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Dye Removal of Textile Waste Water Using Natural Adsorbent

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ABSTRACT
Wastewater discharge into surrounding natural streams has caused surface and subsurface water contamination, leaving water unsafe for potable use and industrial use without major and costly treatment. Colour is a noticeable pollutant and the presence of even minute amounts of colouring substance makes it objectionable due to its appearance. Adsorption is the most used method in physicochemical wastewater treatment, which can mix the wastewater and adsorbent like porous material powder or granules. In the present paper natural adsorbent were used for removal of methylene blue dye from synthetic waste stream which represents textile waste water. The adsorption of Methylene blue, by the natural waste like moringa seed and lemon seed, was carried out by varying the parameters such as contact time, dye concentration, pH, and adsorbent dose. The material were obtained from local fruit market and farm, dried and sieved for adsorption experiments. The adsorbents are capable of removing colour from synthetic waste water, their colour removal capacity for moringa seed powder is 50% and lemon seed is 86% at room temperature and at pH 6 and 7 respectively. The equilibrium time was found about 60 min for moringa seed and 30 min lemon seed. Data obtained from experiments fitted with Langmuir and Freundlich adsorption isotherms. The experimental result shows that, lemon seed powder is much more efficient than moringa seed powder for removal of methylene blue dye. Overall the result shows that the materials have good potential to remove dye and good potential as an alternate low cost adsorbent. Wastewater treated with low cost adsorbents as well as treatment costs reduced at the same time.

Keywords: Textile waste water, Synthetic waste water, Methylene blue dye, Adsorption, Natural adsorbents, Moringa seed, Lemon seed, Adsorption Isotherms

I. INTRODUCTION

Dyes are commonly used in industries like textiles, rubber, plastics, printing, leather, cosmetics, ink, etc., to color their products. As a result, they generate a huge amount of colored wastewater. There are more than 10,000 commercially available dyes with over 7 X 10^5 tonnes of dye produced yearly. It is estimated that 2% of dyes produced annually is discharged in effluents from associated industries (Easton, 1995). Among various industries, textile industry ranks first in usage of dyes for coloration of fibre. Textile industry is the oldest industries in India. It is only second largest industry after agriculture in terms of occupation in country. As developing country with 2nd highest population hub in the world, industrial activities have great potential to grow fast. Textile industry is one of them. The total dye intake of the textile industry worldwide is in excess of 107 kg/year and an estimated 90% of this ends up on fabrics. Consequently, 1,000 tones/year or more of dyes are discharged into waste streams by the textile industry worldwide (Robinson et al., 2001). Though primarily raw materials for textile industry are natural elements, various chemicals, pigments are added later in the various processes to reach at desired end product. Industry uses in large amount of water for the process and output wastewater which is high in color, Biological Oxygen Demand (BOD), Chemical Oxygen Demand (COD), pH, turbidity, temperature, toxics etc. are directly discharged in nearby water sources causing troubles to nature. Discharge of dye-containing wastewater into natural streams and rivers leads severe problems to the aquatic life, food chain and causes damage to the aesthetic nature of the environment. Dyes absorb and reflect sunlight entering water and so can interfere with the progress of bacteria and obstruct photosynthesis in aquatic plants. The problems become severer due to the fact that the complex aromatic structures of the dyes render them ineffective in the presence of heat, light, microbes, and even oxidizing agents and degradation of the dyes become critical (pearce et al., 2003). Dyes can have severe and/or long-lasting effects on exposed organisms depending on the exposure contact time and % of dye concentration. Dyes may cause allergic dermatitis, skin irritation problem, cancer, mutation, etc. Hence, this causes a serious threat to human health and water quality, thereby becoming a matter of vital concern.

