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Adsorption Isotherm and Kinetic Study on the Removal of Cresol Red Dye using Indigenously Prepared Activated Carbons from *Artocarpus hirsutus* and *Annona muricata* Fruit Peel

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Abstract

Objectives: This study investigates the use of carbonized *Artocarpus hirsutus* and *Annona muricata* fruit peel biomass (AHPC and AMPC), as an eco-friendly and cost-effective adsorbents for Cresol Red (CR) dye removal, with a focus on environmental sustainability. **Methods:** The biomass derived carbons are prepared by pyrolysis followed by thermal activation method. Batch adsorption experiments were carried out as function of initial concentration, agitation contact time and pH and dye solution using AHPC, AMPC and CAC (Commercial Activated carbon). **Findings:** FTIR studies confirmed the adsorption of CR over carbons**.** The percentage removal of CR dye decreases with increase in initial concentration and pH and increases with increase in agitation time. The equilibrium adsorption data of CR dye on AHPC, AMPC and CAC were analyzed by Langmuir and Freundlich models. The results indicate that the Freundlich model provides the best correlation of the experimental data. Based on Langmuir isotherm, the maximum adsorption capacity for removing of CR dye was 263.0, 128.54 and 123.07mg/L for CAC, AHPC and AMPC and the order of adsorption capacity is CAC>AHPC>AMPC. The results showed that the experimental data were well-fitted with a pseudo-second-order model. **Novelty:** Valorization of waste fruit pees biomass is a cost effective alternate to commercially available activated carbon. This study presents a roadmap for conversion of fruit peel biomass waste into highly efficient porous carbon adsorbents.

Keywords: Waste Biomass; Cresol red dye; Activated carbon; Adsorption; Water treatment

1 Introduction

In recent years, rapid industrialization has caused a significant rise in the liberation of untreated industrial effluents, frequently containing hazardous compounds into the water bodies. Among the hazardous compounds, dyes are extensively employed in many industries, like textiles, paints, paper products, and cosmetics. The textile industry is a chief source of water pollution. Therefore, dyes are among the most abundant contaminants in wastewater. Among various dyes, Cresol red, also known as *o*-cresolsulfonephthalein, is an acidic dye commonly used as a pH indicator and a tracking dye in molecular biology techniques like electrophoresis. Hence, the presence of CR dye in high concentrations for long times can be caused carcinogenesis, chromosomal fractures, mutagenesis, teratogenicity, and respiratory toxicity in addition to other various side effects. Developing efficient and cost-effective techniques to remove CR dye is crucial to protect the environment and human health [(1)].

Several eco-friendly methods can be used to get rid of dyes in aqueous solutions, for example adsorption, chemical oxidation, coagulation, membrane filtration and others. However, the adsorption method has advantages over the abovementioned methods, such as simplicity in operation and implementation, high efficiency, cost-effectiveness, and recyclability [(2)].

Adsorption is one of the most versatile and widely used methods in dye wastewater treatment technologies due to its low-cost, better performance, easy operation, higher efficiencies and simple design. Efforts have been made to find more efficient, environment-friendly and low-cost adsorbents to remove dye pollutants [(3)]. Agricultural waste is considered to be one of the best alternatives because of its low-cost and high adsorption efficiency. However, because of the elevated cost of production, available agricultural solid wastes are being intensively investigated for the production of low cost effective ACs. The conversion of waste materials into ACs adds economic value helps reduce the cost of waste disposal and provides a potentially less expensive alternative to the existing commercial samples. Therefore, the conversion of these raw materials into valuable adsorbents for the elimination of pollutants from aqueous systems will be a promising alternative to reduce water pollution [(4)].

Artocarpus hirsutus and Annona muricata tress are an abundant genus of quick-growing hardy tree and its fruit peels are one of the important waste materials discarded from household and fruit shops/industry. The fruits peel of *Artocarpus hirsutus* and *Annona muricata* contains a wide range of natural polysaccharides and xanthone compounds. Nevertheless, it has a wide range of applications in the food manufacturing industries are deteriorated by the massive generation of peel. The disposal of the massive amount of *Artocarpus hirsutus* and *Annona muricata* peels producing a severe problem in the community as they slowly and progressively ferments and release foul odors. *Artocarpus hirsutus* and *Annona muricata* waste materials are low-cost, renewable, and feasibly accessible. So far, only few studies have focused on the conversion of *Artocarpus hirsutus* and *Annona muricata* peels into activated carbon (ACs) and their ability to remove dyes, and there is no precedent of any *Artocarpus hirsutus* and *aAnnona muricata* peel-based AC investigated as an adsorbent for cresol red dye in batch modes [(5)].

Therefore, the objective of this study was to evaluate the adsorption potential of activated carbon prepared from peels of *Artocarpus hirsutus* and *Annona muricata* towards CR dye. The equilibrium and kinetic data of adsorption studies were processed to understand the adsorption mechanism of CR dye onto the peel derived carbons.

2 Methodology

2.1 Chemicals and reagents

All the chemicals and reagents are analytical grade used without any further purification.

