STUDY OF SURFACE MORPHOLOGY OF BETEL NUT HUSK BY ANALYTICAL METHODS AND ITS APPLICATION IN WATER TREATMENT

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Abstract:
Betel nut husk, a genre of palm, constitutes fibrous part of the fruit. The activated carbon is prepared from the betel nut husk and its lingo cellulosic composition was employed as bio-adsorbent for treatment of water effluent which was contaminated with Basic Red 22 (BR22) dyes. Activated Betel nut husk carbon (BNHC) was proximally analyzed for suitability of its surface for the adsorption phenomenon after its physical and chemical activation by phosphoric acid. The optimum surface area was found to be 312 m2/g after using BET method for surface analysis. The spectral scans such as FTIR, XRD and also SEM images have been garnered to assess the functional groups; changes in surface structure and crystalline nature respectively were implied to evaluate qualitatively the adsorption of dye. The scans for the surface of activated BNHC were recorded pre and post adsorption of dye. Analysis had revealed the alteration in the FTIR pattern in finger print region, SEM images and lines in XRD which can be related with efficient adsorption of dye particles on the interface of adsorbent. Therefore, activated betel nut husk carbon had been proved to be a potential non-conventional bio adsorbent for the uptake of Basic Red 22 dye from the aqueous solution.

Index Terms: Adsorption, Betel Nut Husk, Basic Red 22, FTIR, SEM, XRD

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INTRODUCTION:

Threats due to the polluted water are well known to the human kind now a day[1-3]. Dyes remained constantly a pollutant for the water resources due to bulk use of dye from the leather, textile, paint, pulp etc. industries[4,5]. This dye when get accumulated into water resources are directly or indirectly affecting the life on the blue planet. Its removal from the affected water includes physical, chemical, and biological methods such as ozonization[6], coagulation – flocculation[7], filtration[8]. Adsorption, a simple and effortless process, is one of physical method which involves linkage between dye molecules and the surface of adsorbent. Adsorbent here in the study is non-conventional bio-adsorbent which are greener, safe, economical, and easy to use. Betel Nut Husk carbon (BNHC) is widely used as efficient bio-adsorbent in the remediation of water effluent polluted with dye particles[9,10]. Earlier reports implied the use of betel nut husk for treatment of textile water effluent. The group of basic red dye in literature was eliminated from the water effluent using non-conventional adsorbents i.e., bentonite[11], saw dust[12], baggasse[13], corn cob[13], cane pith[14], coal[15], bark[15], rice husk[15], sewage sludge[16,17], Egyptian bagasse pith[18], tree fern[19], sludge biomass[20,21], natural clay[22,23], wood[24], bamboo[25], perfil[26], pineapple peel[27], blast furnace slag[28] etc. successfully in laboratories. Here, we are investigating the use of BNHC in lessening the tinge of colored water effluent having Basic Red 22 (BR22) dye using analytical methods.

I. MATERIALS AND METHODS

A. Preparation of Adsorbent

Betel nut husk was collected locally after peeling out the betel nut. The husk was grinded and fibrous part was separated. The powder was treated with phosphoric acid (1:1) and heated at 120°C in oven since phosphoric acid is known to maximize the porous nature of carbon and assists in carbonization time and temperature[29]. After chemical activation, it was washed with distilled water and then soaked in the 2% sodium carbonate solution to remove excess acid. After drying at 120°C, the powder was incinerated at 550°C in muffle furnace to increase the carbon percentage. The carbonized husk was again ground and sieved to obtain the uniform particle size of 250 BSS. It was stored in the air tight container.

A. Preparation of dye solution

The BR22 dye was purchased from local supplier, manufactured in Winchem Ind. Company Ltd. The chemical structure is shown in the **Fig. 1**[30]. A stock solution of 1000 ppm was prepared. Lower concentrations were prepared from further dilution of stock solution. For the present study, 25 ppm of BR22 dye was used.

