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Production of activated carbon from mango seed coat using chemical activation

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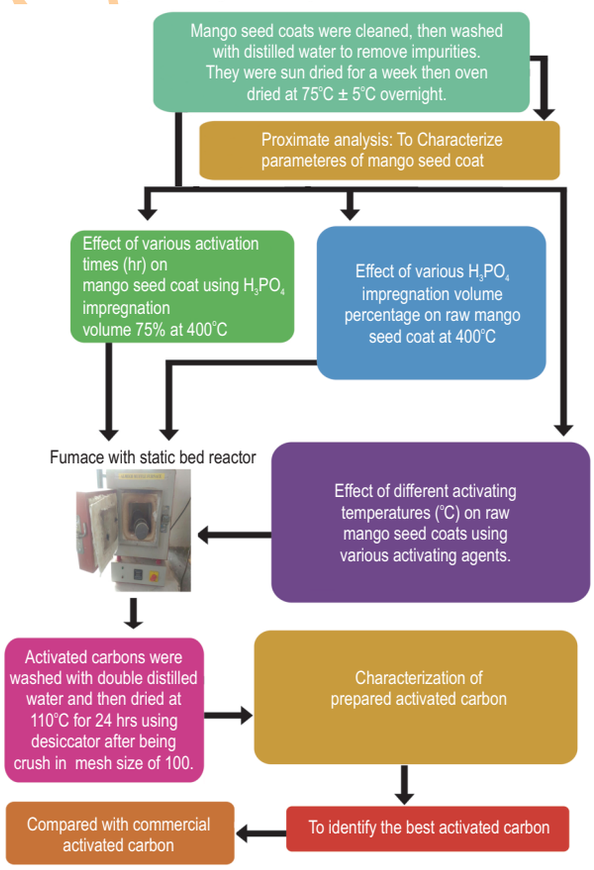
Abstract

Aim : This study was carried out find the feasibility of utilizing raw mango seed coat under various process conditions to produce the best activated carbon by chemical activation and compared with commercial activated carbon.

Methodology : Activated carbon was produced by chemical activation under various process conditions such as different activation temperatures, activating agents, impregnation volume percentages and activation times for pyrolysis in a programmable electrical furnace with reactor in the absence of air.

Results : The results were compared using phosphoric acid having 50% impregnation volume to other activating agents, the activating temperature was 400°C, activation time 1 hr, iodine number, methylene blue number, % yield and B.E.T surface area being 831 mg g^{-1}, 212 mg g^{-1}, 41.09% and 1114 m^2 g^{-1} respectively.



Interpretation : Carbon samples prepared using mango seed coat treated by H_3PO_4, showed clear open porous structures along with a larger pore size compared to commercially available activated carbon.

Key words: Activated carbon, B.E.T. surface area, Iodine number, Mango seed coat, Methylene blue number

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Introduction

Activated carbon is a well-known versatile product used for many applications. It is used to remove pollutants from gases and liquids and for purification in several industrial processes. Micro pollutants generally enter water through discharge from various processes including food, dye, textile, paper, plastic, chemical and petrochemical industries .The presence of organic pollutants in water has attracted much attention in recent decades because of their potential mutagenicity, carcinogenicity and toxicity. Moreover, it leads to undesirable effects on color, odor and taste of drinking water. It has been demonstrated that conventional treatment processes are ineffective in removing micro pollutants. Fluoride contaminated ground water exceeds the limits, creating natural and anthropogenic activities (Koshle *et al.,* 2016). India is among the 23 nations of the world, where fluoride contaminated ground water is creating health problems. 62 million people including 6 million children in 18 Indian states are affected. Activated carbon exhibits good capacity for removal of fluorine from drinking water (Kumar *et al.,* 2008).

