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Valorization of Agricultural Waste into Value Added Products for the Removal of Industrially Important Dye towards Environmental Sustainability

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Abstract

This study explores preparation (thermally) and activation (chemically) activated carbon from *Areca spathe* (AS)for the removal of Amido black 10B (AB10B) dye by batch adsorption method. The surface characteristics of the ASC was analyzed using SEM and FTIR. Adsorption ability of ASC to remove AB10B has been investigated by varying various experimental parameters such as initial concentration, contact time, dose and pH of the dye solution. Acidic pH was more favorable for AB10B adsorption and the equilibrium adsorption data was well described by the Langmuir model with adsorption capacity of 123.0 mgg^{-1} for ASC and 148.08mgg^{-1} for CAC. The adsorption kinetics was found to be best represented by the pseudo-second-order kinetic model. As a result, the optimum ASC can serve as cost effective and efficient adsorbent for removing dyes from industrial wastewater.

***Key words*:** *Areca* spathe, AB10B dye, Batch adsorption, Isotherm, Kinetic studies

Dyeing industries are consumes huge amount of water for manufacturing followed by processing/finishing applications and discharged large volumes of wastewater into water bodies. Amido black 10B (AB10B), an anionic dye, belongs to the azo group of dyes, which consist of nitrogen-containing molecules. Amido black 10B has been widely used in the textile, printing, leather, paper, pharmaceutical and food industries. The presence of an azo group (N=N) on AB10B and its low biodegradability makes it a concern of environmental science. Azo dyes are usually released into the environment from such industrial effluents and the discharge of highly colored dye effluents can be very damaging to the receiving water bodies [1]. Most dyes can cause damage not only to aquatic life but also to human beings because they are toxic, mutagenic or carcinogenic. Hence, the removal of color synthetic organic dyestuff from waste effluents becomes environmentally important [2].

Many treatment methodologies have been adopted for the removal of dyes from wastewater including photocatalysis, sono-chemical degradation, ultrafiltration, cation exchange membranes, electrochemical degradation, adsorption/precipitation processes, integrated chemical–biological degradation, integrated iron (III) photo assisted-biological treatment, solar photo-Fenton and biological processes and adsorption on activated carbon. Among these methods, adsorption of dyes using biomass derived activated carbon has been proven to be a cost effective and simple method for water purification and waste water treatment applications. Nevertheless, commercially available activated carbons remain limited due to the high cost resulting from the use of non-renewable and expensive starting materials [3-5].

Biomass-derived activated carbons have attracted great attention due to their excellent physicochemical properties such as high specific area, large pore volume, well-defined micro porous structure, tunable surface chemistry and low cost. In recent years, special emphasis on the preparation of activated carbons from several agricultural by-products has been given due to the growing interest in low cost activated carbons for application concerning treatment of wastewater [6-8]. The advantage of using agricultural byproducts as raw materials for manufacturing activated carbon is that these raw materials are renewable and potentially less expensive to produce. To the best of our knowledge, there were no reports on the adsorption studies on the removal of dyes using *Areca spathe* carbon (ASC). The objective of this investigation is to explore the preparation and feasibility of using *Areca spathe* carbon activated carbon as adsorbents for the removal of AB10B dye, as an alternative to CAC which is most widely used in various textile-processing industries.

MATERIALS AND METHODS

Chemicals and reagents

Chemicals and reagents are analR grade used as received without any purification. All experiments were conducted at

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room temperature and Double distilled (DD) water was used throughout the studies.

Preparation and activation of adsorbent

The raw material *Areca spathe* was collected from locally, cut into small pieces, washed with water, dried and carbonized at the temperature range of 200–300 °C, in a muffle furnace (Neolab, AUS-101, India) in the absence of air. Then the carbonized materials were activated by digesting 200g of carbon with 600 ml of 1N nitric acid solution for 120 min., at 80°C. It was washed with boiling double distilled water several times to remove the acid (tested with pH Paper) and metal ions present in the adsorbent (tested with Eriochrome black –T indicator). This activated adsorbent was dried in an air oven at 120°C for about five hours. It was then stored in an airtight wide mouth reagent bottle and used for adsorption studies. The adsorbents employed in the present work were label as *Areca spathe* carbon (ASC) and commercial activated carbon (CAC).

