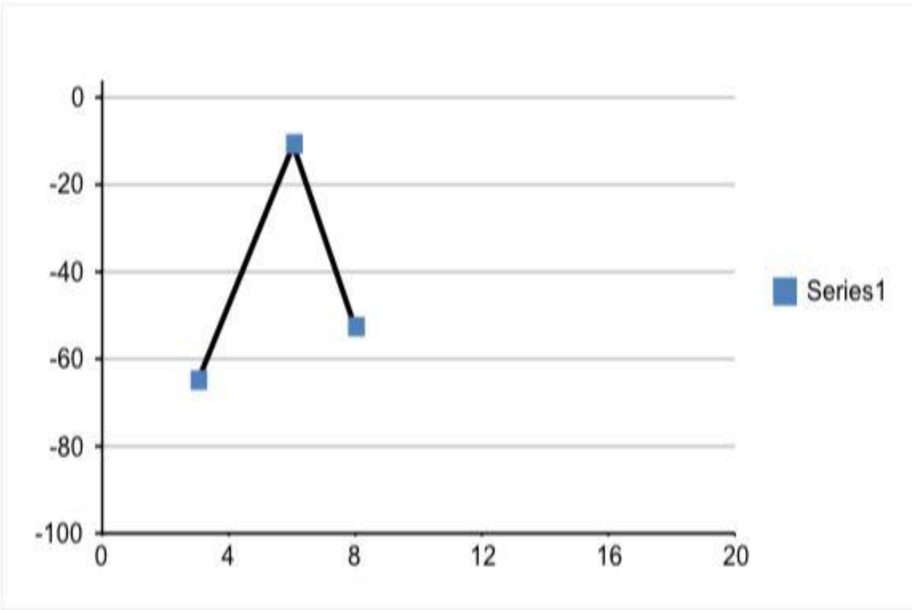
4.1. Below are the results of the experiments carried out

Percentage removal of carbonized and activated palm kernel shell

Initial Conc. (Ce)	Amount removed (qe) Carbonized	Amount removed (qe) Activated	Percentage % Removal Carbonized	Percentage % Removal Activated
1.283	0.216	-0.833	16.836	-64.926
1.283	0.033	-0.139	2.573	-10.834
1.283	0.35	-0.677	27.280	-52.767