2.2 Preparation of *Artocarpus hirsutus* and *Annona muricata* peel carbon

The adsorbent raw materials of waste fruit peel from *Artocarpus hirsutus* and *Annona muricata* were collected locally and cut into small pieces followed by drying in three weeks. The *Artocarpus hirsutus* and *Annona muricata* fruit peels are physically activated by carbonization in a muffle furnace in the absence of air in stainless steel tube at 300°C for 90 minutes. The carbonized raw materials were then subjected to 4N HNO_3 acid activation as well as steam digestion. Finally, the *Artocarpus hirsutus* and *Annona muricata* fruit peel carbon (GMPC & DSPC) were thoroughly washed with distilled water until free from acid, then dried at 120°C in hot air oven for 24 hours, powdered well and sieved (90 micron size) by molecular sieves.

2.3 Batch adsorption studies

Adsorption experiments were carried out by adding a fixed amount of adsorbents into 250mL stopper flasks containing a definite volume (100 ml in each case) of different initial concentrations of cresol red dye solution at room temperature (301°C). The flasks were placed in a mechanical shaker and agitation was provided at 130 rpm for required time to ensure equilibrium was reached. At time t = 0 and equilibrium, the absorbance of cresol red dye solution was determined by the photo colorimeter. The dye removal percentage can be calculated as follows:

$$\text{Percentage removal} = 100(\text{Ci-Ce})/\text{Ci} \tag{1}$$

The amount of adsorption at equilibrium, qe (mg/g), was calculated by

$$\text{Amount adsorbed (qe)} = (\text{Ci-Ce})/\text{m} \tag{2}$$

Where, Ci and Ce were the initial and equilibrium concentration (in mgl-1 or ppm) of dye, respectively and m is the weight of AC (in gl^{-1}). The effect of dose of adsorbents on the amount of CR adsorbed was obtained by adding different amounts of adsorbents by keeping other parameters as constant. In order to study the kinetics/dynamics of adsorption of dyes, the adsorption experiments were conducted by varying the contact time at fixed optimum initial concentration of dyes with a fixed dose of adsorbent and solution pH. The dye concentrations were measured at different time intervals. The effect of pH was investigated at fixed initial concentration, dose and time by varying the pH. The flasks were then removed from the shaker, and the final concentration of dye in the solution was measured at maximum wavelengths of CR dye using UV/vis spectrophotometer.

3 Results and Discussion

3.1 Effect of Initial Concentration

The studies on the removal of dye by various low-cost adsorbents were carried out at different initial concentrations of dye (250-500ppm for CAC, 40-160 ppm for AMPC and AHPC) at fixed dose of adsorbent ($2gL^{-1}$ for CAC, $10gL^{-1}$ for AMPC and AHPC), particle size (90 mic) and agitation contact time (30 min.). The effect of initial concentration of CR dye and the percentage removal of dye by various adsorbents (CAC, AMPC and AHPC) are presented in (Figure 1). The increase in the initial concentration of dye decreases exponentially with the extent of percentage removal of the dye CR. When the initial concentration of CR dyes increases the percentage removal of CR dyes tent to decreases for all the above said adsorbnets. This indicates that there exists a reduction in immediate solute adsorption owing to the lack of available active sites required for the high initial concentration of dye. This may be due to maximum available active sites. In addition, decreasing the percentage removal also due to the fact that after the formation of mono ionic layer at lower concentration over the adsorption surface, any further formation of layer is highly hindered. Similar trend was observed in the himalayan chir pine biomass derived carbon for the removal of industrial dyes [6], [7]. At an optimum initial concentration of dye (230 ppm for CAC, 100ppm for AMPC and AHPC), the maximum percentage removal was found.

3.2 Adsorption Isotherms

Freundlich and Langmuir are the two most common and important adsorption isotherms experimental models used in order to evaluate batch studies. Accordingly, the data of this study have been analyzed in accordance with these both isotherm models [8]. The adsorption isotherms indicate how the adsorption molecules distribute between the liquid phase and solid phase when the adsorption process reaches an equilibrium state. The adsorption data were fitted with Freundlich adsorption isotherm.

$$\log(\text{x/m}) = \log \text{k} + 1/\text{n} \log \text{Ce} \tag{3}$$

A graph plotted between logCe against log x/m is shown in the Figure 2. The linearity of the graph indicates the applicability of Freundlich isotherm to the experimental data. The values of k and n are calculated from intercept and slope of the straight line. The Langmuir model assumes that uptake of adsorbate ions occurs on the homogeneous surface by monolayer adsorption. The Langmuir equation is expressed as follows

$$C_e/q_e = 1/Q_0 b + C_e/Q_o \tag{4}$$

The graph plotted between Ce /qe against Ce is shown in the Figure 2. The Langmuir constants Qo (mg g^{-1}) and b (L mg^{-1}), related to the adsorption capacity and energy of adsorption are obtained from slope and intercept of the straight line.

