



Preparation and Characterization of Activated Carbon from Lapsi (*Choerospondias axillaris*) Seed Stone by Chemical Activation with Phosphoric acid

Shrestha Rajeshwar M.^{1*}, Yadav Amar P.², Pokharel Bhadra P.¹ and Pradhananga Raja Ram ²

¹Department of Engineering Science and Humanities, Institute of Engineering, Tribhuvan University, NEPAL

²Central Department of Chemistry, Tribhuvan University, NEPAL

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Abstract

Activated carbon was prepared from Lapsi seed stone by chemical activation with phosphoric acid at 400 °C. pH of point of zero charge (pHpzc), iodine number, proximate analysis and concentration of surface oxygen functional groups of activated carbon was determined by Boehm titration. The adsorption of methylene blue by thus prepared activated carbon was analyzed by the Langmuir and Freundlich adsorption isotherms. The data fitted well to the Langmuir isotherm with monolayer adsorption capacity 277 mg/g. Thermogravimetric analysis and proximate analysis of Lapsi seed stone was also carried out. The analysis showed that the activated carbon derived from Lapsi seed stone activated with phosphoric acid is comparable with commercial activated carbon and can be used as a potential adsorbent.

Keywords: Chemical activation, lapsi seed stone, methylene blue, iodine number, adsorption isotherm.

Introduction

Activated carbon is an extremely versatile material with high porosity and surface area. It has become one of the technically important and most widely used adsorbents because of high adsorptive capacity. Nevertheless, its application fields are restricted due to high cost. The use of low cost wastes and agricultural by-products to produce activated carbon has been shown to provide economic solution. Many precursors have been used with success for the production of activated carbons including apricot stones¹ guava seeds² black stone cherries³, peach stones^{4,5}, orange peel⁶, Peanut shell⁷ which are the some examples of low-cost accessible raw materials for the production of Activated carbons. Besides offering economic advantages over mineral or bituminous coal, these wastes can be processed at temperature below 600°C while ordinary coal yields its best results at over 800°C⁸.

The preparation of activated carbons can be carried out by one of the following two processes; i. Physical activation: In this process carbonization and activation are done separately. Carbonization process eliminates non carbon elements. Activation or oxidation is carried out by exposing the carbonized material to oxidizing gases like carbon dioxide, or steam at high temperature. ii. Chemical activation: In this process carbonization and activation is done in a single step. The raw material is impregnated with certain chemicals at specific ratios before carbonization. The chemical is typically an acid, strong base, or a salt (phosphoric acid, potassium hydroxides, sodium hydroxide, zinc chloride, etc.). Then, the impregnated raw material is carbonized at lower temperatures

(comparatively lower than physical carbonization). It is believed that the carbonization and activation occurs simultaneously during chemical activation. Chemical activation is preferred over physical activation because it requires lower temperatures and shorter time for activating the material⁹. Phosphoric acid produces better modification than other acids to the botanic structure by penetrating, swelling and breaking the bonds of lignocelluloses materials¹⁰.

Not much information is available regarding the preparation of activated carbon from Lapsi Seed stone using phosphoric acid as an activating agent. Activated carbon was prepared from Lapsi seed stone by chemical activation with zinc chloride for the adsorption of arsenic from water^{11, 12}. No report is available in the literature about the preparation of activated carbon from Lapsi seed stone using phosphoric acid as an activating agent. This lack in existing literature is a motivation for the present study.

Lapsi seed stone is the waste product of Lapsi fruits. (Lapsi) *Choerospondias axillaris*) belongs to the family *Anacardiaceae*. It is a large, fruit bearing deciduous tree native to the hills of Nepal (865-1900m). Lapsi fruit is consumed fresh, pickled or processed into a variety of sweet and sour, tasty food products locally called "Mada" and "candy". It is a rich source of vitamin C. Seed stones are used as fuel in brick kilns in the factories and the trunk of the tree is used as fuel wood and timber^{13,14}.

The aim of the work is to produce activated carbon from Lapsi seed stone by chemical activation with phosphoric acid and compare with that of commercial activated carbon. The

activation with phosphoric acid was selected because of its wide use as an activating agent and because it yields the best adsorption results among acid activating agents.