Colored wastewater is a consequence of batch processes both in the dye manufacturing and dye consuming industries. 2% of dyes produced is discharged directly as aqueous effluent and 10% is subsequently lost during textile coloration process. An indication regarding the scale of problem is given by the observation that the annual market for dyes is more than 7X105 tons per year (Pearce *et al.,* 2003). The removal of color from effluents is one of the major environmental problems. Decolorization of wastewater has become one of the major issues in the waste water industry (Saravanan and Rathika, 2018). Activated carbon has been suggested to remove various pollutants from aqueous solutions (Shahmoradi *et al.,* 2015).

Activated carbon is widely used in many fields and can be produced from a variety of carbonaceous materials. Various materials are used to produce activated carbon, some of the most commonly used ones being agricultural wastes such as. Mango pit (Elizalde-Gonzalez and Henandez-Montoya, 2007), Mango seed (Moreno-Pirajan and Giraldo, 2012), Mango seed kernel (Pandharipade *et al.,* 2012), Mango seed shell (Mise and Jagannath, 2013), Palm kernel shell (Abechi *et al.,* 2013), *Delonixregia* plant litters (Daniel *et al.,* 2015), Sal seeds (Sing *et al.,* 2017) and Coconut shell (Regunton *et al.,* 2017). There are two basic methods for producing activated carbon which include physical and chemical activations (Dubey *et al.,* 2009). Chemical methods are more advantageous than the physical process. Therefore, chemical activation has been selected for the present study.

Mango, one the most important tropical fruits of the world, is usually found in Southern Asia, especially in Eastern India. In India, mango occupies 42.6% of the total area under food category, comprising 0.94 million ha with a total production of 8.21 million tons from various states. Besides, India is by far the largest producer of mangoes, accounting for 66.2% of the world production. There are nearly 1060 mango varieties in India. However, only about 20 of them are grown on commercial scale (Kostermans *et al.,* 1993). The mango pulp industry, while processing mangoes, generates by-products such as peels, kernels (Kittiphoom, 2012) and seed coats, thus producing a large amount of bio-waste. Mango production in India is large, only a small amount is utilized by traditional food industries and the rest is used as raw material for mango pulp industries. In the present study, mango seed coat, which is the bio waste products, of mango was used as a raw precursor for preparing activated carbon. Mango seeds produce a large amount of solid waste. To enhance the value of this bio waste, preparation of activated carbon has been attempted through it. Thus producing activated carbon from mango seed coat not only helps in terms of waste minimization but also helps in reduction of pollution.

Mango seed coat is a lignocellulosic material with hemicellulose, cellulose and lignin as the main components.The present study was carried out to investigate activated carbon prepared from mango seed coat by varying different conditions such as activation temperatures, activating agents, impregnation volume percentages and activation hours. Characteristics such as iodine number, methylene blue number, methyl violet number, percentage yield and Brunauer– Emmett –Teller surface area for various techniques were also measured to identify the quality of activated carbon produced and later compared with commercially available activated carbon.

Materials and Methods

Preparation of mango seed coat activated carbon: Mango seeds were collected from mango pulp industry located in Dharmapuri, Tamil Nadu, India. First, they were separated into seeds and seed coats followed by removal of unwanted fibers from the seed coats and then washed several times with distilled water to remove impurities. In the next step, they were sun-dried for a week and then oven dried at 75ºC±5ºC overnight. Proximate analysis (Lakshmi *et al.,* 2018) of mango seed coat (wet basis). was carried out next which indicated the following parameters: moisture content (2.94%), volatile matter (82.26%), fixed carbon (14.19%), ash content (0.61%) and sulphur (0.098%). After that, they were shredded into small pieces for easy impregnation. Finally, mango seed coat activated carbon was obtained by chemical activation method usingvarying process conditions. Different activating agents (Hagemann *et al.,* 2018) like HNO_3, H_3PO_4, KOH, HCl, $ZnCl_2$, NaOH, all at 1N were employed. Impregnation volume percentages used were 25% (25 ml of activating agent and 75 ml of water per 100 ml aqueous solution), 50%, 75%, 100%. Samples were impregnated using different aqueous solutions for 24 hr and then activated by varying activating temperatures from 300 to 600ºC at various activation time periods such as 1hr, 2hr, 3hr and 4hr (Miranda and Renita, 2008). The impregnated sample kept inside the static bed reactor was placed in an electrical furnace for pyrolysis. Nitrogen from gas cylinder was fed to the electrical furnace to maintain an inert medium. The activated carbon obtained after each experiment was washed repeatedly with double distilled water to remove ions. The