The correlation analysis of Freundlich and Langmuir isotherms are presented in Table 1. The applicability of the isotherm equation is compared by judging correlation coefficient, (r). The results conclude that Langmuir isotherm is best fitted compared to Freundlich, confirming the monolayer adsorption [6].

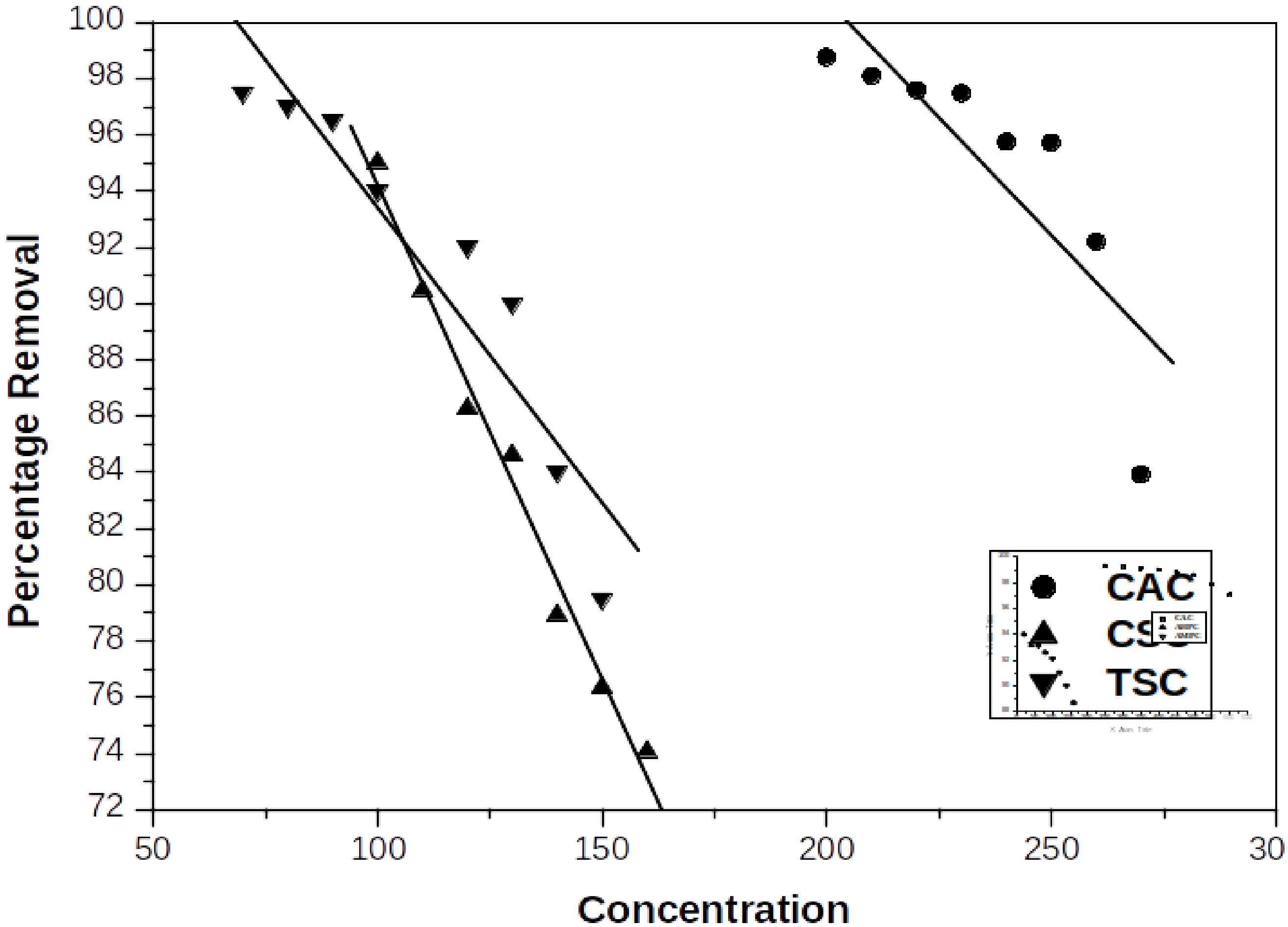

Fig 1. Effect on initial concentration on the removal of CR dye on CAC, AHPC and AMPC