Instrumental Studies

The as prepared ASC adsorbent was grounded and sieved in a mechanical sieve (Jayanth brand, India) to different consistent discrete particle sizes (90 micron). The Ph measurement was done by using Systronics digital pH meter (model: 335). The FT-IR spectra of the adsorbent materials (ACs), before and after adsorption of CH were recorded in KBr pellets by using a BIO-RAD WIN IR Spectrometer (Frequency range: 400–4000cm^{-1}). The scanning electron micro scope (SEM) photographs of the adsorbent materials were obtained using a JEOL JSM – 5300 SEM Instrument.

RESULTS AND DISCUSSION

Effect of initial AB10B dye concentration and adsorption isotherm studies

The effects of initial concentration of AB10B dye on the percentage removal by CAC and ASC are presented in the (Fig 1). Adsorption studies of AB10B dye on CAC and ASC was carried out at a fixed dose of adsorbent (4gL^{-1} CAC and ASC) at different initial concentrations of dye (range :100–200 ppm for CAC and 15–24 ppm for ASC) and contact time (30 min. for both ASC and CAC) and at solution pH (4.8), fixed particle size for CAC and ASC (90 micron) at a temperature 30±1°C.

The range of percentage removal of AB10B dye observed is 98.00-97.50 for CAC and 80.00-67.83 for ASC. It was observed that, the percentage removal of AB10B dye decreases exponentially with the increase in the initial concentration of AB10B dye. This may be due to reduction in immediate solute adsorption, owing to the lack of available active sites on the adsorbents surface compared to the relatively large number of active sites required for high initial concentration of AB10B dye [9-10].

The study of adsorption isotherm has been of importance and significance in the treatment of water and the waste water by the adsorption technique, as they provide an approximate estimation of the adsorption capacities of the adsorbents. The equilibrium data for the removal of AB10B dye by adsorption an CAC and ASC at 30±1^{0}C (Table 2) were used in fitting the Freundlich and Langmuir isotherms [11-13].

$$\text{Freundlich isotherm} = \log (x/m) = \log K + 1/n \log C_e$$

$$\text{Langmuir isotherm} = C_e/q_e = 1/Q_o b + C_e/Q_o$$

where, Freundlich constants K and 1/n are the measures of adsorption capacity and intensity of adsorption, respectively; q_e is the amount of dye adsorbed per unit mass of the adsorbent (in mgg^{-1}) at equilibrium i.e. $q_e = (x/m)$; $x = (c_i - c_e)$, C_i and C_e, initial and final equilibrium concentration of dye, respectively in ppm, m=mass of adsorbent, in gL^{-1}, Q_o is the monolayer adsorption capacity in (mgg^{-1}) and b is the Langmuir constant related to the energy of adsorption (in lmg^{-1}). The data obtained from the adsorption experiments by varying the initial concentration were fitted with Freundlich and Langmuir isotherms. Freundlich and Langmuir isotherms plots are represented in (Fig 2). These two isotherms plots are found to be linear (as evidenced from the values which are close to unity Table 1) indicating the applicability of these two adsorption isotherms for the removal of AB10B dye by adsorption on CAC and ASC and a formation of monolayer of AB10B dye on the surface of the adsorbents [14-15].