washed sample was dried in an air oven at 110ºC ± 5ºC for 24 hrs and then kept in a desiccator. Furthermore, it was crushed using mesh of size 100, stored in polythene cover and kept in an air tight container for further experiments.

Characterization of mango seed coat activated carbon: Iodine number was determined by standard test method ASTM (2006). Methylene blue number and methyl violet number were determined according to standard test method of BIS (1977).

Persent yield: It is a vital parameter in activated carbon preparation and was calculated by dividing mass of mango seed coat activated carbon after carbonization process and initial mass of mango seed coat taken and then was multiplied by 100.

B.E.T. surface area : Internal surface areas of mango seed coat activated carbons were measured by Quanta chrome NOVA (4200) based on standard N_2 adsorption data (at -196 ºC or 77K). Surface area was calculated using Brunauer– Emmett –Teller equation over relative pressure (P/P0) of 0.05 atm to 0.3 atm.

Results and Discussion

It is evident that use of H_3PO_4 as activating agent at 400°C at a higher iodine number produced the highest quality of activated carbon. Also, the impregnation volume percentage and time had significant effect on iodine number as shown in Fig. 1. Higher iodine number of 831 mg g^{-1} was observed when H_3PO_4 was used at 50% impregnation volume 400°C for 1 hr. This result agrees with the studies of activated carbon from rubber wood sawdust (Prakashkumar *et al.,* 2005; Kalavathy *et al.,* 2010), apricot stones (Soleimani and Kaghazchi, 2008) and sisal fiber (Senthilkumar *et al.,* 2013). With HNO_3, KOH, HCl and NaOH, activation temperatures did not have much influence on the iodine number. Furthermore, decrease in the iodine number indicates destruction and shrinkage of pores. Above 400°C, iodine number decreased with all activating agents except for $ZnCl_2$ and was maximum at 400°C with H_3PO_4 and 500°C with $ZnCl_2$ *i.e.,* 831 mg g^{-1} and 684 mg g^{-1}, respectively. This indicates a damage to the structure of activated carbon in the form of collapse of the pore wall with increase in temperature above 400°C for H_3PO_4 and 500°C for $ZnCl_2$.

Activating temperature of 400°C mainly influences methylene blue number for activating agents. H_3PO_4 and KOH, compared to other activating agents. Additionally, Impregnation volume percentage and time also had significant effect on methylene blue number. Maximal methylene blue number observed were 130 mg g^{-1} and 212 mg g^{-1} with activating agents KOH and H_3PO_4, at 50% impregnation volume 400°C temperature for 1 hr with H_3PO_4, having the highest methylene blue number compared to other activating agents as illustrated in Fig. 2. When H_3PO_4 reacts within the internal cellulose structure of mango seed coat, it is assumed to induce polymerization leading to an enhancement of pore volume, and thus global volume expansion favoring formation of mesopores (Olawale *et al.,* 2015). The activating temperature increases methylene blue number, thus increasing the micropore structure and widening of mesopores. Moreover, above 400°C decrease in methylene blue number decreases with any chemical activating agent indicates that there is no new pore formation, with the only possibility being pore widening.