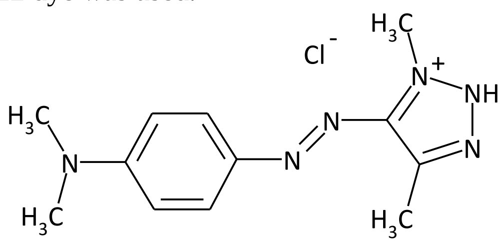

Fig. 1: Structure of Basic Red 22

II. CHARACTERIZATION OF ACTIVATED CARBON

The BNHC was analyzed for its micro, macro and meso pores. The characteristic parameters were tested and 2.8 percent of moisture was found which might not interfere in the study of adsorption process. Therefore, activated BNHC is not hygroscopic in nature. The results obtained are tabulated in the following table no. 1.

Table-1: Physical and Chemical properties of BNHC

Parameters	Obtained Results	Parameters	Obtained Result
Ash content	3.16 %	Bulk density (g/L)	0.47
Water soluble matter	2.1 %	Decolorizing power (mg/g)	22.8
Moisture content	2.8 %	Ion-exchange capacity (meq/g)	0.046
Acid soluble matter	7.5 %	Pore volume (mL/g)	0.12
Oxygen	15.5 %	Pore size (nm)	5.43
Calcium	0.07 %	Phenol number	11.2
Chloride	0.04 %	pH	7.1
Potassium	0.06 %	pHzpc	6.3
Sodium	0.02 %	BET Surface area (m^2/g)	312

It was concluded that activated BNHC has allowed level of contaminants or impurities in its chemical composition. The BET (analyzed on Micrometrics Gemini 2380, micrometrics Instrument Corporation) approach for determining the specific surface area of activated carbon agreed with the external micro-porous area of 312 m^2/g which was comparable with surface areas for better adsorption capacity in the literature[31-33]. According to Indonesian National Standard (SNI No. 06-3730-1995)[34], the admissible limits for the values of properties of activated carbon are depicted in the following table.

Table-2: Quality standards of activated carbon according to SNI (06 - 3730 - 1995)

Sr. No.	Parameter	Unit	Limit
1	Water content	%	Max. 15
2	Ash content	%	Max. 10
3	Volatile matter	%	Max. 25
4	Fixed carbon	%	Min. 60

The lower ash content and higher fixed carbon percentage of the adsorbent was proving the presence of maximum pores making it an ideal choice for the adsorption. Bulk density of the adsorbent particles was found to be 0.47 g/ L. The requirements for activated carbon for Type 1 and Type 2 were partially matching with the Indian Standards (IS 2752: 1995) for BNHC.[35]

I. SEM MORPHOLOGY (SCANNING ELECTRON MICROSCOPY)

The porosity on the surface activated carbon was inferred with SEM study using JEOL Model 6390. SEM images (**Fig. 2**) are pointing towards uneven and rough porosity of BNHC which is a favorable factor for higher adsorption efficiency. Heterogeneous and pores of multiple sizes were noticed in the SEM images. Heterogeneity in surface provides higher surface area which is favorable for the higher adsorption capacity. It was inferred that the dye molecules can diffuse in these pores with ease. After adsorption of dye, partial loss in heterogeneity and fewer hollow micro-pores on the surface were inferred from the SEM images recorded. It can be concluded that the chemical activation of betel nut husk has helped in creating the additional pores and deepening of already present cavities.

