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Synthesis, Characterization and Proximate Analysis of Activated Carbon prepared from Kinnow and Jambhiri Peels

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ABSTRACT
The fruit peels are generally unwanted by-products generated by fruit juice makers and food processing industries. Citrus fruits, mainly oranges and lemon are very popular worldwide. After consumption these peels are often discarded in huge amount which led to a big nutritional and economic loss and environmental problems. The utilization of these low cost waste has now become a great alternative to commercial activated carbon for removal of dyes from waste water. In this study, Kinnow(Citrus reticulate) and Rough lemon(Citrus jambhiri) peels have been carbonized at different temperatures between 250 to 400°C for 15, 30, 45 and 60 minutes for the determination of optimum conditions for pretreatment. The resulting carbon is then chemically treated with different concentrations of H_3PO_4. The results obtainedrevealed that the optimum carbonization temperature for Kinnow and Jambhiri peels were 300°C and 350°C for 30 minutes, respectively. The highest percentage reduction of dye considered was one treated with 1M phosphoric acid. Therefore, it has been concluded that these peels can be considered as raw materials for the production of activated carbon.
Keywords: *Activated carbon, Carbonization temperature, Carbonization time, dye.*

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INTRODUCTION

Throughout our daily lives, we consume a variety of fruits.Fruits are a good source of fibre, vitamins, and minerals [1]. One of the most significant commercial fruit crops grown in India is citrus. Citrus fruits are often used in the juice industry, but the peels, pomace, rind and seeds are considered to be among the most common wastes. Peel waste is a challenge for the processing businesses and environmental monitoring organisations since it decomposes quickly and is seasonal [2]. Citrus trash is not an exception to the rising focus on turning waste materials into valuable goods. High resource use and certain dyes and compounds that are challenging to remove from the waste water stream, both contribute to environmental damage [3]. Fruit waste product recovery can boost the overall economics of the processing units. In addition, the issue of environmental contamination might be significantly lessened. Using an activated carbon, adsorption technique is one potential method for reducing pollution [4]. A variety of substances can be removed, recovered, separated, and modified using activated carbon in both the environmental and industrial sectors. From an industrial standpoint, activated carbon is one of the most significant, adaptable, and widely utilised adsorbents [5]. In this work, the peels of Citrus reticulate blanco (Kinnow) and Citrus jambhiri (Rough lemon) were selected.
The citrus fruit Kinnow (*Citrus reticulata* Blanco), a cross between a king and a willow leaf, is a significant crop in India. It is commonly grown in northern India, particularly in Punjab, Haryana, Rajasthan, and Himachal Pradesh [6].
Citrus jambhiri, commonly known as JambiraNimbu in Sanskrit, is most commonly used citrus rootstock belonging to the family Rutaceae. It grows frequently and is primarily found in north India [7].
This study's focus is thus on determining the optimum conditions needed for pre-treating peels waste in order to make it viable as an adsorbents within this framework. This covers elements like the carbonization time and temperatures. In addition, the produced carbons were later subjected to chemical changes using 85% H_3PO_4 at various concentrations [8]. Next, the viability of employing both peels as adsorbents for dye removal is examined [9]. The developed adsorbents were characterised for proximate and FTIR analyses [10].

MATERIAL AND METHODS

Collection of fruit peels and preparation

The primary raw materials used in this study were Kinnow and Jambhiri or Rough lemon peels that were acquired from different fruit vendor locations in Jodhpur and Pushkar, Rajasthan state. In preparation for the carbonization studies, both samples were exposed to sun drying for four to five days to significantly lower their moisture contents, and they were subsequently reduced in size by being crushed with a mortar and pestle. The dried samples (Kinnow peels, KP and Jambhiri peels, JP) were then grounded into powder and sieved in sieve shaker and stored in a sealed plastic bag with silica gel to minimize dampness. Powdered sample was further preserved in dessicator and used in adsorption studies. All chemicals used in this study were of analytical grade. Deionised water was used to prepare all the solutions.