The quality of % yield was maximum for mango seed coat activated carbon at 49.06% and 41.09% with H_3PO_4 at impregnation volume of 50% for 1 hr at 300°C and 400°C, respectively, (Fig. 3). It is also evident that % yield decreases with increasing activation temperature for various chemical activating agents. This result agrees with the studies of activated carbon from waste materials (Aajish and Thirumal, 2014) and groundnut foliage (Lakshmi *et al.,* 2018). During the carbonation process, the lignocellulose in the mango seed coat reacts with phosphoric acid. Consequently, the acid first attacks hemicellulose and lignin as cellulose is more resistant to acid hydrolysis. These reactions are accompanied by further chemical transformations which include dehydration, degradation and condensation. As the acid concentration increases, aromatic condensation reactions also take place among the adjacent molecules which result in the formation of gaseous products from the hydro aromatic structure of carbonized char leading to decreased yield of carbon. The % yield from H_3PO_4 is relatively higher than other activating agents, which indicates the ability of H_3PO_4 to retain carbon and to avoid the loss of volatile materials. In addition to that, H_3PO_4 activation promotes dehydration and redistribution of biopolymers and also favors the conversion of aliphatic compounds to aromatic, thus increasing the yield of activated carbon.

Iodine number and methylene blue number value indicate the micropore and mesopore developed in the mango seed coat activated carbon during the activation process. Based on iodine number and methylene blue number, 400°C was selected as optimum activation temperature (Cheung *et al.,* 2017) and the performance of selected activating agents was found in the order: H_3PO_4 > $ZnCl_2$.> KOH >NaOH>HCl> HNO_3. H_3PO_4 has been widely used as an activating agent because of its tendency to create palpable surface area and better yield, which was also confirmed in this study.

The influence of H_3PO_4 at 400°C for 1 hr at different impregnation volume percentage on mango seed coat has been shown in Fig. 4. Four different impregnation volumes of 25%, 50%,75% and 100% were used. The iodine number was maximum at H_3PO_4 impregnation volume of 75% *i.e.,* 976 mg g^{-1} and methylene blue number at H_3PO_4 impregnation volume of 50% *i.e.,* 212 mg g^{-1}. Furthermore, iodine number increased as H_3PO_4 impregnation volume percentage increased from 25% to 75%, after which there was a decrease in iodine number because activated carbon micropores decreased with increasing impregnation volume due to excessive carbon burn-off, collapse of pore walls and expansion of micropores to mesopores. Other than that, methylene blue number also increased with increase in

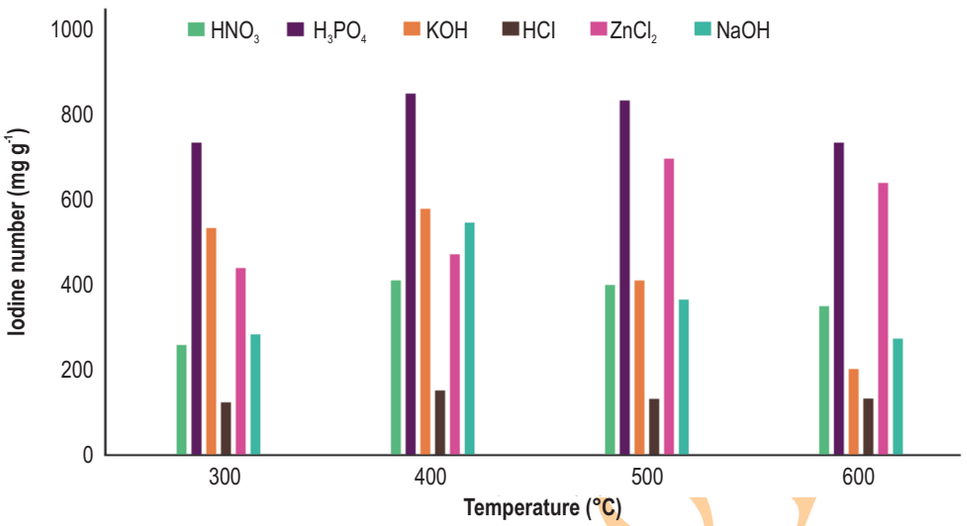

Fig. 1 : Effect of different activating temperatures (°C) on raw mango seed coats using various activating agents and the corresponding Iodine number (mg g^{-1}) for mango seed coat activated carbon. Activating condition: Impregnation volume percentage : 50; Normality of activating agents: 1N; Activating temperature: 300°C, 400°C, 500°C, 600°C; Activating time: 1hr and Activating agents: HNO_3, H_3PO_4, KOH, HCl, $ZnCl_2$, NaOH.