Dye removal is one of the major treatments required on waste water as its direct visibility creates panic in living beings on surrounding/downstream of water body. However, color removal from textile wastewater by means of cheaper and environmental friendly technologies is still a big challenge. For removing color industrial wastewater can be treated biologically, chemically and physically (Elsagha A. et al., 2013). The process of waste-water treatment can be selected by some requirements such as cost, characteristics and availability of resources. Physicochemical techniques are widely used primary treatment of wastewater in various industries. These techniques include adsorption, chemical reaction, filtration, ion-exchange, coagulation/flocculation reverse osmosis, electro dialysis etc (Velmurugan et al., 2011). Amongst all treatment technologies, adsorption is rapidly gaining prominence as a method of treating aqueous effluent. Benefits of adsorption process are potential regeneration at low cost, availability of known process equipment, sludgefree operation and recovery of the sorbate (Kapdan and Kargi 2002). Activated carbon is the most widely used adsorbent for dye removal process because of its extended surface area, micro-pore structures, high adsorption capacity and high degree of surface reactivity. The sustainability of adsorption technique is based on the availability of better quality low cost adsorbent. Activated carbon is a big potential for the treatment and used as an adsorbent.

However, the price of commercially available activated carbons is expensive. This is mainly, due to the use of non-renewable and relatively expensive starting material such as coal, which is unjustified in pollution control application. Therefore, in recent years, this has urged a growing research interest in the production of activated carbons from renewable and cheaper precursors which are mainly industrial and natural byproduct, especially for application concerning wastewater treatment. Still it required conversion of natural product in activated carbon. Natural waste materials have little or no economic value and often pose a disposal problem. The utilization of natural waste is of great significance (Geopaul, 1980). Different low cost sorbents have been discovered by various researchers for dye removal from wastewater.

These includes Palm kernel fibre (El-Sayed, 2011), rice husk (Gupta et al., 2006; Lakshmi et al., 2009), sawdust (Batzias and Sidiras, 2007; Khattri and Singh, 2009), tea waste (Uddin et al., 2009), peanut shell (Tanyildizi, 2011), orange peels (Khaled et al., 2009; Arami et al., 2008), wheat shell (Bulut and Aydin, 2006), pineapple stem (Hameed et al., 2009), and coconut based sorbent; babassu coconut mesocarp (Vieira et al., 2009), coconut husk (Jain and Shrivastava, 2008; Low and Lee, 1990; Gupta et al., 2010), coconut shell fibre (de Sousa et al., 2010; Babel and Kurniawan, 2004), coconut coir pith (Namasivayam et al., 2001), coconut bunch waste (Hameed et al., 2008).

The present study is undertaken to explore the efficiency of moringa seeds and lemon seed powder (without physical/chemical activation) as a low cost adsorbent for the removal of methylene blue dye. Methylene blue (MB) is the most commonly used substance for dying cotton, wood and silk. It can cause eye irritations and burns which may be responsible for permanent injury to the eyes of human and animals. On inhalation, it can give rise to short periods of rapid or difficult breathing while consumption through the mouth produces a burning sensation and may cause nausea, vomiting, sweating, mental confusion and methemoglobinemia. Moringa seeds are obtained from dry mature drumsticks/ moringa from the farm and lemon seeds are obtained from used lemon from fruit juice center as they are natural waste i.e. residue.

II. MATERIAL AND METHODOLOGY

2.1 Selection and Preparation of Adsorbent

Moringa seeds and lemon seeds were chosen as adsorbent. As they are easily and locally available also have zero cost. Methylene blue (MB) was purchased from chemist. MB has molecular formula $C_{14}H_{18}N_3SCI$ (Mol. wt. 319.85 g/mol). It was used without further purification. H2SO4 and NaOH were used to vary pH. All reagents were of analytical grade. Double distilled water was used throughout the experiment. Instruments used for the work include UV–visible spectrophotometer (Spectroquant pharo 300), FTIR spectrophotometer (3000 Hyperion microscope with vertex with 80 FTIR system), mechanical shaker 20- 880, weighing balance, pH-meter-16 and sieves of about 300-600 µm size.