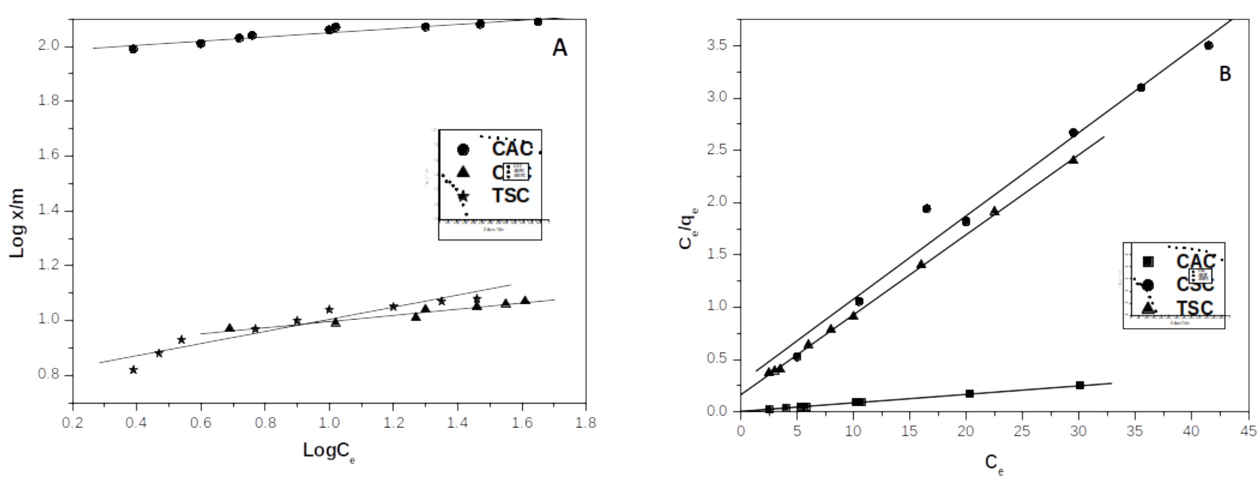

Fig 2. Figure 2 (A) Freundlich and (B) Langmuir adsorption isotherm on the removal of CR dye on CAC, AHPC and AMPC

Table 1. Results of various adsorption isotherms on the removal of CR dye on CAC, AHPC and AMPC

Isotherm models equation	CAC	AHPC	AMPC
Freundlich Isotherm			
Slope (1/n)	0.076	0.112	0.221
Intercept (Log k)	1.973	0.883	0.783
Correlation coefficient (r)	0.998	0.986	0.983
Langmuir Isotherm			
Intercept ($1/Q_o b$)	0.004	0.282	0.164
Correlation coefficient (r)	0.999	0.988	0.999
Q_o (mg g^{-1})	263	128.54	123.07
b (L mg^{-1})	1.775	0.282	0.466
R_L	0.002	0.021	0.014

Further the Langmuir constants are used to find the favourable or unfavourable adsorption by a dimensionless parameter RL.

$$RL = 1/(1 + bC0) \tag{5}$$

The value of RL indicates the isotherm to be unfavourable, linear or favourable.

RL value	Nature of adsorption process
RL=1	Linear
o<RL<1	Favourable
RL=0	Irreversible
RL>1	Unfavourable

In these cases, RL value is found to be less than 1. Hence the nature of adsorption is favourable. Another Langmuir constant Q_0 indicates the adsorption capacity. The monolayer adsorption capacity of adsorbents is in the order.[(9)],[(10)]

3.3 Effect of Contact Time

In the adsorption studies, contact time plays a vital role, irrespective of the other experimental parameters affecting the adsorption kinetics. The percentage removal of CR at 120min of contact time reveals that maximum removal efficiencies of the dye on CAC, AHPC and AMPC adsorbents are 98%, 95% and 89% respectively. The percentage removal of CR dye increases with increase in contact time, reaches a maximum value (Figure 3). The removal of CR dye by adsorption on adsorbents was found to be rapid at the initial period of contact time and then become slow and stagnant with increase in contact time. This may be due to (i) migration of dye from the bulk solution to the surface of the adsorbents, (ii) diffusion through the boundary layer to the surface of the adsorbents, (iii) adsorption at an active site and (iv) intra-particle diffusion into the interior of the adsorbent particle[(10)],[(11)].

3.4 Kinetic Modeling

To apprehend the adsorption mechanism in terms of adsorption rate and rate-limiting steps, the investigation of kinetics parameters is necessary. In order to study the kinetics and dynamics of adsorption of CR dye, adsorption experiments were carried out at different contact time (5 to 120 min) at constant optimum initial concentration of dye (230ppm for CAC, 100 ppm for AHPC and AMPC). The experimental data are fitted to the following kinetic models.

$$\text{Natarajan and Khalaf equation: Log (Ci/Ct)} = kt/2.303 \tag{6}$$

$$\text{Bhattacharya and Venkobachar equation : Log}[1 - U(t)] = -kt/2.303 \tag{7}$$