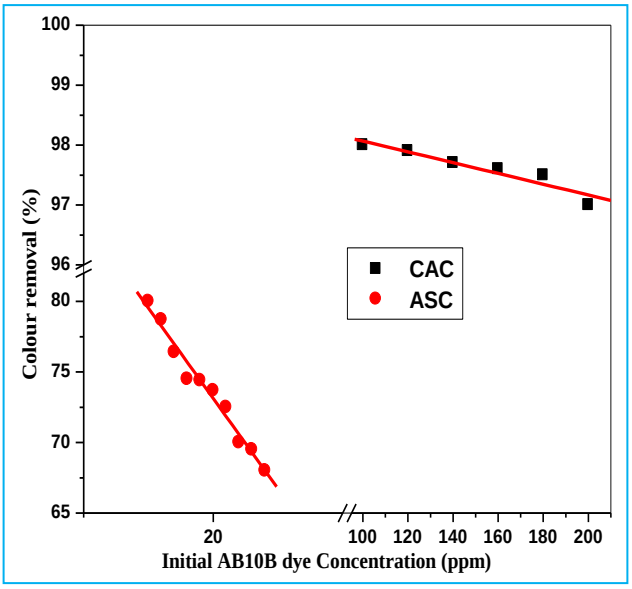

Fig 1 Effect of initial AB10B dye concentration on ASC and CAC

Table 1 Freundlich and Langmuir parameters for AB10B adsorption on CAC and ASC

Correlation analysis	CAC	ASC
Freundlich isotherm		
Correlation coefficient	0.998	0.983
Slope	0.736	0.293
Intercept	1.170	0.337
K	14.81	2.173
Δq (%)	0.004	0.012
Langmuir isotherm		
Correlation coefficient	0.985	0.994
Slope	0.007	0.199
Intercept	0.070	0.428
Q_o	148.08	123.0
B	0.097	0.464
R_L	0.068	0.108
Δq (%)	0.009	0.027

Further, the essential characteristics of Langmuir isotherm can be described by separation factor, R_L, which is defined by Weber and Chakravarthy – as described by others as given below:

$$R_L = 1/(1 + bC_i)$$

Where, C_i is the optimum initial concentration of amido black 10B dye (ppm) and b is the Langmuir constant (Lmg^{-1}).

The separation factor R_L indicates the shape of isotherm and nature of adsorption process as given in (Table 3). In the present study the R_L values were computed and given below:

R_L Value	Nature of process
$R_L > 1$	Unfavourable
$R_L = 1$	Linear
$0 < R_L < 1$	Favourable
$R_L = 0$	Irreversible

The results of correction analysis of adsorption data viz., correlation coefficient and the Freundlich and Langmuir constants and adsorption capacity (Q_o, (1/n) log K, b and R_L) are given in (Table 1). The results of statistical analysis of adsorption data reveal that both the Freundlich and Langmuir adsorption isotherms are applicable and the correlations are statistically significant. The values of R_L observed are found to be fraction, in the range of 0 to 1 (0.068 for CAC and 0.108 for ASC) indicating that the adsorption process is favorable.

In order to compare the validity of each model (isotherm) more efficiently, a normalised standard deviation, Δq(%) is calculated by following equation:

$$\Delta q\ (\%) = 100 \times \{(\sum [\ (\ q_t^{exp.} - q_t^{cal.}\ ) / q_t^{exp.}\ ]^2) / (n - 1)\}^{1/2}$$

Where, the superscripts, exp. and cal. are the experimental and calculated values of q_t *viz.*, the amount adsorbed at different time t and n is the number of observations. Based on the low values of Δq(%), it is concluded that, the adsorption of dye can be best described by the Freundlich adsorption isotherm [16].

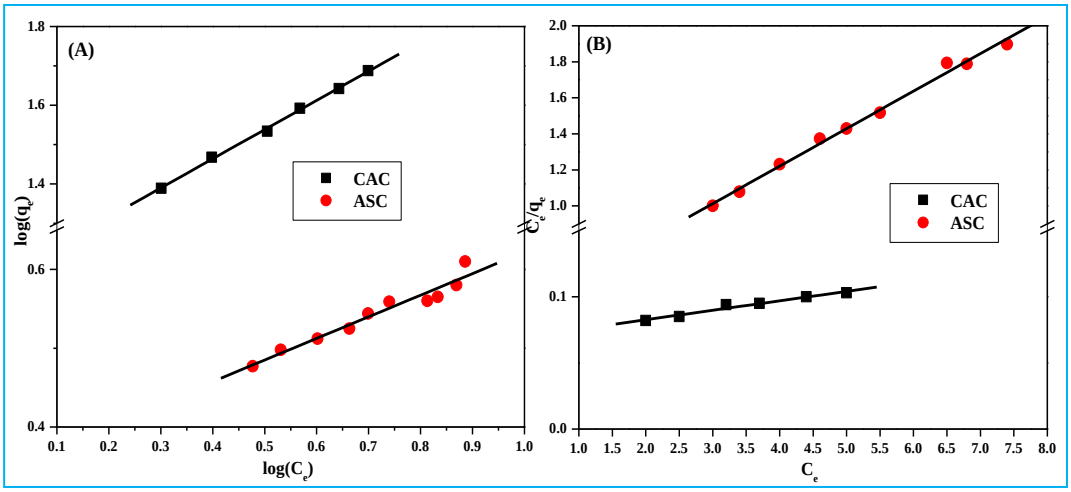

Fig 2 Freundlich (A) and Langmuir (B) isotherms plots of AB10B on *Areca* spathe carbon