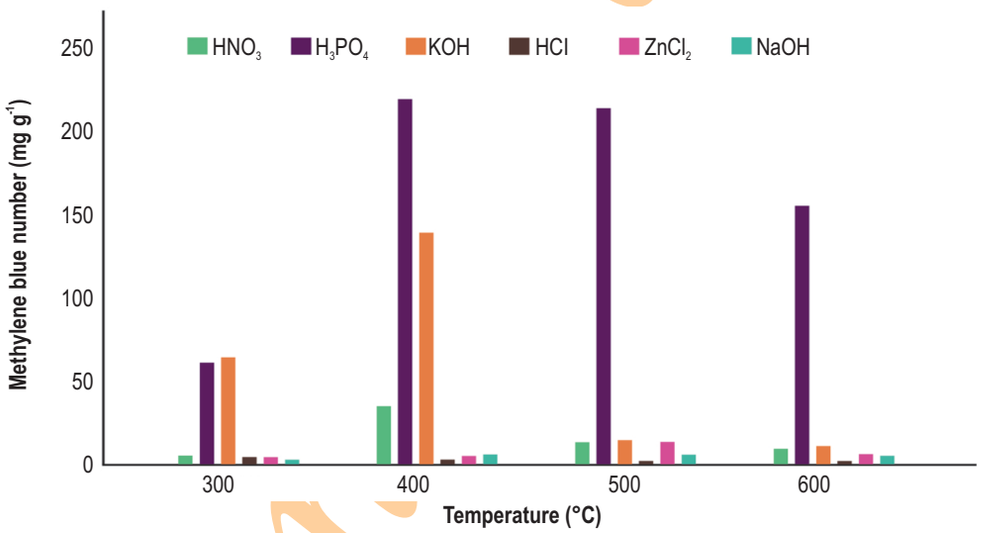

Fig. 2 : Effect of different activating temperatures (°C) on raw mango seed coats using various activating agents and the corresponding Methylene blue number (mg g^{-1}) for mango seed coat activated carbon. Activating condition: Impregnation volume percentage: 50 Normality of activating agents: 1N; Activating temperature: 300°C, 400°C, 500°C, 600°C Activating time: 1 hr and Activating agents: HNO_3, H_3PO_4, KOH, HCl, $ZnCl_2$, NaOH.

H_3PO_4 impregnation volume percentage from 25% to 50%. The decrease in this number may be attributed to new micropores being created inside the mesopore of mango seed coat activated carbon. Moreover, the % yield increases with increased in H_3PO_4 impregnation volume from 25% to 50%, after which it decreased. Yakout and El-Deen, (2016) observed that % yield of carbon decreases as H_3PO_4 impregnation volume percentage increases. Excessive phosphoric acid promotes gasification of char and increase the total weight loss of carbon (Prahas *et al.,* 2008). With increasing quantity of acid, it is expected that mass transfer within the substrate will be enhanced, leading to higher rate of reaction which manifests as reduced yield. Based on iodine number, H_3PO_4 impregnation volume of 75% was selected as impregnation volume percentage for mango seed coat activated carbon for varying activation hours. The effect of various activation time employed on H_3PO_4 at impregnation volume of 75% at 400°C on various parameters is shown in Fig. 5. Consequently, a high iodine number for activation time of 1 hr was found *i.e.*, 976 mg g^{-1} (Senthilkumar *et al.,* 2013). Additionally, methyl blue number was highest at 173 mg g^{-1} for 2 hr activation

and % yield was maximum at 41.09% for 1 hr activation. Alternatively, methyl violet number was highest at 110 mg g^{-1} for 4 hr activation time. As activation time increases, iodine number decreases (Ceyhan *et al.,* 2013). Besides, residence time also influences the porosity development during the activation process. As it increases, the cleavage of oxygen groups are inhibited due to the presence of phosphate esters, and thus cyclization reaction does not occur (Rajagopal *et al.*, 2006).

