Synthesis, Characterization of Activated Carbon prepared from S*echium edule* and its application for the removal of Rhodamine B from aqueous medium.

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ABSTRACT *Among various environmental pollutants, dyes have been reported to contaminate a large quantity of surface water. Rhodamine B, a widely applicable basic dye is known for its hazardous impact on environment. The present work proposes the production, characterization of activated carbon and its application for the removal of Rhodamine B from aqueous medium. The activated carbon was prepared from Sechium edule commonly known as chayote by chemical activation using nitric acid (HNO_3) as an activating agent. The prepared activated carbon was used for the characterization of matter soluble in water, matter soluble in acid, conductivity, pH, apparent density, iodine number, determination of zero-point charge, determination of surface functional groups by Boehm's titration and proximate analysis which includes moisture content, volatile matter content, ash content and fixed carbon followed by the application for the removal of rhodamine B by preparing Stock solution and testing the concentration with the help of UV-VIS spectrophotometer.*
Keywords: Pollutants, basic dyes, activated carbon.

I.   INTRODUCTION

Dyeing industry effluent can create serious environmental pollution problems when they are discharged into water bodies [1]. The disposal of wastewater through various industries is a source of several hazardous pollutants which include toxic organic, inorganic, dissolved solids, dyes, acids and bases etc. [2-5]. Among all contaminants, dyes seem to be the most prominent as it can be easily recognized by human eye. It is due to the fact that water gets color in the presence of dyes [6]. Dyes are defined as colored substances that when applied to fibers impart them a permanent color which are resistant to action of light, water and soap [7]. Over 10,000 types of dyes have been manufactured and production is more than 70,000 tons per year worldwide [8]. Dyes have been used in many industries such as paper, textiles, plastics, cosmetics and pharmaceutical industries [3,9]. Dyes are found to be toxic, non-biodegradable and stable. These can be carcinogenic, genotoxic, mutagenic and teratogenic in nature [8-10]. So, it is important to adopt some treatment methods before discharging the effluents into water to reduce the harmful effects. The methods employed for the removal of dyes from wastewaters include anaerobic decolourization [11], chemical oxidation [12], reverse osmosis [13], ion exchange [14], adsorption [15], and biological treatment methods [16]. Among these methods, adsorption onto activated carbon has been proven to be the most effective method for the color removal in terms of initial cost, simplicity of design, easy operation and insensitivity to toxic substances [17]. Adsorption is more advantageous than the other conventional methods due to its sludge free clean process and high adsorption capacity for the coloured particles. However, usage of commercial activated carbon for adsorption has some limitations like high cost and regeneration problems. This has necessitated the exploration of some effective, cheaper and easily available alternatives. Such alternatives include low cost activated carbons prepared from natural products like date pits [18], bagasse [19], fruit stones and nut shells [20], jute fiber [21], rice husks [22] and plum kernels [23]. Activated carbons prepared from various products are widely used as adsorbents due to their high adsorption capacity, large surface area and microporous structure. The wide usefulness of activated carbon as adsorbent is a result of their chemical and thermal stability [24].

1.1. HARMFUL EFFECTS OF DYES

➢   They pose acute as well as chromic effects on most of the exposed organisms. These effects vary depending on the time of exposure and the concentration of dyes.

➢   They can absorb or reflect sunlight which enters the water bodies and thus affect the growth of bacteria and cause an imbalance in their biological activities.

➢ They are highly visible and even a minor amount may cause abnormal colouration of water bodies [25,26].

➢ They consume dissolved oxygen and affect the aquatic ecosystem [26].