Lemon seeds were collected from juice centre and moringa seeds were collected from farms. After selecting the adsorbent they were cleaned by distilled water until the clean water comes as wash water. After that it was sun dried near about for a week and powdered with the help of home grinder. It was sieved using a 450 - 600 µm sieve to obtain particles in this range. Particle retain on 600µm sieve were used for experiments. This obtained powder was stored in a plastic container prior to use for adsorption studies. No chemical or physical treatments were performed prior to adsorption experiments.

2.2 Dye solution preparation and Calibration of Methylene Blue

Characteristics of methylene blue used for present work are given below table 1. The dye stock solution was prepared by dissolving 1.0 g of dye in double distilled water in a 1 liter volumetric flask and prepared to a concentration of 1 g L-1. The working solutions were obtained by diluting the dye stock solution in accurate proportions to needed initial concentrations (5–50 mg L-1) and were used to obtain a calibration curve shown below.

TABLE 1
PROPERTIES OF METHYLENE BLUE

Chemical Formula	**$C_{14}H_{18}N_3SCI$**
Molecular weight	319.85 g/mol
Melting point	100-110˚C
Types of dye	Basic blue
Boiling point	Decomposes
λ_{max}	665 nm

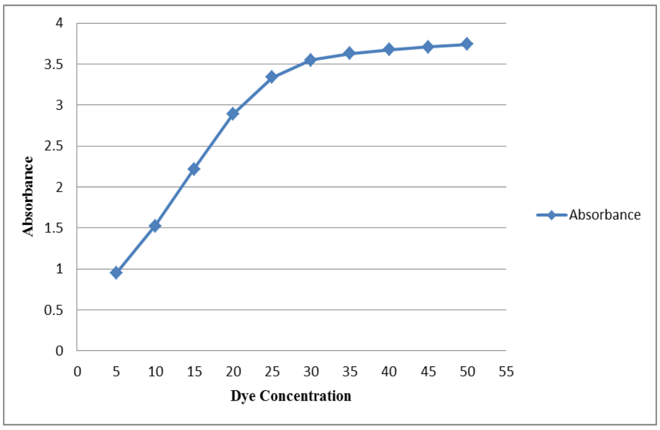


Figure.1: Calibration curve of methylene blue on spectrophotometer.

2.3 Batch Adsorption experiments and Study

The batch adsorption experiments were conducted to study optimum removal of colour from textile waste water. It was done for 10, 20, 30 mg/l initial concentrations. The equilibrium time and optimum dose of adsorbent were determined by repeating the same experiment at different conditions. The effect of contact time, concentration of the dye, and amount of adsorbent and pH of methylene blue (MB) adsorption were studied for all three concentrations. The batch adsorption experiments were carried out in 250 ml flask and total volume of solution was kept 50 ml throughout the experiments. The flasks were shaken at 150 rpm on mechanical shaker. The effect of time on removal of methylene blue dye was studied for interval 15, 30, 45, 60, 75, 90 min at pH 6 and adsorbent dose 0.5 g. Then samples were withdrawn for analyses using UV visible spectrophotometer by monitoring absorbance changes at λmax 651 nm at room temperature.

The effect of adsorbent dose on removal of methylene blue dye was investigated by varying the adsorbent dose range from 0.1 g to 0.8 g in 50 ml solution. It is obtained by shaking the dye solution up to the equilibrium time at pH 6. The effect of pH on removal of methylene blue dye was investigated at pH values ranging from 3.0 to 11.0 in 50 ml dye solution for equilibrium time. The pH values were adjusted by using 0.1 M HCl and 0.1 M H_2SO_4. The effect of dye concentration on removal of methylene blue dye was studied at different dye concentration. The range of dye concentration taken for study was from 5 mg/l to 50 mg/l. After adsorption experiment the adsorbent and the supernatant were separated by centrifugation at 3000 rpm for 10-15 min. Then samples were withdrawn for analyses using clinical syringe and analyzed for residual dye concentration using UV visible spectrophotometer by monitoring absorbance changes at λmax 651 nm, at room temperature. The amount of dye adsorbed per gram of adsorbent (qe) is given as

$$q_e = \frac{V}{M} (C_e - C_0) \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

The percentage removal (R) was calculated using Eq. (2)

$$\%R = \frac{Ce - C0}{C0} X100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (2)$$

Where, Co and Ce are the initial and equilibrium MB concentrations respectively (mg L^{-1}), V is the MB solution volume (L) and m is the mass of the adsorbent (g).The equilibrium data were evaluated using the Langmuir and Freundlich isotherm and characteristic parameters for both isotherms were determined.