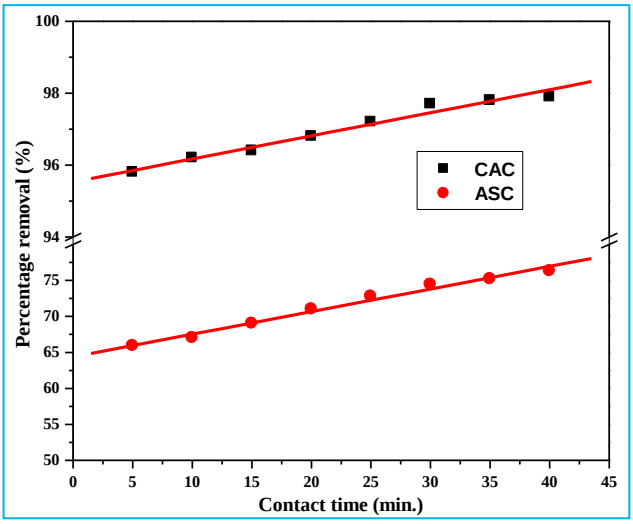

Fig 3 Effect of contact time on the extent removal of AB10B on ASC and CAC

Effect of contact time and kinetic studies

In the adsorption system, contact time plays a vital role, irrespective of the other experimental parameters affecting the adsorption kinetics. In order to study the kinetics of adsorption of AB10B dye, the batch type adsorption experiments were carried out by varying the contact time (range 5-40 min., for CAC and 5-30 min., for ASC) at constant optimum initial concentration of AB10B dye (140 ppm for CAC & 18 ppm for ASC) with constant dose of adsorbent (4gL^{-1} of CAC and ASC) at 30^{0}C (Figure 3). The values of percentage removal of AB10B dye and amount adsorbed increase exponentially with the increase in contact time. The percentage removal was found to rapid at the initial period of contact time and then become slow and stagnated with increase in contact time. The mechanism of removal of dye by adsorption on CAC and ASC from aqueous solution may be assumed to involve the following four steps:

i) Migration of dye from the bulk of the solution to the outer surface of the adsorbent
ii) Diffusion of dye through the boundary layer to the outer surface of the adsorbent
iii) Adsorption of the dye at on active site on the outer surface of the adsorbent
iv) Intra-particle diffusion of the dye from outer surface into the interior pores of the adsorbent particle.

The boundary layer resistance will be affected by the rate of adsorption and increase in contact time, which will reduce the resistance and increase the mobility of the adsorbate in the adsorption system. Since, the uptake of AB10B dye at the active sites of the adsorbent is a rapid process, the rate of adsorption is governed by either liquid phase mass transfer rate or intra-particle mass transfer rate [12].

Adsorption Kinetic Studies

In order to study the kinetics of adsorption of AB10B dye, the batch type adsorption experiments were carried out by varying contact time at optimum initial concentration of AB10B dye and fixed dose of adsorbent 4 g/L of CAC and ASC at 30ºC. The boundary layer resistance which affects the rate of adsorption. Increase in contact time will reduce the mobility of the adsorbate [AB10B] in the adsorption system. In order to find out the nature and order of kinetics of adsorption in the

present study, the applicability of various first order kinetic equations such as Natarajan-Khalaf, Lagergren and Bhattacharya –Venkobhachar equations was tested. The values of rate constant computed from the above rate equations are collected in Table 2. The linear kinetic plots (Figure 4) observed to be linear and the computed r (correlation coefficient) values which are very close to unity (range of r values : 0.936 - 0.984) as shown in Table 5 indicate the applicability of these first order kinetic equations and the first order nature of the adsorption process of dye. The minimum and maximum values of first order rate constants (k min^{-1}.) are noted for AB10B–CAC (8.82-8.93 min^{-1}) for AB10B–ASC (1.904-11.36 min^{-1}) system, respectively. This indicates that the removal of AB10B dye by CAC is maximum and ASC is minimum [17].