Material and Methods

Precursor: The precursor used for the preparation of activated carbon in this study was seed stone of Lapsi fruits. The Lapsi fruits were collected from Kalimati market, Kalimati, Kathmandu. The fruits were stripped for the pulp by boiling to expose rigid centers or stones. The seed stones were washed with tap water and then distilled water to remove impurities, dried at 110°C for 12 hours and crushed with mortar and electric grinder. The crushed particles were then sieved to obtain the fraction 0.3 to 0.5mm.

Thermal Behavior of Lapsi seed stone particles: Thermal behavior of Lapsi Seed Stone was observed by TGA analysis in N₂ gas environment to find the appropriate temperature for carbonization for the production of Activated carbons.

Impregnation and Carbonization: Lapsi seed stone particles were mixed with 50% H₃PO₄ in 1:1 weight ratio and kept in an oven at 80°C for overnight. The mixture was then carbonized in a horizontal tubular furnace under a flow (75 ml/min) of N₂ for 4 hours. After well cooled, the carbon prepared was washed with solution of NaHCO₃ and then with distilled until the pH of the washing was between 6 and 7. The carbon was sieved to get the particles of size 106 μm and dried in an oven maintained at a temperature of 110°C for 24 hours. The carbon was used for further study.

Determination of pH: In order to determine pH of PALSC (Phosphoric acid Activated Lapsi Seed Stone Carbon) and CAC (Commercial Activated Carbon), the standard test method ASTM-D3838 -80 was used¹⁵. 1.0 g of each activated carbon PALSC and CAC was transferred into 100 ml distilled water taken in a beaker and kept in a magnetic stirrer for one hour. pH was then measured by pH meter. The samples were tested in duplicates.

Iodine number: The adsorption of aqueous iodine is considered a simple and quick test for evaluating the surface area of activated carbons associated with pores larger than 1 nm. The iodine number, defined as the amount of iodine adsorbed per gram of activated carbon at an equilibrium concentration was measured according to the procedure established by the American Society for Testing and Materials (ASTM 2006)¹⁶. 0.1 g of dry activated carbon and commercial activated carbon was separately taken in dried 100 ml conical flasks. The samples were run in duplicates and added 5 ml of 5% HCl. The flasks were swirled until the carbon was wetted. 10ml of 0.1N iodine solution was added to each flask and was shaken properly for 4 minutes. 10 ml filtrate was titrated against standard (0.1N) hypo solution using starch as an indicator. The concentration of iodine adsorbed by activated carbon was calculated as amount of iodine adsorbed in milligrams.

Iodine number = C * Conversion factor

The conversion factor can be calculated as follows:

$$\frac{\text{Mol. wt of Iodine} * \text{Normality of Iodine} * 10}{\text{Wt. of activated carbon} * \text{Blank reading}}$$

C= Blank reading – volume of hypo consumed after the adsorption of Activated carbon.

Iodine Number is accepted as the most fundamental parameter used to characterize activated carbon performance. It gives the measure of activity level (higher number indicates higher degree of activation).

Determination of Moisture content: For determination of moisture content ASTM –D1762 (1990) was used¹⁷. 1.0 g each of dried PALSC and CAC was separately taken in dried and weighed porcelain crucibles. The samples were run in duplicates. The crucibles were kept in an oven maintained at a temperature of 105 °C for 4 hours. The crucibles were placed in a desiccator for 1 hour and weighed. The drying, cooling and weighing were repeated to get constant weight. The percentage of moisture content was calculated as follows:

$$\text{Moisture}(\%) = \left[\frac{(A - B)}{A} \right] * 100$$

Where: A = weight of dried sample, B = weight of the sample after drying at 105°C

Determination of Ash content: Ash content was determined by the same method as done in moisture content. 1 g of each of LSPAC and CAC was taken dried and weighed crucibles. The samples were run in duplicates as before and placed in a muffle furnace at 750 °C for 6 hours. The crucibles were cooled in a desiccator for 1 hour and weighed. Ash content in percentage was calculated as follows:

$$\text{Ash}(\%) = \frac{D}{B} * 100$$

Where, D = weight of residue left in gram, B = weight of dried sample in gram.

Determination of pH of point of zero charge (pHpzc): pHpzc of an adsorbent is important because it indicates the net surface charge of the carbon in solution. The pHpzc is the point where the curve of pH_{final} vs pH_{initial} intersects the line pH_{initial} = pH_{final}. In order to determine the pH of point of zero charge 0.15 g of PALSC and CAC was taken to eleven 100 ml conical flasks containing 50 ml of 0.01M NaCl, the pH of which was adjusted from 2 to 10 by addition of 0.01M HCl or NaOH for each Activated carbon. The conical flasks were sealed and placed in a shaker for 48 hours. The content of the flasks was filtered and pH was then measured by pH meter.