1.2 RHODAMINE B

A class of toxic organic dyes is Rhodamine which is used in textile printing. It ishighly used due to its property to provide resistance to light, heat, rubbing and washing. Among the different rhodamines, rhodamine B (RHB) is an aromatic, water soluble and basic dye [27]. It is widely used in tissues and foodstuff as oneof the main colorants [28]. In a generic aquatic perimeter, Rhodamine B are difficult to remove from the aqueous media causing harm to the receiving water bodies [29]. Rhodamine B is considered as a toxic colorant due to its harmful effects on human beings such as allergic dermatitis, skin irritation, mutation andcancer [4].
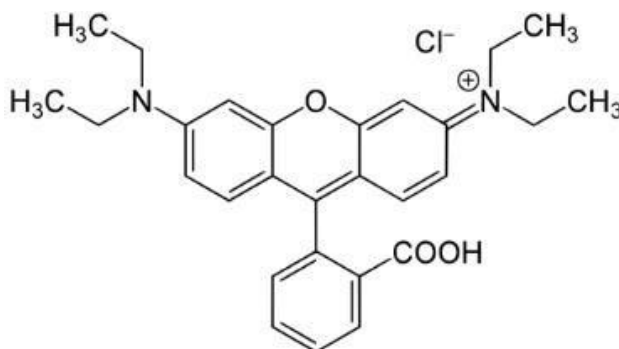

Fig 1: Structure of Rhodamine BMolecular formula: $C_{28}H_{31}ClN_2O_3$

II.   MATERIALS AND METHODS

2.1 Raw materials for Activated Carbon

The raw materials taken for the preparation of activated carbon was Chayote stems and leaves which were collected from local household in Kohima, Nagaland. It was then washed with water and kept in the sun for drying for 2-3 days. Finally, the dried stems and leaves were used as bio-waste materials for the synthesis of activated carbon.

2.2 Preparation of Activated Carbon

By pyrolysis: Powdered activated carbon was prepared by the pyrolysis of the bio-waste material *Sechium edule.* In this method, the bio-waste was collected, washed, dried and crushed. It was then carbonized at a temperature of 900℃ for 4 hours in a muffle furnace. The prepared activated carbon was then cooled at room temperature and washed with deionized water until the effluent was clear. Finally, the synthesized activated carbon was dried in an oven at 60℃ for 24 hours.

By chemical activation: The synthesized carbon was chemically activated with 0.1 N of HNO_3 solution to modify the chemical structure. Structure modification was also done by subjecting the activated carbon to liquid phase oxidation.

2.3 Preparation of Stock solution

A stock solution of Rhodamine B was prepared by dissolving 1 g of RhodamineB in 1000 ml distilled water and subsequently dilute it with distilled water up to the mark of the volumetric flask. The resultant solution contains 1000

mg/L of Rhodamine B and this solution is known as the stock solution.

2.4 Physical Characterization of Activated carbons

i. Matter soluble in water:

1 g of *Sechium edule* activated carbon was added to 100 ml of distilled water and shaken thoroughly for 30 minutes and then filtered. The residue was then dried, cooled and weighed [30].

Calculation:

Matter soluble in water (%) = [(C-D)/(C-B)] x 100

Where,   B = Weight of the crucible(g)

C = Weight of the crucible with original sample(g)

D = Weight of the crucible with cover + dried sample(g)

ii. Matter soluble in acid:

0.5 g of *Sechium edule* activated carbon mixed with distilled water was placed in an evaporating disk. To it 5-10 ml of concentrated HCl was added and warmed for digestion until the sample is nearly dry. The digestion was repeated three times with 5 ml of acid. It is then diluted with 100 ml of water and filtered using a weighed sintered crucible. The weight of the insoluble matter is calculated after drying for a constant weight at 103℃.
Calculation:

Matter soluble in acid (%) = [(C-D)/(C-B)] x 100

Where, B = Weight of the crucible(g)

C = Weight of the crucible with original sample(g)

D = Weight of the crucible with cover + dried sample(g)

iii. Conductivity:

0.1 g of *Sechium edule* activated carbon was taken and mixed with 50 ml of distilled water. It was then equilibrated for 1 hour by agitating at 120 rpm. Finally, the conductivity was analyzed using conductivity meter.

iv. Determination of pH:

The standard test method ASTM D3838-80 was used for the determination of pH of *Sechium edule* activated carbon [31]. 0.1 g of activated carbon and 100 ml distilled water was measured and was transferred into a beaker. It was then kept in a magnetic stirrer for one hour at 120 rpm. The pH was then determined with the help of a Systronics digital pH meter-335.