2.4 Spectral analysis

The spectra of moringa seed powder and lemon seed powder were recorded by a FTIR spectrophotometer (3000 Hyperion Microscope with Vertex 80 FTIR System) in the range of 4500–500 cm-1. The bands and stretch vibration occur in IR graph shows the presence of various functional groups like hydroxyl, aromatic, aliphatic, carboxylic acid groups on both samples. All peaks show a shift to a new wavelength which is an indication of the involvement of the functional groups in the adsorption process of methylene blue.

III. RESULT AND DISCUSSION

Results were compare between the efficiencies of adsorbents on removal capacity of methylene blue dye. It was done on the basis of batch experiments. Adsorption was studied as a function of amount of adsorbent, pH, time and initial dye concentration. The colour removing capacity of adsorbent is compared for initial concentration 20 mg/l.

3.1 Effect of contact time

To study the effect of time on removal of methylene blue dye the experiments were done for 3 different concentrations which are 10mg/l, 20mg/l, and 30 mg/l at pH 6. From the comparative results, it is clearly known that, time plays important role in adsorption process of colour removal. It was observed that uptake of dye was fast at initial stages of contact time and there after becomes near equilibrium and reached a steady value at equilibrium for all 3 concentrations. The color removal efficiency of the moringa seed powder have break through at 60 min, 60min, 75 min for 10mg/l, 20 mg/l and 30mg/l dye concentration respectively. It gives maximum efficiency of 75% for removal of dye at 60 min for 10 mg/l. It gives efficiency of 50.6% for 20mg/l and 38.34% for 30mg/l concentration which is minimum. The color removal efficiency of the lemon seed powder have break through at 45 min for 10 mg/l and at 30 min for both 20 mg/l and 30mg/l dye concentration. It gives maximum efficiency of 94.5% for removal of dye at 45 min for 10 mg/l and 86% for 20mg/l and 82.33 % for 30mg/l concentration. From result it is clear that lemon seed are much more efficient than the moringa seed powder. Lemon seed powder has efficiency of 86% and moringa seed has 50.6%. Lemon seed shows higher efficiency very early, at 30 min while moringa seed achieve its maximum efficiency at 60 min.

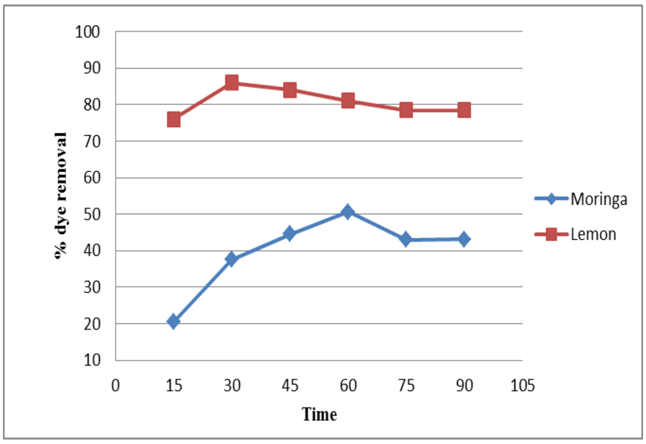


Figure.2: Comparative results for efficiency of both adsorbents with respect to time

3.2 Effect of Adsorbent dose

The adsorbent dose is an important parameter in adsorption studies because it determines the capacity of adsorbent for a given initial concentration of dye solution. . It is found that adsorption of dye decreases or nearly reach to the equilibrium further increase in dosage for all the three concentration. Due to overlapping or aggregation of adsorption sites resulting in a decrease in the total adsorption surface area available to the dye and an increase in the diffusion path length. Lemon seed found to be very effective with color removing efficiency of 84.5 % at adsorbent dose 0.5 g in 30 min duration whereas moringa seed gives efficiency of 50.6 % at 0.4 g in 60 min duration. Though moringa seed powder achieve its optimum point at lower adsorbent dose 0.4 g than lemon seed powder but it gives only 50.6 % of efficiency.