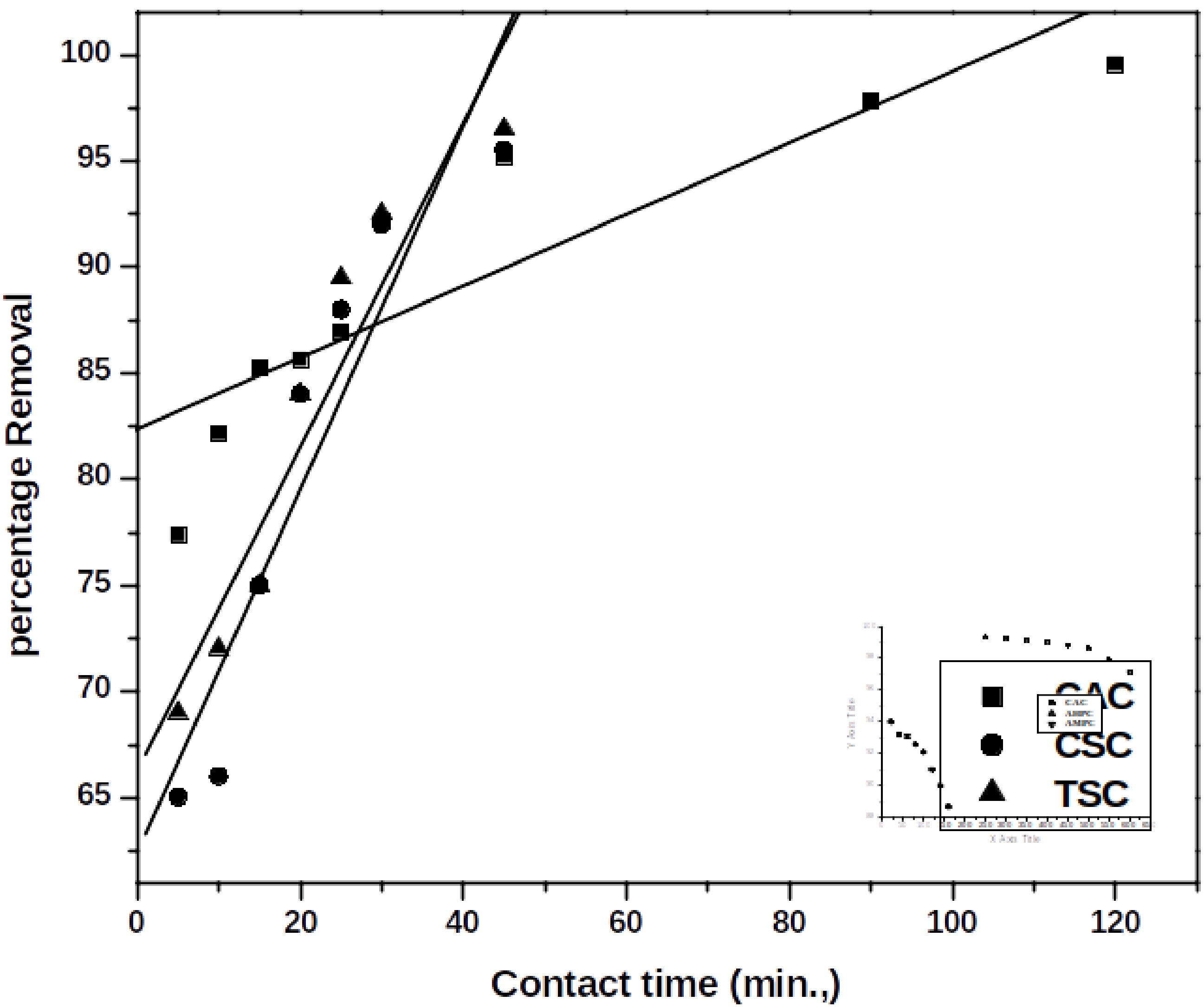

Fig 3. Effect of contact time on the removal of CR dye on CAC, AHPC and AMPC

$$\text{Where } U(t) = (Ci\text{-}Ct)/(Ci\text{-}Ce) \tag{8}$$

$$\text{Lagergren equation: Log } (qe\text{-}qt) = \log qe - kt/2.303$$

Figure 4 shows the plots of log Log (Ci/Ct) against time; Log [1 – U (t)] against time and Log (qe-qt) against time were found to be linear. The applicability of these equations indicates the first order nature of the adsorption kinetics of dye on CAC, AHPC and AMPC.

All the linear correlations are statistically significant as evidenced from the value of ‘r’ (Table 2). This indicates the applicability of these kinetic equation and first order of adsorption process. Among the three models, Lagergren first order kinetics yielded the highest ‘r’ value. This analysis suggests that Lagergren first order model was the most suitable equation to describe the adsorption kinetics of the dye CR on the adsorbents CAC, AHPC and AMPC[12], [13].

Table 2. Parameters for various kinetic equations on the removal of CR dye on CAC, AHPC and AMPC

Parameters	CAC	AHPC	AMPC
Natarajan and Khalaf kinetic equations			
Correlation coefficient (r)	0.981	0.977	0.968
k (min^{-1})	0.026	0.054	0.018
Bhattacharya and Venkobachar kinetic equations			
Correlation coefficient (r)	0.968	0.925	0.976
k (min^{-1})	0.05	0.099	0.145
Lagergren kinetic equations			
Correlation coefficient (r)	0.971	0.976	0.974
k (min^{-1})	0.05	0.1	0.116