v. Apparent Density:

The standard test method ASTM D2854-96 was used for the determination of apparent density [32]. A 25 ml specific gravity bottle was taken and filled with activated carbon and packed with the help of a rubber stopper. The weight of the adsorbent was determined by the formula: weight(g) divided by the volume(ml) which gives the apparent density (g/ml) of the adsorbent.

vi. Iodine Number:

Weight 40 g of KI and dissolve in 100 ml of water. To it add 12.7 g of iodine. Stopper the bottle and shake it until the iodine completely dissolves. Then add 3 drops of concentrated HCl. According to the procedure given by ASTM D4607-94(2006) [33] for determination of iodine number, 0.7-2 g of activated carbon was added to 100 ml of 5% HCl and swirled in a conical flask until the entire activated carbon was wetted. The wetted solution was then boiled for exactly 30 seconds and cooled to room temperature. Next, 100 ml of 0.1 N iodine solution was added to the conical flask. The solution was then filtered by using Whatman 2V filter paper. 50 ml of the filtrate was taken and titrated against 1.1N sodium thiosulphate solution until the yellow color disappears. Then 1ml of starch indicator was added and the titration was continued until the blue color disappears. Equilibrium concentration is determined using the amount of sodium thiosulphate used during titration.

Calculation of iodine number:

$X/M = A - (DF \times B \times S)/M$

Where, X/M = iodine number (mg/g)

$A = 12693 \, N_2$; $B = 126.93 \, N_1$; S = sodium thiosulphate (ml) M = carbon used (g)

N_1 = concentration of sodium thiosulphate (N); N_2 = concentration of iodine (N)

DF = dilution factor; = (I+H)/F ; Where I = initial iodine(ml); H = 5% HCl used(ml)

F = filtrate(ml)

vii. Determination of zero-point charge (pHzpc):

To determine the pH of zero-point charge 0.1 g of activated carbon was added to 200 ml solution of 0.1 M NaCl whose initial pH has been measured and adjusted with HCl. Containers were sealed and placed on a shaker for 24 hours. Finally, the pH was measured. The results were plotted with $pH_{final} + pH_{initial}$ against pH_{final}.

viii. Surface functional groups (Boehm's Titration):

Surface functional group was determined by Boehm titration method [34,35]. 0.1 M solution of NaOH, $NaHCO_3$ and Na_2CO_3 was prepared out of which 50 ml was taken out from each solution and 1 g of activated carbon was added and mixed with each of the 50 ml solution. It was continuously stirred for 24 hours after which the solid phase was separated from the aqueous solution by vacuum filtration. 10 ml of each filtrate was used for the excess acid titration by 0.1 M HCl. The carboxylic group content was calculated by the difference in the amount of 0.1 M Na_2CO_3 consumed by the activated carbon from the amount of 0.1 M NaOH consumed. Phenolic group content on the surface was determined as the amount of 0.1 M $NaHCO_3$ consumed by the sample. The lactonic group content was calculated by the difference between the amount of 0.1 M Na_2CO_3 and 0.1 M $NaHCO_3$ consumed by the activated carbon sample. This method was used for calculating the concentration of

acid groups on activated carbon surface under the following assumptions. NaOH neutralizes phenolic, carboxylic and lactonic groups. Na_2CO_3 neutralizes only carboxylic and phenolic groups whereas $NaHCO_3$ neutralizes only carboxylic groups.

ix.PROXIMATE ANALYSIS

As defined by ASTM proximate analysis is the determination for prescribed methods for moisture, volatile matter, fixed carbon and ash content.

a. Moisture Content:

Determination of the moisture content of activated carbon sample was done by using standard test method [36]. The activated carbon was put in a pre-dried crucible and covered with a lid. The weight was taken to the nearest 0.5mg. Lid was removed followed by heating of the crucible in a ventilated drying oven at a temperature of 150℃ for 3 hours after which the plate was covered quickly and cooled in a desiccator. Finally, the weight was taken. The loss in weight of the adsorbent represents the moisture content.