Influence of activation time on the development of micropore volume decreased by increasing activation time from 1hr to 2 hr, leading to rapid decrease in the iodine number. By increasing activation time, both methylene blue number and methyl violet number increased. Beyond activation time of 2 hr, there was no significant change in methylene blue number however, methyl violet number increased. Clearly, for the production of activated carbon, activation time of 2 hr or more is

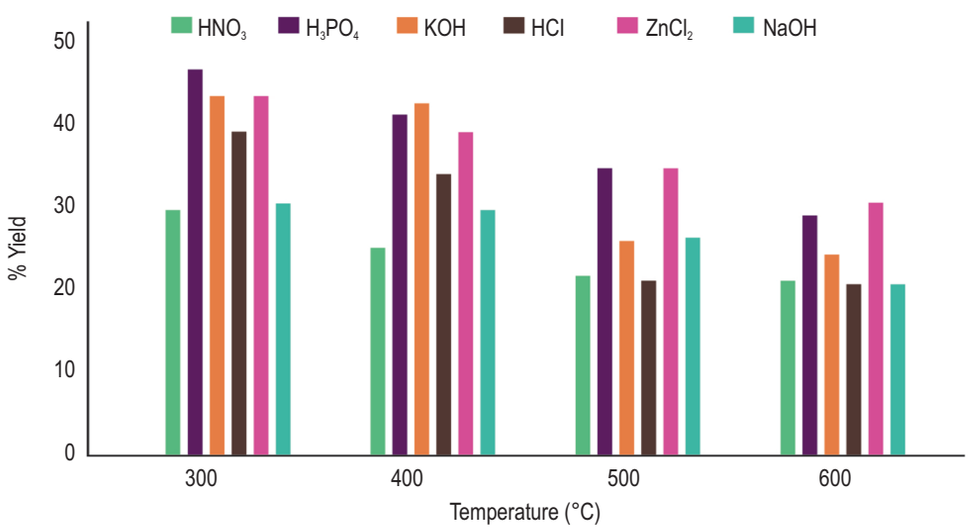

Fig. 3: Effect of different activating temperatures (°C) on raw mango seed coats using various activating agents and the corresponding % yield for mango seed coat activated carbon. Activating condition: Impregnation volume percentage: 50; Normality of activating agents:1N; Activating temperature: 300°C,400°C,500°C,600°C Activating time: 1hr and Activating agents: HNO_3, H_3PO_4, KOH, HCl, $ZnCl_2$, NaOH.