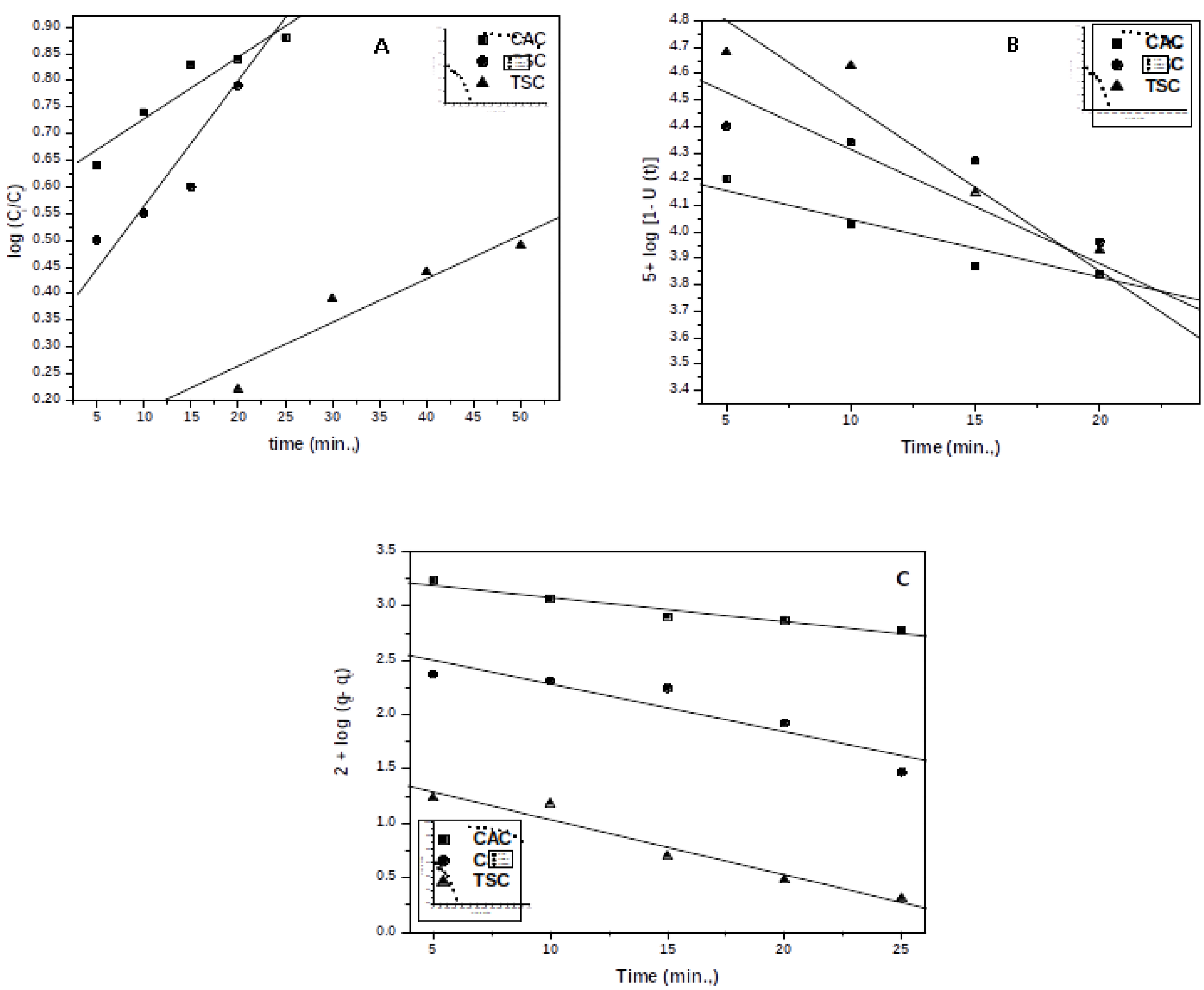

Fig 4. Natarajan-Khalaf (A), Bhattacharya-Vencobachar (B) and Lagergren (C) kinetic plot for the removal of CR dye on ACs

3.5 Effect of Initial pH of Dye Solution

The adsorption of CR on CAC, AHPC and AMPC (pH: 3-9) with constant optimum initial concentration (230, 100ppm), contact time (30 min) and optimum dose of -1 for CAC and 10gL-1 for AHPC and AMPC) were also studied (Figure 5) in order to find out the variation on adsorption potential of these adsorbents as a function of pH. The adsorptions of CR on CAC and AHPC, AMPC adsorbents are found to be highly pH dependent. The data reveals that the removal of CR by is maximum at pH = 3, after that its tent to decreases. The pH affects the change on the surface of the adsorbents altering its capacity to absorb materials. The zero point charge (pHzpc) of CAC is 7.25 and the zero point charge of AHPC is 5.8 and 5.6 for AMPC. In acidic medium (pH > 6.2) the surface of CAC and AHPC are positively charged, whereas it is negatively charged under alkaline medium (pH < 6.2). The adsorbent surface is negatively charged (pHzpc > pH) resulting in the increased adsorption of the cationic dyes and reverse is observed when pHzpc < pH. CR is anionic dye; their electrostatic attraction to the adsorbents surface is favorable in acidic solution and forbidden in basic media due to the columbic repulsion between the positively charged surface of CAC and AHPC and the negatively charged dye molecules. The percentage removal of dye linearly decreases with the increase in initial pH for adsorption of CR dye for all adsorbents, which indicates that the acidic pH = 3 is found to be more suitable for the removal of CR dye[14].