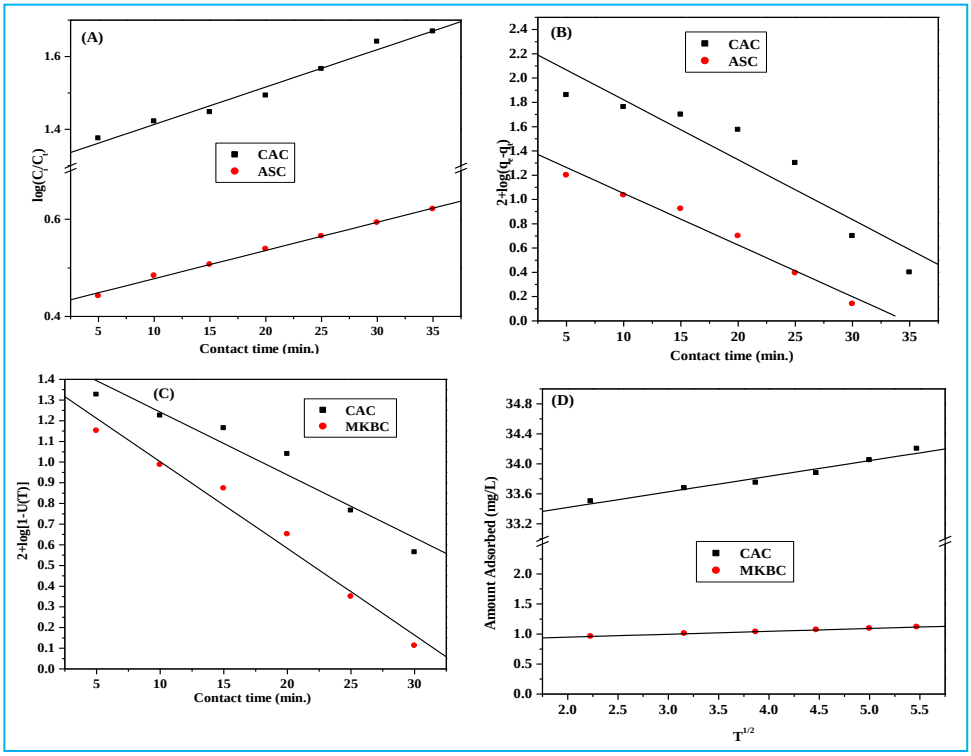

Fig 4 Kinetic plots of Natarajan-Khalaf (A); Lagergren (B); Bhattacharya-Venkobachar (C) and Intra-particle diffusion (D) of AB10B on ASC and CAC

Apart from the adsorption at the outer surface of the adsorbent, there also exists a possibility of intra- particle diffusion of adsorbent molecule from the outer surface into the internal pores of the adsorbent material. In diffusion-controlled adsorption process, the amount of solute adsorbed varies. Intra-particle diffusion model/equation is of the form.

Table 2 Kinetics parameters for AB10B adsorption on CAC and ASC

Kinetic Models	CAC	ASC
Natarajan Khalaf		
Correlation coefficient (r)	0.936	0.971
10^2 k (min^{-1}.)	8.820	1.904
Δq (%)	72.01	57.15
Lagergren Equation		
Correlation coefficient (r)	0.984	0.940
k (min^{-1}.)	8.930	11.36
Δq (%)	8.804	7.468
Bhattacharya and Venkobachar		
Correlation coefficient (r)	0.984	0.940
k (min^{-1}.)	8.930	11.36
Δq (%)	11.53	7.881
Intra – Particle diffusion model		
Correlation coefficient (r)	0.967	0.980
K_p (mgg^{-1} $min^{-0.5}$.)	0.037	0.183
Δq (%)	33.28	47.12
Intercept	0.890	33.10
Log (%R) Vs Log (Time)		
Correlation coefficient	0.993	0.958
Δq (%)	56.15	112.88

The plots of (x/m) *versus* $t^{½}$, resulted in linear curve (Fig 4D). It indicates that the intra particle diffusion is the significant rate limiting step in the adsorption process, under the given set of experimental conditions.