As the dose increases the dye removing capacity of adsorbent also increases up to a certain limit. From graph obtain below we can say that, after achieving the optimum value of adsorbent dose the adsorption becomes slower and near to equilibrium. It is found that adsorption was decreasing further increasing dose for both adsorbent powder.

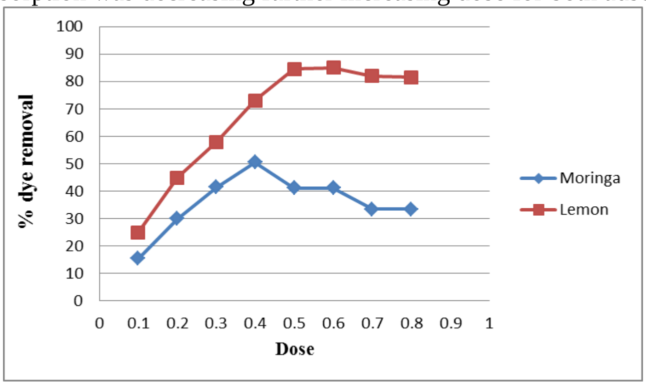


Figure.3: Comparative results for efficiency of both adsorbents with respect to adsorbent dose.

3.3 Effect of pH

The pH factor is very crucial in adsorption studies especially dye adsorption. The pH medium controls the magnitude of electrostatic charges which are imparted both by the ionized dye molecules and the charges on the surface of the adsorbent. The rate of adsorption changes with the pH of an aqueous medium. To study the effect of pH on colour removal capacity of moringa seed powder, colour removal was studied at pH ranging between 3 to 11. The pH values were adjusted by using 0.1M HCl and 0.1 M H_2SO_4. Lower adsorption of MB at highly acidic pH may be due to the presence of excess H+ ions competing with dye cations. As the solution pH increased, the number of negatively charged surface sites on the adsorbent increased, which may result in the increase in adsorption of cationic dye molecules due to the electrostatic attraction. The percentage of adsorption had change within the pH range of 3–11, which indicated that the electrostatic mechanism was not the only mechanism for dye adsorption in the solution but was also affected by the chemical reaction between the adsorbent and dye molecules. Color removal under acidic condition found to be less efficient, whereas for near alkaline condition it is more efficient. From the obtained results, it is clear that both adsorbent gives their maximum efficiency near or at neutral pH. Lemon seed powder has efficiency of 87 % at pH 7 and moringa seed powder has 50.8 % at pH 6. Lemon seeds are more effective than moringa powder for removal of dye methylene blue by adsorption process.

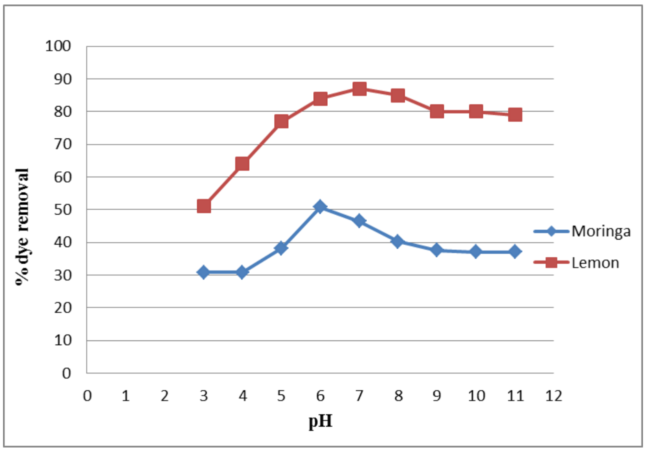


Figure.4: Comparative results for efficiency of both adsorbents with respect to pH