Calculation:

Moisture content (%) = [(C-D)/(C-B)]x 100

Where,

      B = Weight of crucible with cover(g)

      C = Weight of crucible with cover + original sample(g)

      D = Weight of crucible with cover + dried sample(g)

b. Volatile Matter Content:

Standard test method was used for the determination of the volatile matter content [37]. In this 1g of activated carbon was put in a pre-dried crucible and covered with lid. It was then heated in a muffle furnace for 30 minutes at 950℃ after which the plate was covered quickly and then cooled in a desiccator. It was then finally weighed.

Calculation:

Volatile matter content (%) = [(C-D)/(C-B)]x100

Where,   B = Mass of crucible with cover (g)

      C = Mass of crucible with cover + original sample (g)

      D = Mass of crucible with cover + de-volatized sample (g)

c. Ash Content:

Standard test method was used for the determination of ash content of activated carbon sample [38]. The crucible was ignited for 1 hour at 650℃ in the muffle furnace which after heating was placed in a desiccator and then cooled at room temperature. 1 g of activated carbon was pre-heated at 150℃ for 3 hours and was taken in a crucible for heating in the furnace at 630℃ for 3 hours. The crucible was kept in a desiccator and cooled at room temperature after which the weight was taken.

Ash content (%) = [(D-B)/(C-D)]x100

Where,   B = Weight of the crucible(g)

      C = Weight of the crucible + original sample(g)

      D = Weight of the crucible + ash sample(g)

d. Fixed Carbon:

Determination of the fixed carbon content was done by subtracting the sum of percentage compositions of moisture content, volatile matter content and ash content from 100. The obtained value is the amount of fixed carbon present in the sample expressed in percentage.

Calculation:

Fixed carbon (%) = 100 - (% moisture content + % ash content + % volatile matter content)

2.5 Adsorption Studies of Rhodamine B by Batch method

Batch method is a technique used to study the adsorption capacity of activated carbons. The following solutions of the dye (10ppm, 20ppm, 30ppm,40ppm, 50ppm and 60 ppm) were prepared in a reagent bottle using the prepared stock solution. 0.5 g of *Sechium edule* activated carbon (HNO_3) was added in each of the reagent bottle. Then put the reagent bottles in a shaker for 2 hours. After it is done, the solution was filtered with the help of Whatman filter paper. It was followed by the preparation of different solution concentration again but without adding activated carbon (HNO_3). Then the concentration of each solution was tested using UV-VIS spectrophotometer.

After which the percentage removal, equilibrium concentration and amount adsorbed of each solution

concentration was calculated by the formulas given below:

Percentage removal $= 100\,\frac{(C_i - C_f)}{C_i}$

Amount adsorbed $(q_e) = \frac{(C_i - C_f)V}{M}$

where C_i and C_f are the initial and final equilibrium solution concentrations of the dye (mg/ L), V is the volume of the solution (L) and M is the mass of the activated carbon (g).