The minimum and maximum values of intra particle diffusion rate constant (K_p, $mgg^{-1}min^{-0.5}$) are noted for AB10B–CAC and for ASC as 0.037and 0.183, respectively.

$$(x/m) = K_p t^{1/2} + C$$

where, C=constant (intercept); K_p = intra- particle diffusion coefficient (mg g^{-1} $min^{-0.5}$)

The minimum and maximum values of intercept (C) of intra particle diffusion plots are noted for AB10B–ASC (33.10) and AB10B–CAC (0.890) systems. The correlations of the values of log (% removal) and log (time) for CAC is 0.993 and for ASC is 0.958. The corresponding plots (Figure 10) are found to be linear [18,19].

Effect of Initial pH of dye solution

The adsorption of AB10B dye on CAC and ASC at different initial pH values at constant optimum initial concentration of AB10B dye dose of adsorbent, contact time was also studied, in order to find out the variation in adsorption potential of CAC and ASC as a function of initial pH in adsorbing the dye. The process of adsorption of AB10B dye is found to be pH dependent. The variations in percentage removal of AB10B dye by adsorption on CAC and ASC with initial pH are shown in Figure 5 and this graph clearly revealed that the removal of AB10B dye is maximum in acidic medium up to 2 there after it decreases with increase in initial pH.

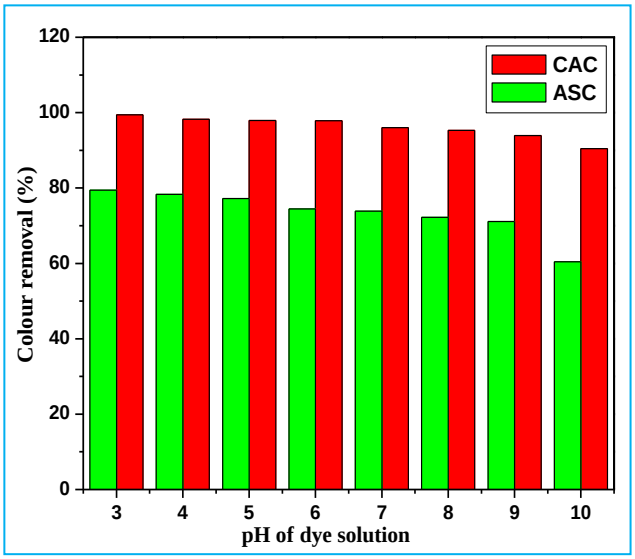

Fig 5 Effect of pH on the removal of AB10B dye on ASC and CAC

The pH affects the charge on the surface of the adsorbent, altering its capability to adsorb adsorbate molecules or ions/ species. The basic medium is highly favorable for the removal of AB10B dye. The variation in the removal of AB10B dye with pH can also be explained by considering the surface charge of the adsorbent materials which very much depends upon the zero point charge (ZPC) value of CAC and ASC. The uptake of AB10B dye could be quite similar under these conditions due to electrostatic repulsions with increasing pH, the negative charge density on the surface of adsorbent would increase, resulting in sudden enhancement in amido black 10B dye adsorption. The effect of pH variation on the extent of removal of dye would be exactly explained only, if mechanistic studies are carried out extensively [20].

FTIR and SEM studies

The (Fig 6) shows the FTIR and SEM images of the ASC before and after adsorption of AB10B dye. The FTIR spectrum of ASC (Figure 6) shows several peaks at 3430, 1713, 1362, 1231 and 698cm^{-1}, owing to various oxygen-containing functional groups, such as −OH, C=O, C−H, C−O, C-C respectively. The O−H stretching observed in both of the samples around 3430cm^{-1} was mainly attributed to chemisorbed water molecules and hydroxyl groups on carbon [21].