Determination of surface functional groups: The presence of surface functional groups in the activated carbons was quantified by Boehm titration method. 1.0 g of activated carbons was taken in contact each with 25 ml of 0.05M NaOH, and

Na_2CO_3 for acidic functional groups and 0.05 M HCl for basic groups for 24 hours.

The solutions were filtered and 5 ml of the filtrate was titrated against 0.05 M HCl for acidic functional groups and against 0.05M NaOH for basic groups. The method based on that the weakest base NaHCO_3 neutralizes only the strongest acidic carbon surface functionalities which are carboxyl groups, while Na_2CO_3 neutralizes carboxylic and lactic groups. The strongest base NaOH neutralizes carboxylic, lactic and phenolic groups. On the basis of amount of acid and bases consumed the different kinds of functional groups can be quantitatively calculated. The difference between the groups titrated with Na_2CO_3 was assumed to be lactones and those titrated against Na_2CO_3 was assumed to be phenol. Basic functional groups were determined by titration against 0.0M HCl.

Methylene blue adsorption: Methylene blue adsorption tests were conducted by mixing 0.1 g of each, prepared activated carbon and commercial activated carbon separately with 100 ml of 100, 150, 200 and 250 mgL^{-1} methylene blue solution. After shaking for 24 hours, the suspensions were filtered and methylene blue residual concentration was measured at 660 nm using an UV /Vis spectrophotometer (CECIL- CE-100) .Linear Beer-Lambert relationship was used for the determination of concentration.

Results and Discussion

Chemical activation: Activated carbon was prepared by chemical activation impregnated with phosphoric acid at 400°C . The activation with phosphoric acid was selected because of its wide use as an activating agent and it yields the best adsorption results among acid activating agents. An important advantage of chemical activation is that the process normally takes place at lower temperature and for a shorter time than those used in physical activation. In addition, very high surface area activated carbons can be obtained. Moreover, the yield of carbon in chemical activation is usually higher than those in physical activation because the chemical agents used are substances with dehydrogenation properties that inhibit formation of tar and reduce the production of other volatile products. On calcinations, the impregnated chemicals dehydrate the raw materials, which results in changing and aromatization of the carbon skeleton by the creation of a porous structure and surface area.

Thermogravimetric analysis: Thermal behavior of Lapsi seed stone was observed by TGA in N_2 gas environment to study the weight loss during chemical activation (figure 1). TGA curves showed that the weight of Lapsi Seed Stone decreased steeply from 200°C to about 400°C and from 400°C the weight decreases slowly to about 700°C . No more reduction in weight is above 700°C . The major weight loss was observed between 200°C and 400°C during TGA analysis which may due to the

decomposition of polymeric network of cellulose and lignin, loss of water, carbondioxide and wide range of organic molecules¹⁸. The appropriate temperature for the carbonization of Lapsi seed stone is found to be 400°C .

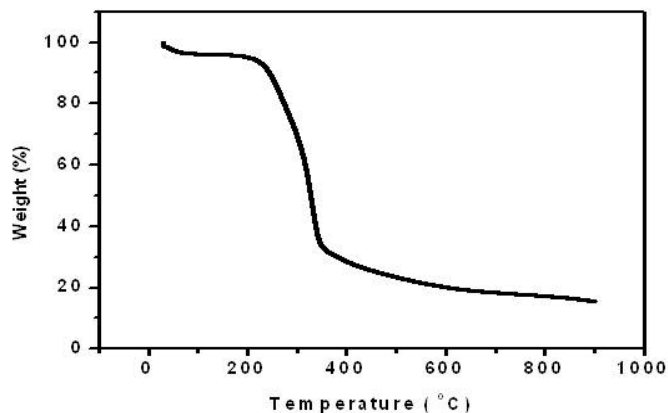


Figure-1
Thermal behavior of Lapsi seed stone by TGA

Surface characterization of activated carbon: The low ash and moisture content presented in table- 1 exhibited that the raw material is good for the preparation of activated carbons. Ash content reduces adsorptive power of activated carbons and the efficiency of reactivation. A small increase in ash content causes a decrease in adsorptive properties of activated carbon. So lower the ash content better the activated carbon for use in adsorption process¹⁹. The activated carbon prepared from the waste materials of Lapsi fruits can be used as a potential adsorbent in place of commercial activated carbon.