3.4 Effect of initial dye concentration

The percentage of dye removal is highly dependent on the initial amount of dye concentration. Relation between the dye concentration and the available binding sites on an adsorbent surface depend on the initial dye concentration factor. Generally the percentage of dye removal decreases with an increase in initial dye concentration, which may be due to the saturation of adsorption sites on the adsorbent surface and the adsorption capacity increased with an increase in the initial concentration of the dye. Result shows that residual concentration of dye molecules will be higher for higher initial dye concentrations. In the case of lower concentrations, the ratio of the initial number of dye molecules to the available adsorption sites is low and subsequently the fractional adsorption becomes independent of the initial concentration. As we know, dye removal efficiency depends upon the initial dye concentration. Graph plotted below shows the percentage of dye removal decreases with an increase in initial dye concentration. As the initial concentration increases the removal efficiency of both adsorbents decreases. The maximum efficiency for lemon seed is observed 94 % at 5 mg/l and for moringa 92 % at 5 mg/l. The minimum efficiency for lemon seed is observed 82 % at 50 mg/l and for moringa seed it is 35.6 % at same 50 mg/l dye concentration. Effectiveness of moringa seed powder as a adsorbent was reduces much more. By comparing all above results lemons seed powder is more effective than moringa seed powder for removal of methylene blue dye.

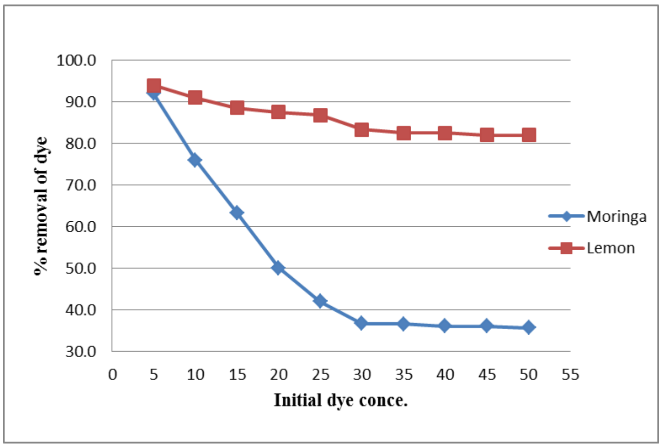


Figure.5: Comparative results for efficiency of both adsorbents with respect to initial dye concentration

3.5 Adsorptoin isotherm

Adsorption isotherm provides important models in the description of adsorption behaviour. It describes how the adsorbate interacts with the adsorbent and offers explanation for the nature and mechanism of the adsorption process. The study of isotherm data is significant to find out the adsorption capacity of various adsorbents. In order to investigate the adsorption isotherm, two equilibrium isotherms were analysed: Langmuir and Freundlich isotherms are used for fitting the experimental data in adsorption studies to recognize the extent and degree of favourability of adsorption.

The Langmuir model was developed based on the assumption of the formation of a monolayer of the absorbent species onto the surface of the adsorbent. The study of the Langmuir isotherm is necessary in evaluating the adsorption efficiency of the adsorbent. This study is also useful in improving the operating conditions for effective adsorption. The plots of 1/Qe against 1/Ce, for both adsorbents were shown in graph below. The experimental data were plotted in graph and the obtained equation from the graph is compared to the y = mx + c equation. Putting the values from graph the value of R^2, K_L and q_{max} are obtained, listed in Table no.1. The graph formed a straight line which indicates that the system fits the isotherm. Langmuir equation is given below.

$$q_e = \frac{q_{max} \; Kl \, Ce}{1 + Kl \, Ce} \quad \ldots\ldots\ldots\ldots(3)$$

This can be linearized to

$$\frac{1}{qe} = \frac{1}{q_{max}.KL.Ce} + \frac{1}{q_{max}} \quad \ldots\ldots\ldots\ldots(4)$$

Where, Ce is the equilibrium concentration (mg/L), q_e is the amount of dye adsorbed per unit mass of adsorbent at equilibrium (mg/g), q_{max} is the theoretical maximum adsorption capacity (mg g^{-1}), K_L is the Langmuir isotherm constant (L mg^{-1}).