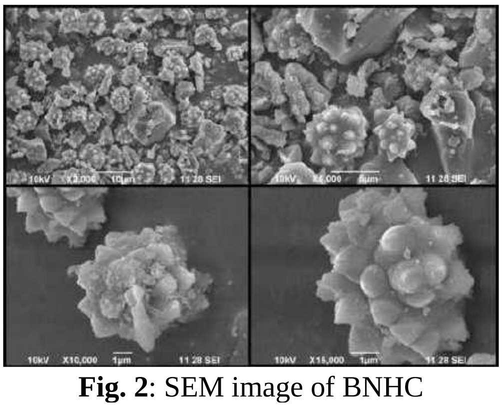

Fig. 2: SEM image of BNHC

SEM images (**Fig. 3**) of Basic Red 22 dye uptake by the surface on BNHC substantiated for monolayer agglomeration of dye molecules on adsorbent. It seems that adsorbate had tried to drill all hole of multiple sizes and maximizing the amount of adsorption.

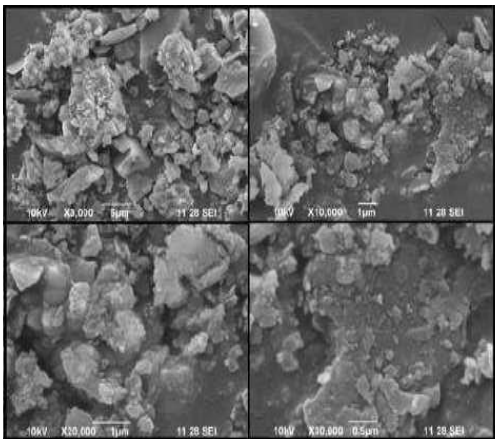

Fig. 3: SEM image of BR22 loaded BNHC

I. DETERMINATION OF FUNCTIONAL GROUP

FTIR (PerkinElmer, USA spectrometer) was employed to see the functional and finger print region of surface of the adsorbent with KBr disc. Functional groups on the surface can be identified by the referring the peaks in the respective region of FTIR spectrum. It was recorded in the range of 4000 – 400 cm^{-1}. Interpretations can be used to conclude about involvement of interactive forces which might play crucial role in the adsorption process. The **Fig. 4** is the FTIR scan of BNHC particles. The peak at 3755.3 cm^{-1} is attributed to $–NH_2$ stretching frequency and peak at 3425.5 cm^{-1} is due to –OH group stretching vibrations. Presence of alkenes and alkyl groups is marked by the peaks at 2854.6 and 2924.1 cm^{-1} respectively in **Fig. 4**. The absorption bands present in the range 1400-1450 cm^{-1} noted in the **Fig. 5** was assigned to the azo bonds[36] which points towards the uptake of dye on adsorbent. The corresponding changes in the finger print region may be due to adsorption of Basic Red 22 dye. Due to the removal of dyes from BNHC surface, some of the peaks FTIR of BNHC have vanished as removal of dye might have to some extent changed the morphological properties of the surface of BNHC.