The pH of PALSC and CAC was found to be 6.3 and 6.8 respectively. A carbon of pH 6-8 is acceptable for most application such as for sugar decolorization, water treatment etc²⁰. pH is affected not only by the reaction of carbon dioxide but also by organic and inorganic solutes present in water. Any alteration in water pH is accomplished by the change in other physicochemical parameters²¹. The significance of pH_{pzc} of activated carbon surface is that it will have a positive charge at solution pH less than their pH_{pzc} and thus be a surface on which anion may be adsorbed. On the other hand, if the solution pH has greater than that of pH_{pzc} of activated carbons, the surface of the carbons will bear negatively charged and cations may be adsorbed on the surface. The pH_{pzc} of PALSC and CAC were found to be 6.1 and 6.6 respectively.

In order to gain further knowledge of the porous structure of activated carbon, iodine adsorption from liquid phase was adopted by other researchers in the characterization of activated carbons. The adsorption of aqueous iodine is considered a simple and quick test for evaluating the surface area of activated carbons associated with pores larger than 1 nm^2 . Iodine number is an indication of the adsorption capacity in microspores; therefore it is often employed to examine the adsorption

capacity of the activated carbons by researchers. Table 1 exhibits the iodine number of PALSC and CAC. A higher value of iodine number for CAC in comparison to PALSC is due to greater surface area and available microspores for adsorption of iodine molecule on the surface. The iodine number of PALSC is lower in some extent to that of CAC as shown in figure 2. CAC is actually expensive for the adsorption process. Adsorption is a natural process by which molecules of a dissolved compound collect on and adhere to the surface of an adsorbent solid. Adsorption occurs when the attractive forces at the surface of carbon surface overcome the attractive forces of the liquid²³. The activated carbon prepared from Lapsi seed stone, the inexpensive readily available material, can be used as an adsorbent.

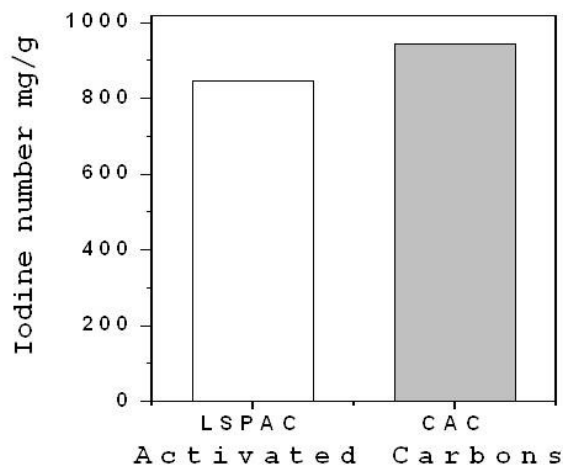


Figure-2
Iodine adsorption PALSC and CAC

The quantification of surface functional groups of activated carbons was determined by Boehm titration. The method based on that the weakest base NaHCO_3 neutralizes only the strongest acidic functional groups which are carboxyl groups, while Na_2CO_3 neutralizes carboxylic and lactonic groups²⁴⁻²⁶. The strongest base NaOH neutralizes carboxylic, lactonic and phenolic groups. On the basis of amount of acid and bases consumed the different kinds of functional groups can be quantitatively calculated. The presence of basic groups in activated carbons was determined by titration with HCl . For the determination of amounts of surface functional groups, Boehm titration has been widely used by many researchers.

Adsorption isotherm: The adsorption isotherm gives information how the adsorption molecules distribute between the liquid phase and the solid phase when the adsorption process attains an equilibrium state. The relationship between the amount of a substance adsorbed at constant temperature and its concentration in the equilibrium solution is known as adsorption isotherm²⁷. Langmuir and Freundlich adsorption isotherm models are employed in this study to describe the experimental adsorption isotherm²⁸. The applicability of the isotherm equations was compared by judging the coefficients of

determination R^2 . Langmuir adsorption is based on the fact that maximum adsorption corresponds to a saturated monolayer of solute molecules on the adsorbent surface. The linear form of the Langmuir equation can be represented by

$$\frac{C_e}{q_e} = \frac{1}{q_m b} + \frac{1}{q_e} C_e$$

Where q_e is the amount of methylene blue adsorbed (mg g^{-1}) and C_e is the equilibrium concentration of methylene blue in the bulk solution (mg L^{-1}) while q_m is the monolayer adsorption capacity (mg g^{-1}) and b is the Langmuir constant^{29,30}. Langmuir constant and adsorption capacity are determined from the slope and intercept of the plot C_e/q_e versus C_e and are presented in table 3.