III. RESULTS AND DISCUSSION

The amount of water-soluble matter of the adsorbent is given in the table below. The result shows that the water-soluble matter is low in the given sample. The acid soluble matter of the activated carbon is given below. The values indicate that the amount of matter soluble in acid in the sample is low. This low value indicates that the carbon is almost insoluble in acid. Thus, this may be used in treating water with acidic nature. The given values show that there is an increase in the electrical conductivity (EC) of the adsorbent. pH of the activated carbon was determined and the result is shown in the table below. The values indicate that there is an increase in the pH of the activated carbon. Here the values obtained indicates that the synthesized carbon have higher apparent densities. Higher the density of the activated carbon greater is the volume activity and thus better is the quality of the activated carbon. The given values shown in the table below were obtained for the determination of iodine number. The values indicate that the iodine number is high. Higher the value of iodine number higher is the degree of activation which enables more adsorption of iodine molecules on the surface. The values of the zero-point charge obtained is above 7 i.e. pH<pHzpc. This indicates that the surface is positively charged which may be due to the presence of basic sites on the surface combining with protons from the medium. The table given below shows the values obtained for the determination of surface functional groups by Boehm's Titration. It is clear from the table that the total basic group is slightly greater than the total acid groups i.e. carboxylic, phenolic, lactonic groups and carbonyl groups. Boehm states that the chemisorptions of oxygen suggest the basicity maybe due to oxygen functional groups and the existence of pyrone-type structures on the edge of the poly-aromatic layers. Support for the hypothesis of pyrone-type structure came from theoretical calculations which showed that the base strength increases strongly when the carbonyl group and the ring oxygen of a pyrone-type structure are distributed on polycyclic aromatic compounds [40]. The values obtained for the determination of moisture content of the activated carbon are shown in the table below. It indicates that that the moisture content in the sample is less. The results obtained for volatile matter content are shown in the figure below. The values indicate that the sample contains fewer volatile materials. This may be due to the high surface area and high adsorption capacity of the activated carbon sample towards various volatile matters. The results obtained for ash content of the activated carbon are shown in the table below. The values indicate that the percentage of the ash content is less. This indicates that the raw material can be used as a source of activated carbon. A good activated carbon must have low ash content [31]. Small increase in the activated carbon causes a decrease in the adsorptive properties of the activated carbon. With increase in the carbonization temperature there is an increase in the ash content of the sample [39]. The results obtained for the fixed carbon content of the activated carbon is shown in the table. The values indicate that the fixed carbon content of the sample is high.

Table:1 Surface properties of the adsorbent

Sample SEAC (HNO₃)	
Matter soluble in acid (%)	5%
Conductivity (micromhos)	216
Matter soluble in water (%)	0.5%
pH	7.4
Apparent Density(g/ml)	0.3036
Iodine number (mg/g)	962.12
pHzpc	7.5
Moisture content%	2.5
Volatile matter content %	24
Ash content %	6.6
Fixed carbon %	69.4
Carboxylic (meq/g)	10
Phenolic (meq/g)	25
Lactonic (meq/g)	5
Total basic groups (meq/g)	30

The results obtained from UV Spectrophotometer for the removal of rhodamine B dye by using *Sechium edule* activated carbon (HNO₃) are shown in the table below:

Table 2: Initial (C_i), final (C_f) and equilibrium concentration (Ce)of Rhodamine B dye

C_i	C_f	$C_e = C_i\text{-}C_f$	% removal
10 PPM	1.4	8.6	86
20 PPM	3.5	16.5	82.5
30 PPM	7.3	22.7	75.7
40 PPM	18	22	55
50 PPM	23.9	26.1	52.2
60 PPM	30	30	50

The data obtained shows that an increase in initial concentration enhances the interaction between the dye molecules and the surface of the activated carbon. The dye molecules have to encounter the boundary layer effect before diffusing from boundary layer film onto the adsorbent surface followed by diffusion of the dye into the porous structure of the adsorbent which eventually will take relatively longer time [41]. In addition, increasing the initial dye concentration increases the number of collisions between dye molecules and the adsorption which enhances the adsorption process.