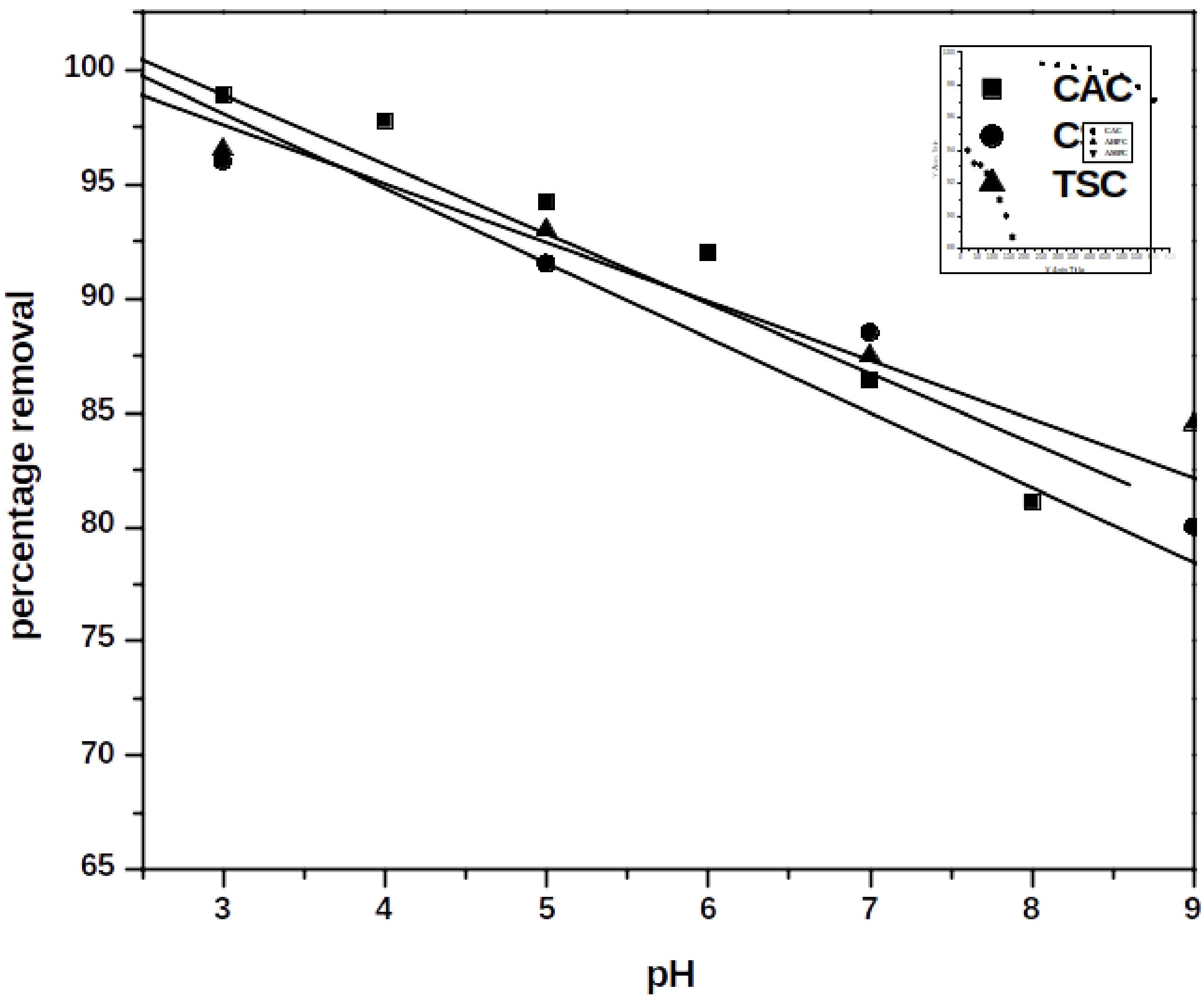

Fig 5. Effect of initial pH of dye solution on the removal of CR dye on CAC, AHPC and AMPC

3.6 FT-IR Analysis

The FT-IR spectral analysis of activated carbon viz., CAC, AHPC and AMPC before and after adsorption of Cresol Red dye was shown in Figure 6. The FT-IR spectrum of the activated carbon showed that the most prominent peaks in the spectrum originate from OH vibrations, CH_2 and CH_3 asymmetric and symmetric stretching vibrations. The peaks at (1155–1033cm^{-1}) region related to lignin [13]-[15]. Therefore, it is possible that cellulose, hemicelluloses as well as lignin, having many OH groups in their structure, make up most of the absorbing layer. Two strong peaks observed in the range between 2963cm^{-1} and 2853cm^{-1} are assigned to asymmetric C–H bands and symmetric C–H bands, respectively, present in methyl and methylene groups. Stretching vibration band around 1600cm^{-1} is assigned to carbonyl C=O group present in aldehyde, ester, ketone and acetyl derivatives.