Table-1
Physicochemical analysis of phosphoric acid activated Lapsi seed stone carbon (PALSC) and commercial activated carbon (CAC)

Property	Phosphoric acid activated Lapsi seed stone carbon	Commercial activated carbon
pH	6.3	6.8
Moisture (%)	0.27	0.23
Ash (%)	2.94	3.12
Iodine number (mg/g)	845	942
Adsorption capacity of Methylene blue (mg/g)	277.0	302.1
Carboxylic (meq g^{-1})	0.39	0.27
Lactonic (meq g^{-1})	0.62	0.53
Phenolic (meq g^{-1})	0.32	0.29
Basic site (meq g^{-1})	0.37	0.45
pHpzc	6.1	6.6

Table-2
Proximate analysis of Lapsi seed stone

S.N.	Property	Value (%)
1	Ash content	2.87
2	Moisture content	0.28

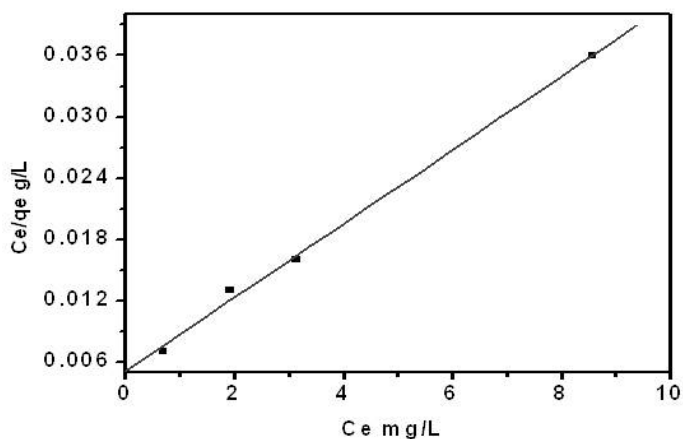


Figure-3
Langmuir isotherm for adsorption of MB onto CAC

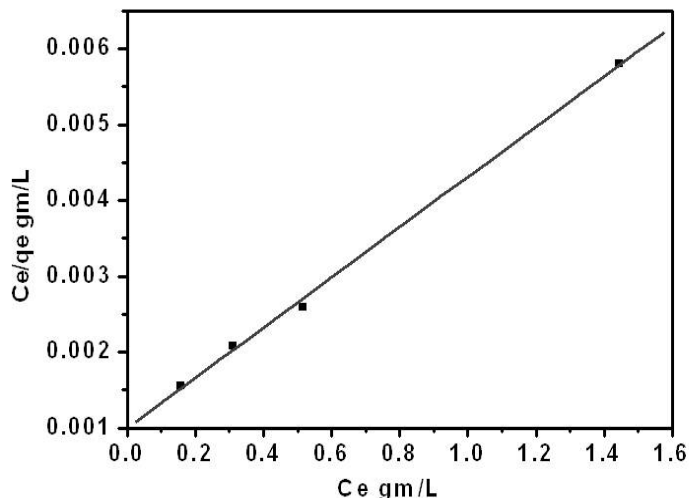


Figure -4

Langmuir isotherm for adsorption of MB onto PALSC

Freundlich isotherm is an empirical equation describing the heterogeneous adsorption and assumes that different sites with several adsorption energies are involved. The linear form of the Freundlich equation is shown below.

$$\log q_e = \log K + \frac{1}{n} \log C_e$$

Where K and n are Freundlich constants related to adsorption capacity and adsorption intensity respectively. From the slope and intercept of straight portion of the linear plot obtained by plotting log q_e versus log C_e, the values of Freundlich parameters are calculated.