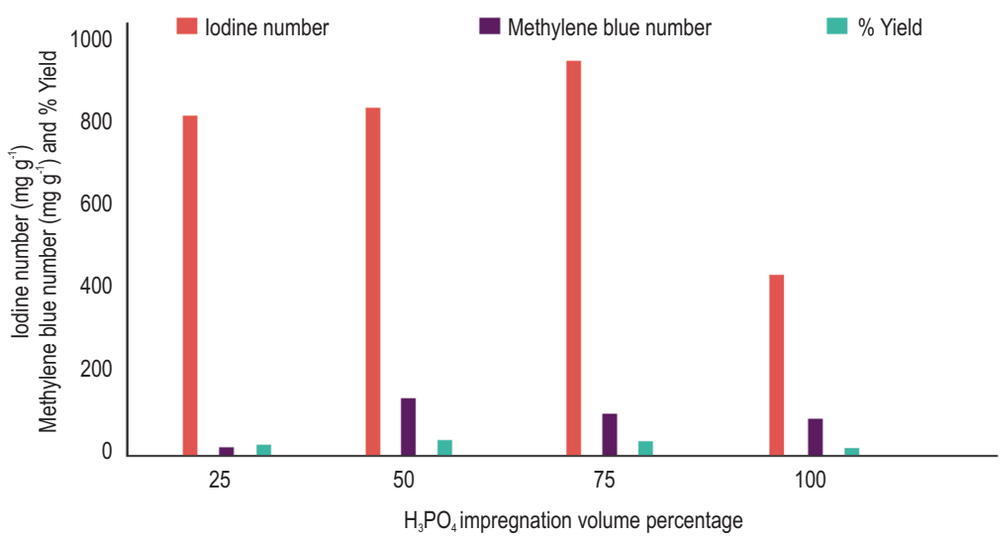

Fig. 4: Effect of various H_3PO_4 impregnation volume percentages on raw mango seed coat at 400°C. Activating condition: H_3PO_4 Impregnation volume percentage: 25, 50, 75, 100; H_3PO_4 Normality:1N and Activating time: 1hr.

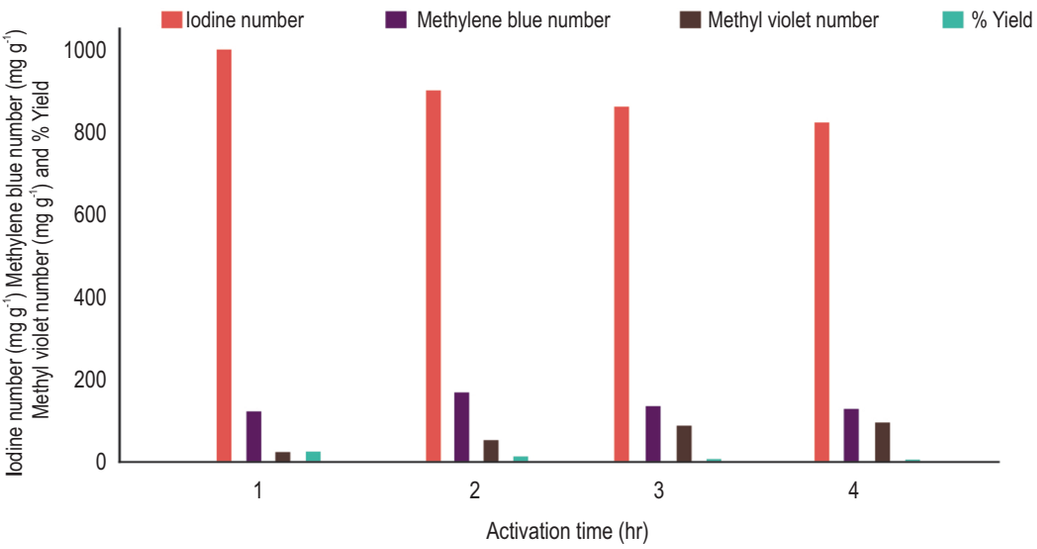

Fig. 5: Effect of various activating time (hrs) on mango seed coat H_3PO_4 impregnation volume of 75% at 400°C. Activating condition: Activating temperature: 400°C; H_3P0_4; Normality: 1N and Activating time: 1hr, 2hr, 3hr and 4hr.