A graph was plotted for % removal vs initial concentration using the obtained values.
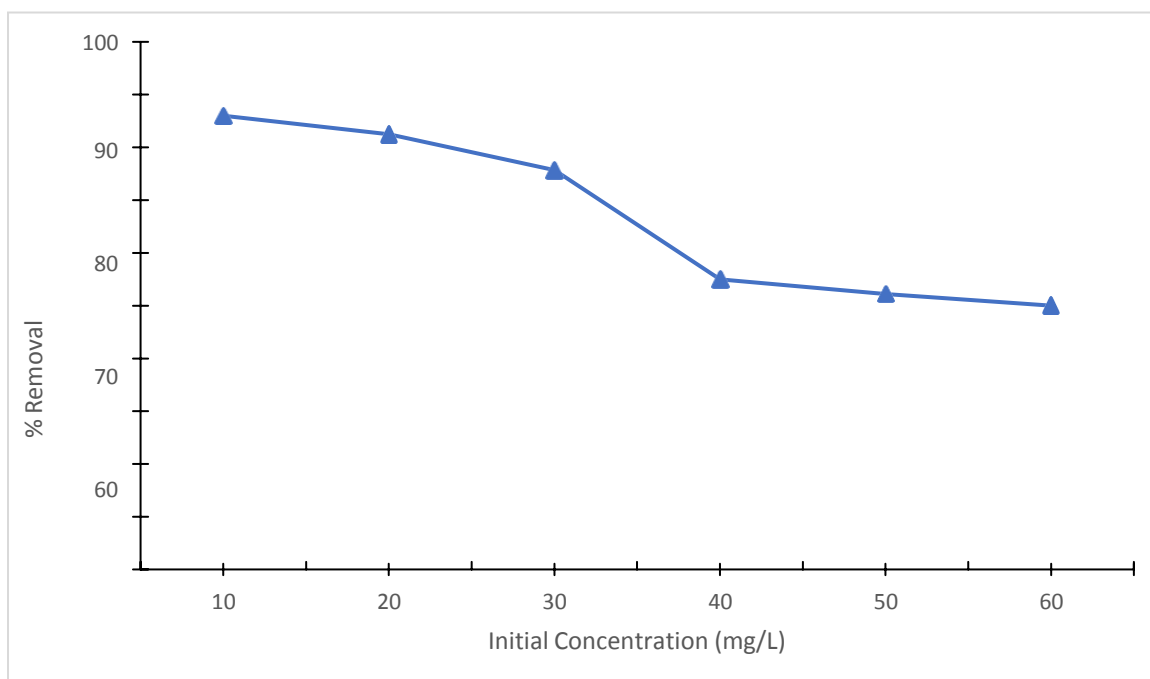

Fig 2: Variation of % removal of Rhodamine B with increasing initialconcentration.

The results displayed in fig 2 shows that % removal of Rhodamine B decreased from 86% to 50% with increase in the initial concentration from 10 PPM to 60 PPM which means that the adsorption is highly dependent on the initialconcentration of dye. It is because of the fact that at lower concentration the ratioof the initial number of dye molecules to the available surface area is low subsequently. The fractional adsorption becomes independent of initial concentration. However, at high concentration the available sites of adsorption become fewer and hence the % removal of dye becomes dependent upon initial concentration [24,41]. The initial concentration provides an important driving force to overcome all mass transfer resistances of Rhodamine B between the aqueous and solid phases. Hence, a higher initial concentration will enhance the adsorption process [42].

IV.   CONCLUSION

After preparation of the activated carbon from *Sechium edule* physical characterization of the activated carbon was done and it was found that the matter soluble in water was low i.e. 0.5 which indicates that the prepared activated carbon is well suited for ground and surface water analysis. The value of apparentdensity was

0.3036 g/ml which means that higher the density of the activated carbon greater is the volume activity and thus better is the quality of the activatedcarbon. The pH of the given activated carbon was 7.4 while that of the iodine number was 962.12 mg/g which indicates that higher the value of iodine number higher will be the degree of activation which enables more adsorption of iodine molecules on the surface. Moisture content of the adsorbent was found to be 2.5 whereas that of fixed carbon was found to be 69.4%. Higher the percentage of fixed carbon, greater is the carbon content found in the material left after volatilematerials are driven off. Thus, better activated carbon is produced when the percentage of fixed carbon is higher.

The removal of Rhodamine B by *Sechium edule* activated carbon was investigated and was observed that as the initial concentration increased from 10 PPM to 60 PPM the amount adsorbed, q_e increased this is because the increase in initial concentration enhances the interaction between the dye and adsorbent. It was also observed that % removal of the dye decreases with increase in the initial concentration i.e. from 86% to 50%. The percentage removal of Rhodamine B dye was highest at 10 PPM i.e. 86% due to the reason that at lower concentration, maximum dye particles in solution occupythe binding sites on adsorbent which results in better adsorption whereas the % removal of Rhodamine B was lowest at 60 PPM i.e. 50% because at higher concentration the available sites on the adsorbent becomes limited and there is nofurther adsorption indicating that their exist reductions in immediate solute adsorption owing to the lack of available active sites required for high initial concentration of Rhodamine B . Hence the % removal of dye is dependent on initial concentration.

Conflict of interest
There is no conflict to disclose.

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