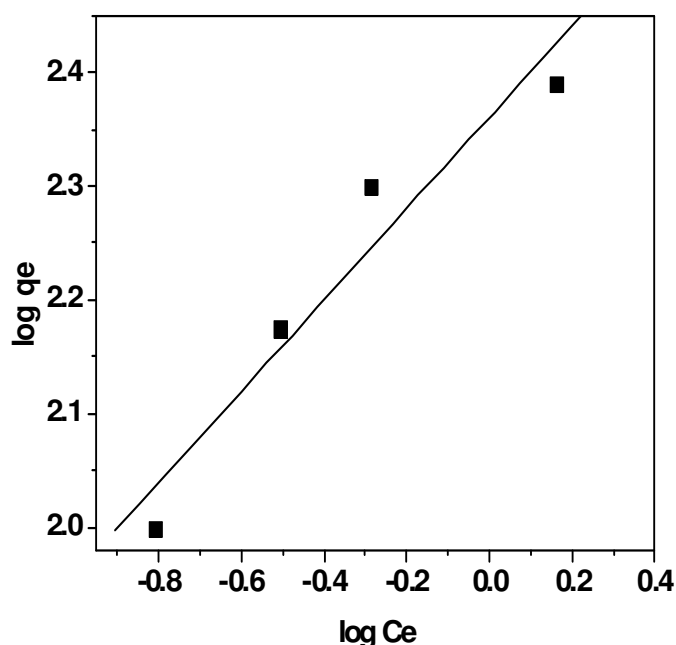


Figure-5

Freundlich isotherm for adsorption of MB onto CAC

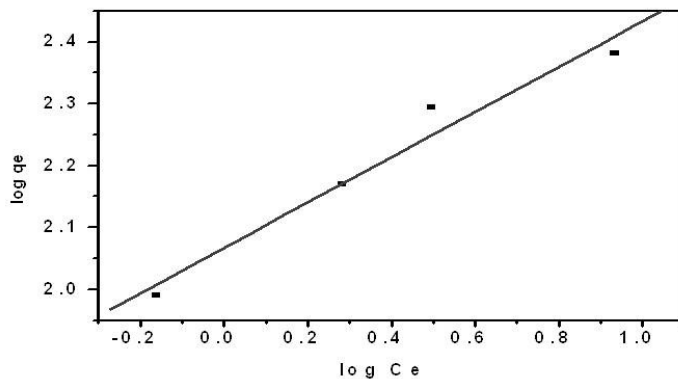


Figure -6

Freundlich isotherm for adsorption of MB onto PALSC

Langmuir and Freundlich constants are given in table 3. Figures-3 and 4 show that the isotherm data better fits the Langmuir equation than Freundlich equation since the values of coefficient of determination ($R^2=0.998$ and 0.996 for CAC and PALSC respectively) are higher than that of Freundlich isotherms ($R^2=0.931$ and 0.962). This supports the theory that the number of active sites on the carbon surface is limited and methylene blue forms a monolayer on the surface.

Table-3

Langmuir and Freundlich constants for methylene blue adsorption onto CAC and PALSC

Adsorbents	Langmuir			Freundlich		
	b	q _m	R ²	log K	1/n	R ²
Commercial activated carbon	3.3	302.1	0.998	2.36	0.40	0.931
Phosphoric acid activated Lapsi seed stone carbon	0.7	277.0	0.996	2.06	0.36	0.962

Table-4

Adsorption capacity of various adsorbents on methylene blue

Precursor	Adsorption capacity q _m mg/g	References
Orange peel	379.53	Foo.K.Y et al, 2012
Bamboo	286.10	Liue et.al, 2010
Coconut stalk	294.12	Deng et al. 2010
Pine wood powder	200.00	Wang et al .2009
Piassava fibres	276.40	Avela et al, 2010
Duran peel	284.00	Nuithitkul et al, 2010
Apricot stone	221.00	Demirbas et al, 2008
Lapsi seed stone	277.00	Present study

Conclusion

Activated carbon was prepared from Lapsi seed stone by chemical activation with phosphoric acid in the 1:1 ratio of using 50 % H₃PO₄ at 400 °C for 4 hours. Different parameters like iodine number, methylene blue adsorption, ash content, moisture content, pH_{pzc}, surface functional groups have been determined to find the quality of the activated carbon and compare with that of the commercial activated carbon. The experimental data of methylene blue adsorption is good in agreement with Langmuir adsorption model and has shown to a better fitting to the experimental data. The monolayer adsorption capacity calculated from Langmuir model are 277 mg/g and 302.1 mg/g for PALSC and CAC respectively. The quality of the activated carbon is found to be comparable to the commercial activated carbon. The activated carbon prepared from Lapsi seed stone can be applied as an adsorbent.

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