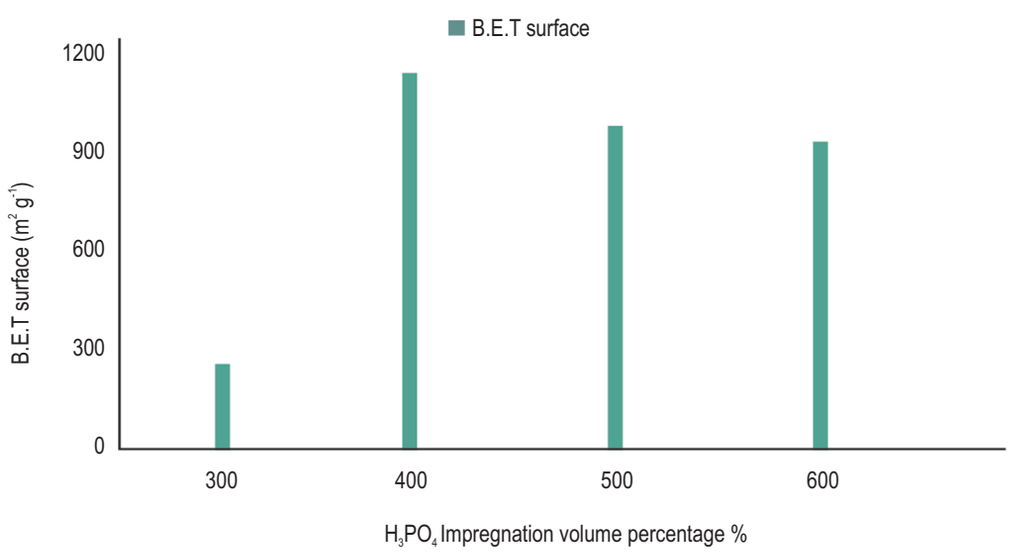

Fig. 6: Effect of different activating temperatures (°C) on B.E.T surface area ($m^2 g^{-1}$) of mango seed coat activated carbon. Activating condition: Activating Agent: H_3PO_4; Normality of activating agents: 1N; Impregnation volume percentage: 50% and Activating time: 1hr.

not helpful for further pore development, though transformation of micropores to mesopores can be expected. This pore drilling dominant mechanism increases the diameter of pores only near their mouths (Herbert *et al.,* 2008), thus, increasing activation time and decreasing % yield (Aajish and Thirumal, 2014).

Based on methylene blue number, H_3PO_4 impregnation volume of 50% was selected as the optimum impregnation volume percentage for mango seed coat activated carbon and was further used to evaluate Brunauer–Emmett–Teller surface area. Fig. 6 shows the effect of various activating temperatures on the said surface area. It was highest at 1114 $m^2 g^{-1}$ with H_3PO_4 at 50% impregnation volume at 400°C for 1 hr, thus showing that the surface area adsorption capacity was high. Subsequently, as activating temperature increases from 300°C to 400°C (Dubey *et al.,* 2009), the B.E.T surface area also increases accordingly after which it decreases gradually. This can be attributed to degradation of cell wall, causing larger pore size and less surface area above 400°C (Boonpoke, 2015). The mango seed coat activated carbon's adsorption capacity is

based on its iodine number and methylene blue number. Gas adsorbing carbons usually have more micropores and liquid adsorbing carbons have significant number of mesopores due to larger size of liquid molecules (Wang *et al.,* 2013). In the present study, mango seed coat activated carbon was used for the removal of gaseous as well as liquid pollutants.

The process conditions for removal of gaseous impurities were: Activating agent H_3PO_4 (1N), impregnation volume 75%, activation time 1 hr, activating temperature 400°C: Iodine number 976 mg g^{-1}, Methylene blue number 153 mg g^{-1} and Methyl violet number 45 mg g^{-1}. On the other hand, for removal of impurities from liquids process conditions were: Activating agent H_3PO_4 (1N), impregnation volume 50%, activation time 1 hr, activating temperature 400°C. Iodine number 831mg g^{-1}, methylene blue number 212 mg g^{-1} and B.E.T. surface area 1114 $m^2 g^{-1}$, respectively. The commercial activated carbon (e-Merch India Ltd) had iodine number 834 mg g^{-1}, methylene blue number 210 mg g^{-1}, methyl violet number 150 mg g^{-1} (Rajgopal *et al.,* 2006) and B.E.T. surface area 890.0479 $m^2 g^{-1}$ (Shalna and Yogamoorthi, 2015).

US Environmental Protection Agency has recommended activated carbon as one of the best dye removers (Karthikeyan *et al.,* 2012). However, cost is the limiting factor for use of commercial activated carbon. From the above results, it can be concluded that the study of preparing activated carbon from mango seed coat produces good quality results as compared to commercial activated carbon. It can be used commercially for necessary applications. Thus, production of activated carbons with high surface area from mango seed coat (a waste material from mango pulp and fruit juice industries) is indeed important from economic and environmental aspects.

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