Fig 1 & 2: Graph showing the Percentage removal of carbonized and activated palm

3	-64.926
6	-10.834
8	-52.767



kernel shell

Fig 1

3	16.836
6	2.573
8	27.28

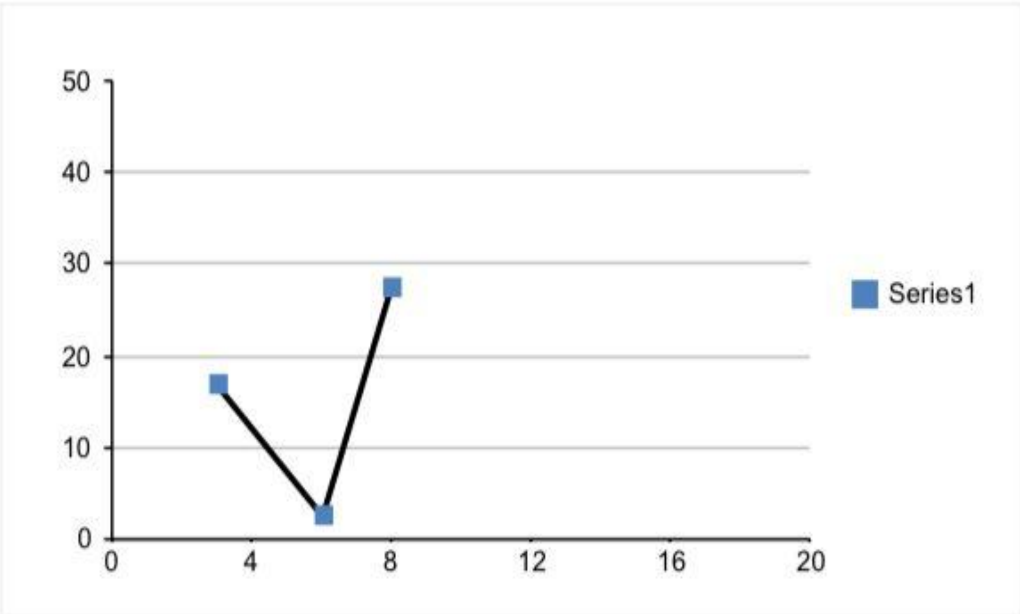


Fig 2

Percentage removal of carbonized and activated coconut shell

Initial Conc. (Ce)	Amount removed (qe) Carbonized	Amount removed (qe) Activated	Percentage % Removal Carbonized	Percentage % Removal Activated
1.283	-0.012	-0.806	-0.935	-62.822
1.283	-0.815	0.249	-63.523	19.408
1.283	-0.537	-0.477	-41.855	-37.178

3	-62.822
6	19.408
8	-37.178

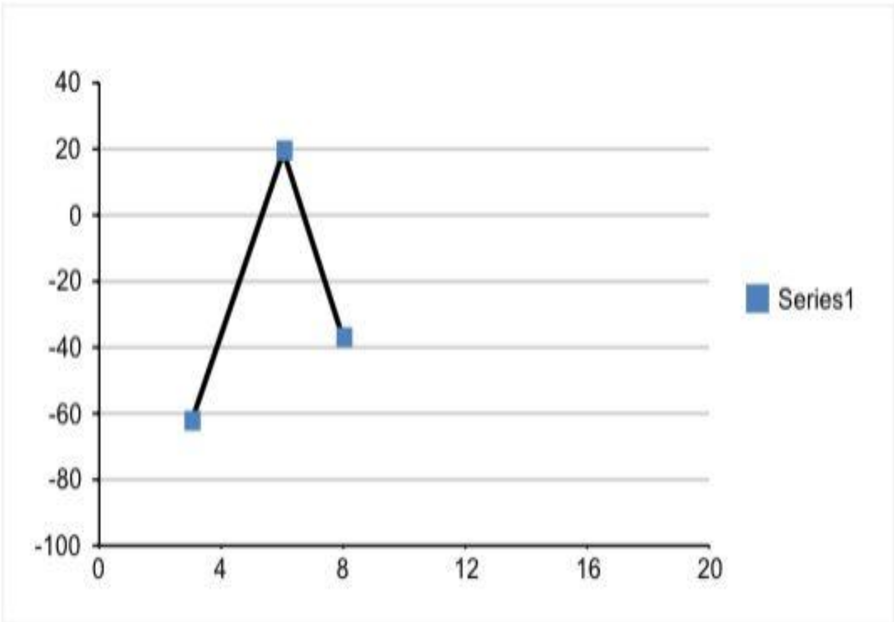


Fig 3 & 4: Graph showing the Percentage removal of carbonized and activated coconut shell