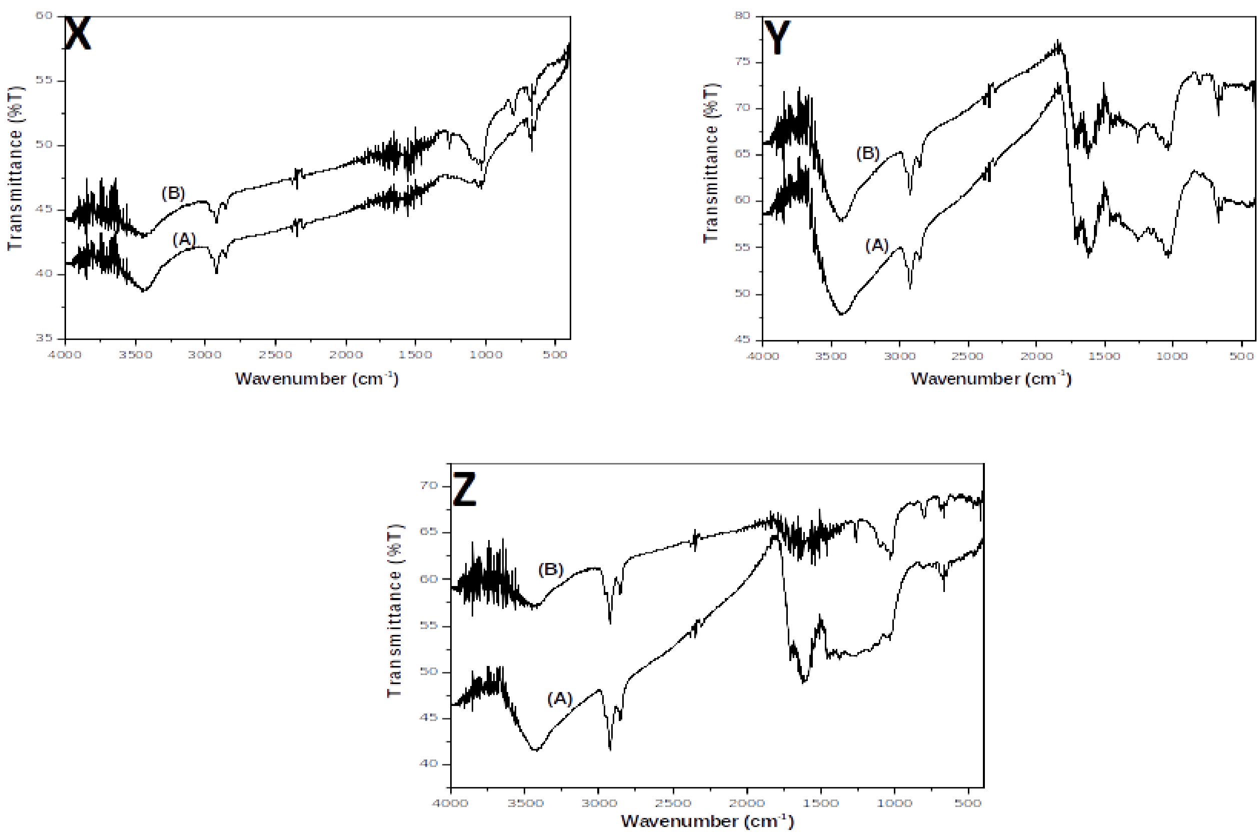

Fig 6. FTIR spectral data of adsorbents (X = CAC; Y = AHPC & Z = AMPC) before (A) and after (B) adsorption of CR dye

Moreover, the peak intensity of the peaks after adsorption is get reduced for all adsorbents after adsorption of CR dye, which indicates that, the adsorption dye on the adsorbents [15]-[16].

4 Conclusion

Waste biomass based fruit peel of *Artocarpus hirsutus* and *Annona muricata* were utlised for the preparation of activated carbon and their adsorption ability towards CR dye was investigated by batch studies, further the results are compared with CAC. FTIR studies confirmed that the adsorption of CR dye over AMPC and AHPC pore surface. The percentage of colour removal increases with decreasing the concentration, and increase with increase in agitation contact time on CAC, AMPC and AHPC increases. The anionic dye CR shows maximum percentage removal at lower pH (acidic medium). The linearity of curve and correlation coefficient value of isotherms such as Freundlich and Langmuir shows that the system covered monolayer adsorption. Langmuir model provides the best correlation and the adsorption capacities of the adsorbents for CR dye were found to be 263.0, 128.54 and 123.07mg/L for CAC, AHPC and AMPC. Adsorption data were modeled using the pseudo-first-order, pseudo-second-order and intra-particle diffusion kinetics equations. It was shown that pseudo-second-order kinetic equation could best describe the adsorption kinetics for both AMPC, AHPC and CAC. From the above results we conclude that the adsorbents such as AHPC and AMPC proved to be effective one for the removal of the dye.

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