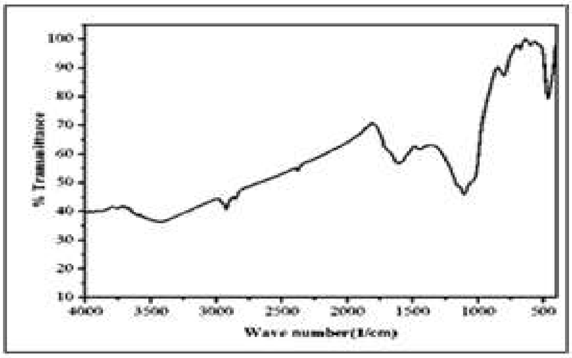

Fig. 4: FTIR spectra of fresh BNHC

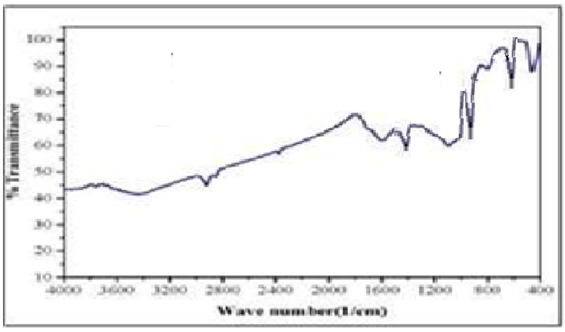

Fig. 5: FTIR spectra of BR22 loaded BNHC

I. X-RAY DIFFRACTION STUDIES

Shimadzu – Model XRD 6000 (Cu-Kα radiation y=1.54A°) with a microprocessor recorder was lined up for the X-Ray diffraction studies. The scattering angle 2θ was maintained for the intensity of the observed rays. The pattern of XRD image for unloaded surface of BNHC (**Fig. 6**) was correlated with the crystalline nature of activated carbon. The prominent peaks in diffraction images were noted at 2θ= 20.0, 21.1, 23.7 and 26.2. The diffraction images of BNHC loaded with dye (**Fig. 7**) exhibited some hanging peaks in comparison with earlier XRD images. The transition in diffraction pattern can be accounted for migration of dye molecules towards cavities of micro and meso pores present and get adsorbed. The accumulation of dye molecules had brought the alteration in crystalline appearance of the adsorbent particles.

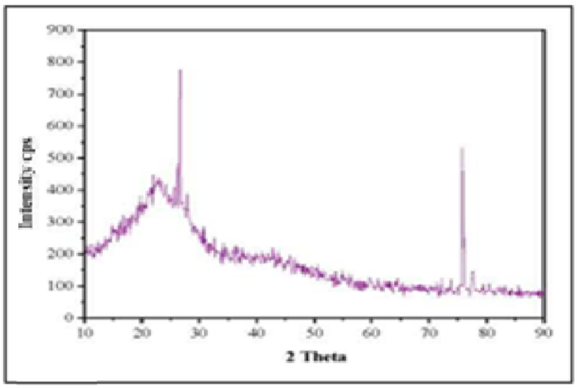

Fig. 6: XRD spectrum of fresh BNHC

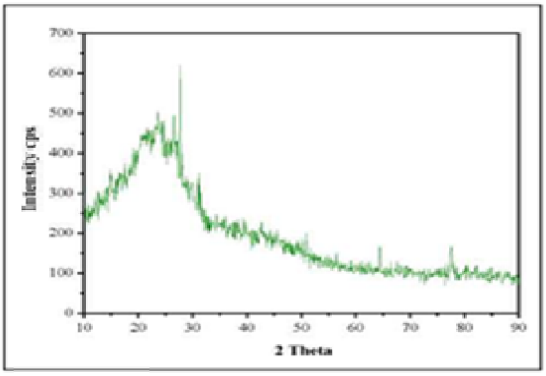

Fig. 7: XRD spectrum of BR22 loaded BNHC

I. ANALYSIS OF REAL WATER SAMPLE

BNHC played the role of active surface for the adsorption process in remediation of real water sample. The sampling for water was performed from the nearby Bhiwandi Taluka of Maharashtra. Since Bhiwandi is also famous as textile city locally, hence effluent from the river located near the city was collected and analysed for the bio-sorption study and results were published.[37]

II. CONCLUSION

The present biochar was analyzed for its physical and chemical properties and it was found suitable for the adsorption studies. Analysis of SEM images recorded pre and post adsorption signified substantial amount of adsorption of dye molecules. High carbonization content was reflected by high surface area and lower degree of fixed carbon. The extent of adsorption shown by the BNHC manifested the use of BNHC particles as potential adsorbent for the BR 22 dye molecules from the aqueous solution. However, these analytical methods were helpful in determining the adsorption on empirical level. The quantitative estimation of dye requires additional batch adsorption methods which we are working at present. To establish the relationship between adsorption parameters, study of thermodynamics and kinetics will be needed. In addition, the analysis of real water effluents which are supposed to be collected from water sources having Basic Red 22 dye as one of the contaminants. The real water effluent was studied and it was found that BNHC marginally lowered the BOD and COD of water. But analysis of real water sample has their own complexities such as simultaneous adsorption of other substances present as soluble or insoluble impurity in water sample.

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