Fig 3

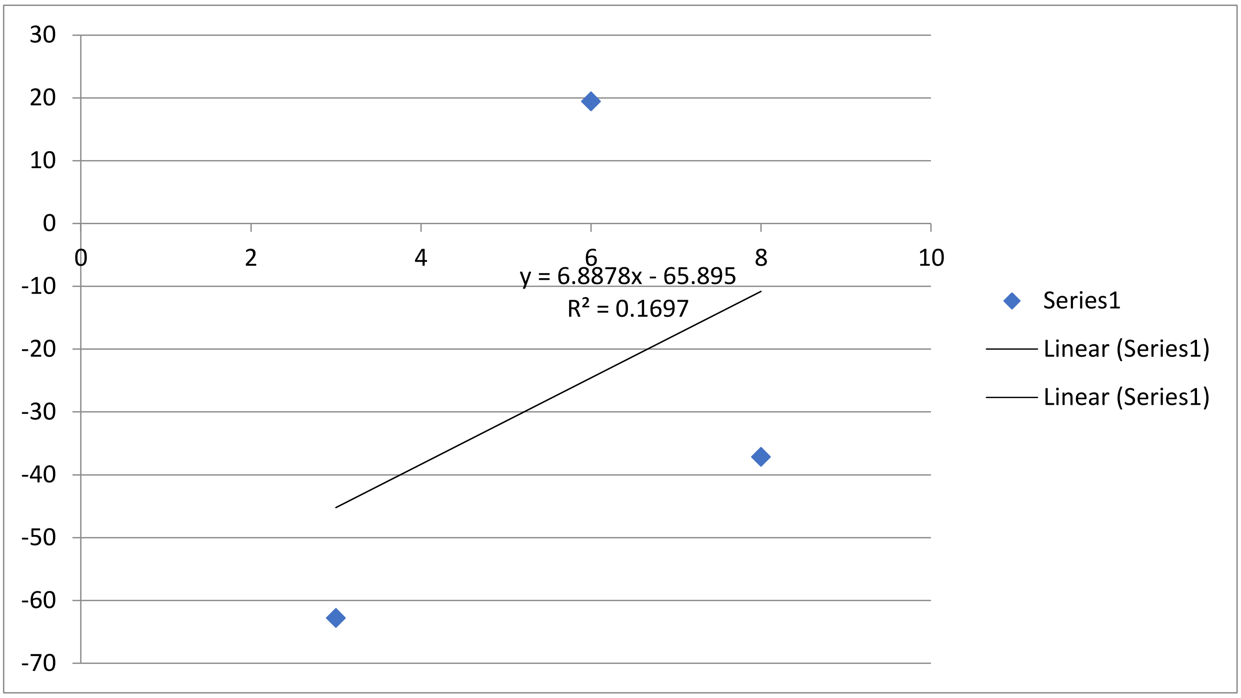


Fig 4

Adsorption capacity of carbonized and activated Palm Kernel shell

PH	Initial Concentration (Ce) C1	Amount Removed C2	V	M	Absorption Capacity Carbonized
3	1.283	0.216	5ml	1g	5.335
6	1.283	0.033	5ml	1g	6.26
8	1.2383	0.35	5ml	1g	4.665

PH	Initial Concentration (Ce) C1	Amount Removed C2	V	M	Absorption Capacity C Activated

3	1.283	-0.833	5ml	1g	10.58
6	1.283	-0.139	5ml	1g	7.11
8	1.2383	-0.677	5ml	1g	9.8

Adsorption capacity of carbonized and activated Coconut shell

PH	Initial Concentration (Ce) C1	Amount Removed C2	V	M	Absorption Capacity Carbonized
3	1.283	-0.012	5ml	1g	6.475
6	1.283	-0.815	5ml	1g	10.49
8	1.2383	-0.537	5ml	1g	9.1

PH	Initial Concentration (Ce) C1	Amount Removed C2	V	M	Absorption Capacity Activated
3	1.283	-0.806	5ml	1g	10.445
6	1.283	0.249	5ml	1g	5.17
8	1.2383	-0.477	5ml	1g	8.8

4.2. Discussion

More of the Methylene Blue was removed at Alkaline Medium than the Acidic Medium and near to neutral.

A sigmoidal curve was obtained and the acid and base peak absorbance gradually change over the pH range from 3 to 8.

Therefore, the absorbance of the acidic medium decreases and that of the basic medium increases as the pH value increases.

CHAPTER FIVE

CONCLUSION AND RECOMMENDATION

5.1. Conclusion

The study was carried out to determine the adsorption capacity of palm kernel and coconut shells from aqueous solution and the variation of PH. Variation of PH study shows that the absorbance of the acidic peak decreases and that of the basic peak increases as the PH value increases.

It is therefore sufficed to conclude that palm kernel and coconut shells could serve as cheap readily available effective absorbent of the removal of MB from waste water as a way of treatment before discharge into the environment.

5.2. Recommendation :

From the study carried out, it shows that:

1. Palm kernel and coconut shells are good adsorbent;It is therefore recommended that more research should be carried out on their agricultural waste products such as melon shell docanut seed shell etc.in order to reduce the cost of imported absorbent.

2. The government should try to provide enough of the necessary machines and equipments needed to this investigation and also made them available for the various higher institutions especially polytechnics for easy research and to save time.

3. The Nigeria government should try to provide more workshops to educate and train personnel on how to manage our agricultural waste product to avoid environmental pollution.

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