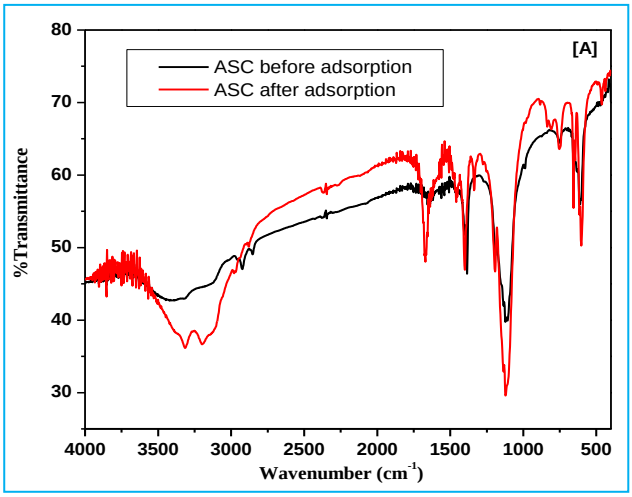

Fig 6 FTIR *Areca* spathe carbon before and after adsorption of AB10B dye

SEM pictures clearly revealed (Fig 7) that the surface texture and porosity of the carbon. Many large pores in a honeycomb shape were clearly found on the surface of the activated carbon (Fig 7A). The well-developed pores had led to the large surface area and porous structure of the activated carbon. The adsorbed dye molecules are either engulfed or surrounded by the porous adsorbent particle (Fig 7B) [22].

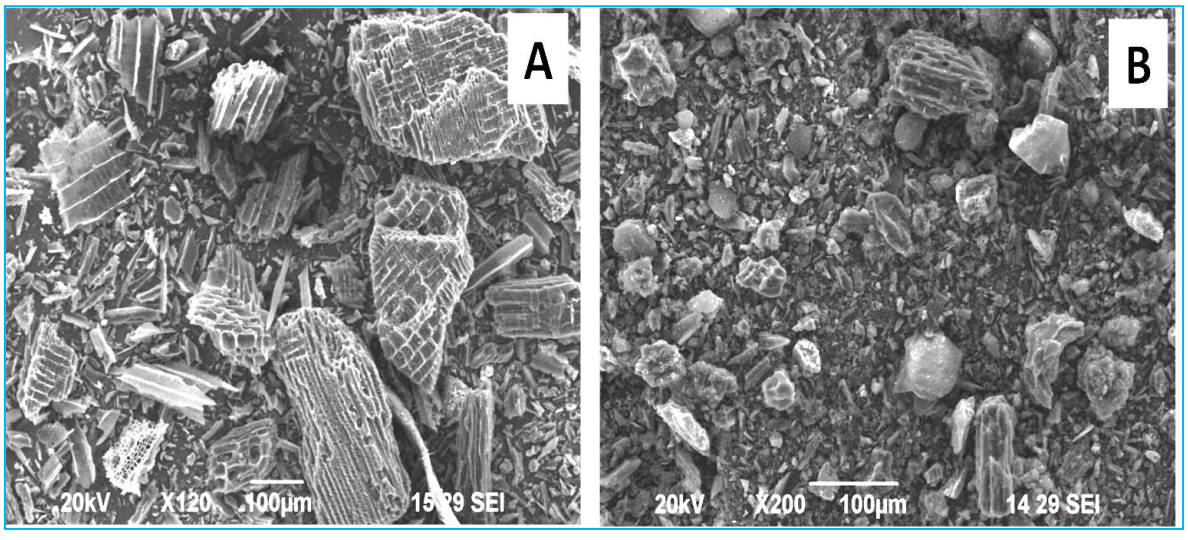

Fig 7 FTIR *Areca* spathe carbon before (A) and after (B) adsorption of AB10B dye

CONCLUSION

First time we have prepared and utilized the activated carbon from *Areca spathe* towards the removal of AB10B dye on CAC and ASC by batch adsorption technique. FTIR and SEM studies confirm that the ASC possessed excellent structural and morphological features. Adsorption studies indicated that the percentage removal of AB10B dye on these adsorbents {*Areca spathe* carbon (ASC) and commercial activated carbon (CAC)} is found to decrease with increase in initial concentration of AB10B dye and the amount of AB10B dye adsorbed per unit mass of adsorbent are found to increase exponentially with increase in contact time, dose and pH of dye solution. Langmuir and Freundlich Isotherm models were tested and found to be applicable. Monolayer adsorption of AB10B dye occurs on the surface of the adsorbents. Adsorption data are modeled with various first order kinetic equations like Natarajan-Khalaf, Lagergren and Bhattacharya and

Venkobachar equations. The intra particle diffusion model is found to be applicable. This indicates that the AB10Bdye adsorption on CAC and ASC is first order with the intra-particle diffusion as one of rate determining steps. The result of the study reveals that the adsorption process of AB10B dye on CAC and ASC could be employed treatment of effluents containing the AB10Bdye in neutral or slightly basic medium. However, from the economic point of view, ASC is concluded to be better cost–effective adsorbent, even though its adsorption capacity is slightly lesser than CAC.

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