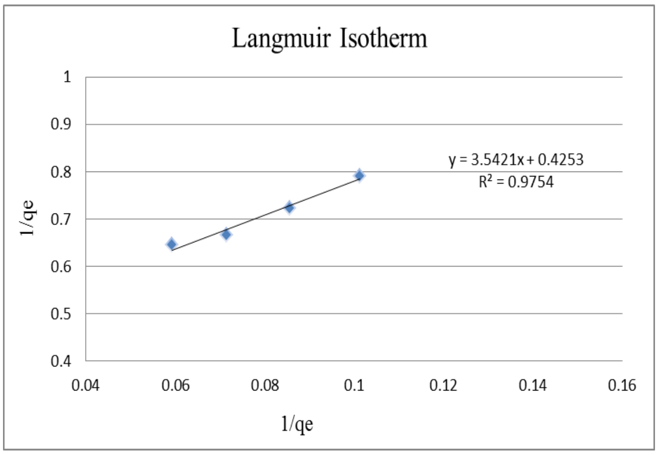


Figure. 6: Graphical Representation of Langmuir Isotherm for moringa seed powder.

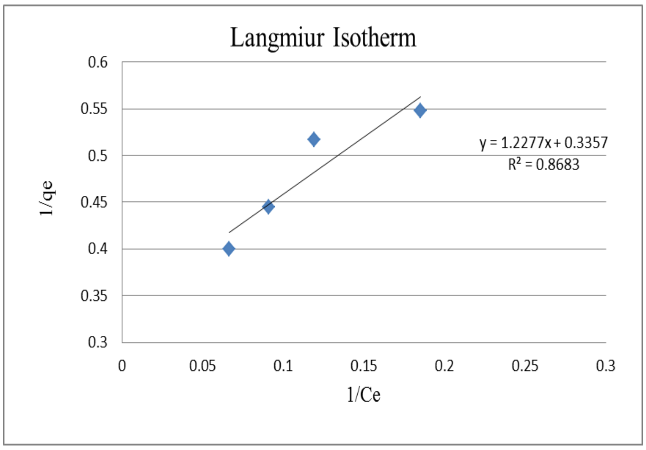


Figure 7: Graphical Representation of Langmuir Isotherm for lemon seed powder.

TABLE 2
LANGMUIR CONSTANTS FOR VARIOUS ADSORBENTS

Adsorbent	Langmiur Constant		
	Q_{max}	KL	R2
Moringa seed	2.351	0.12	0.9754
Lemon seed	2.9788	0.273	0.868

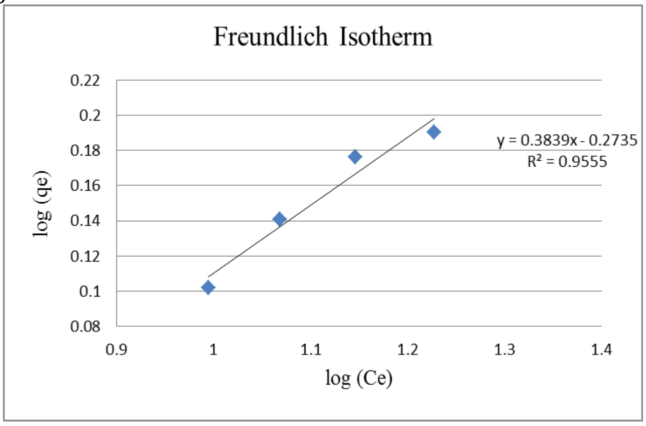
The equilibrium adsorption isotherms are of fundamental importance in the design of adsorption systems. The equilibrium adsorption data could be satisfactory by the Freundlich isotherm. The Freundlich equation is given as:

$$q_e = K_f \, Ce^{1/n} \quad \ldots\ldots\ldots\ldots(5)$$

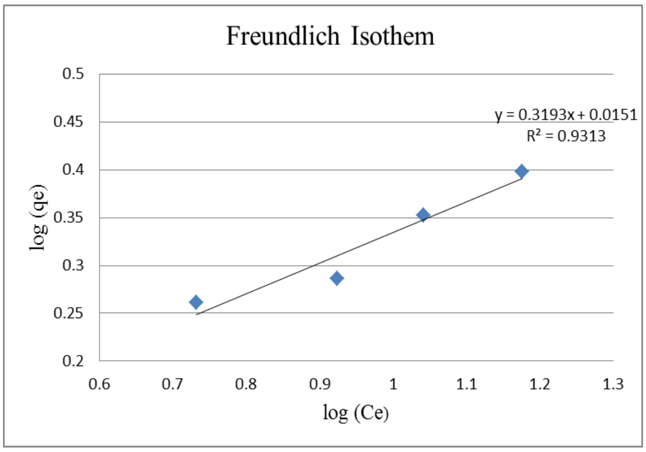
where q_e is the amount of dye adsorbed per unit mass of adsorbent at equilibrium (mg/g), Ce is the equilibrium concentration (mg L^{-1}), K_f is the Freundlich adsorption constant related to the adsorption capacity of the adsorbent and n, a dimensionless constant, which can be used to explain the extent of adsorption and the adsorption intensity between the solute concentration and adsorbent respectively. A linear form of the Freundlich equation is generally expressed as:

$$\log_e q_e = \log_e K_f + (1/n) \log_e C_e \qquad (4)$$

The experimental data from table 3 were plotted on graph no. 8 and 9 and the obtained equation from the graph is compared to the y = mx + c equation. The plots of Log Qe against Log Ce are shown below. The formation of straight line indicates that the system fits the Freundlich Isotherm. Putting the value from the graph on the isotherm equation following data were obtained given in table no.3



Graph no.8: Graphical Representation of Freundlich Isotherm for moringa seed powder



Graph no.9: Graphical Representation of Freundlich Isotherm for lemon seed powder.

TABLE 3
FREUNDLICH CONSTANTS FOR VARIOUS ADSORBENTS

Adsorbent	**Freundlich Constant**		
	1/n	**K_f**	**R^2**
Moringa seed	0.3839	0.53272	0.9555
Lemon seed	0.3193	1.0353	0.9313

The value of R indicates the type of the isotherm. Value of R was found favorable for both adsorbent. This again confirmed that the Langmuir isotherm and Freundlich isotherm were favorable for adsorption of MB on natural adsorbent under the conditions used in this study. The value of R indicates the type of the isotherm. If $R > 1$ isotherm is unfavorable, If $R = 1$ isotherm is Linear, if $0 < R < 1$ isotherm is Favorable or if $R < 0$ isotherm is Irreversible.

IV. CONCLUSION

The present study shows that moringa seed powder and lemon seed powder were a capable to be used for the removal of methylene blue dye from aqueous solutions. Methylene blue was found to adsorb strongly onto the surface of both adsorbents. The functional groups present in the proximate composition of the moringa seeds and lemon seeds were investigated by Fourier transform infrared (FTIR) spectroscopy. Data obtained from this experimental work discovered that lemon seed powder is more effective natural adsorbent than moringa seed powder for the removal of MB dye. Moringa seed gives near about 50% efficiency for removal of MB dye whereas lemon seed gives 86% at room temperature, which is much more than moringa seed's efficiency. For moringa seed powder it gives its maximum efficiency at pH 6 and for lemon seed at pH 7. The isotherm analysis revealed that lemon seed prepared under nominal treatment was found to be very effective than the moringa seed. Adsorption parameters calculated from Langmuir, Freundlich isotherms are useful for the explanation of the mechanisms of the adsorption process as indicated by the good linear correlation coefficient values. Overall natural adsorbent like lemon seed and moringa can be a good replacement for activated carbon in adsorption process.

V. ACKNOWLEDGMENTS

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