Carbonization of Kinnow and Jambhiri peels:

The dried peels were ground into a powder for the carbonization studies, and 15 g of the powdered samples were then divided among six distinct clean, pre-weighed crucibles and placed in the hot zone of a muffle furnace. At various temperatures, the peels underwent carbonization (250, 300, 350 and 400˚C). In order to determine the ideal conditions for the procedure, the samples were maintained at each temperature for a variety of lengths of time (15, 30, 45, and 60 min). Once the predetermined time had passed, the contents were taken out of the muffle furnace to cool for an additional hour in the open air. Up till a significant amount of carbonised material was acquired, this process was repeated. The carbonaceous materials produced at different temperatures and time were then characterized.

Chemical Activation of carbonized materials

The activation of carbonaceous materials produced using phosphoric acid were carried out in accordance with description reported in work by S.Ho [11].

Proximate Analysis of produced carbon

The resulting activated material underwent characterization to assess its fixed carbon, ash content, volatile content, and moisture content, among other characteristics [12]. The ASTM [13] process was used to estimate the fixed carbon content, whereas the AOAC [14] (Association of Analytical Chemistry) protocol was used to determine the volatile, ash, and fixed carbon contents (1994). Using FTIR spectrophotometer Agilent FTIR Cary 630, the produced sample's spectra were captured before and after the dye was adsorbed using activated carbon. The features of the carbon for use in treating industrial wastewater were assessed using the standard values of the physicochemical properties of the adsorbent as references.

Preparation of Adsorbate

Stock solution of the dye(Congo Red) was prepared by dissolving 1g of the powdered dye in 1L distilled water to have 1000mg/L dye concentration in the solution. Experimental dye solution of desired concentrations were obtained by appropriate dilution of each stock solution [15].

RESULTS AND DISCUSSION

Effect of Carbonisation Temperature and Time:

In the process of making activated carbon from kinnow and jambhiri peels, the effects of carbonization temperature and duration on fixed carbon content, charcoal yield, moisture content, volatile content, and ash content were examined. The findings are shown in Tables 1,2,3 and 4.

Table 1: Measured parameters of peel of Kinnow material carbonized for various temperatures

S/N	Temperature	Time(min)	Mass(g)	Ash Content(%)	Volatile Content(%)	Moisture Content(%)	Fixed Carbon(%)	Charcoal Yield(%)
1	250	15	5	7.08	30.45	4.83	57.64	45.61
2	300	15	5	7.10	29.64	4.75	58.51	34.54
3	350	15	5	7.89	31.27	4.31	56.53	33.91

Table 2: Measured parameters of peel of Kinnow material carbonized for various times

S/N	Temperature	Time(min)	Mass(g)	Ash Content(%)	Volatile Content(%)	Moisture Content(%)	Fixed Carbon(%)	Charcoal Yield(%)
1	300	15	5	7.10	29.64	4.75	58.51	34.54
2	300	30	5	7.01	27.05	4.20	61.64	34.80
3	300	45	5	7.93	26.9	4.33	60.84	33.72

Table 3: Measured parameters of peel of Jambhiri material carbonized for various temperatures

S/N	Temp.	Time (min)	Mass(g)	Ash Content(%)	Volatile Content(%)	Moisture Content(%)	Fixed Carbon(%)	Charcoal Yield(%)
1	300	15	5	7.02	31.6	4.74	56.64	39.89
2	350	15	5	7.12	28.9	4.29	59.69	37.04
3	400	15	5	7.55	29.5	4.12	51.28	35.20

Table 4: Measured parameters of peel of material carbonized for various

S/N	Temp.	Time (min)	Mass(g)	Ash Content(%)	Volatile Content(%)	Moisture Content(%)	Fixed Carbon(%)	Charcoal Yield(%)
1	350	15	5	7.12	28.9	4.29	59.69	37.04
2	350	30	5	7.15	28.7	4.11	60.04	38.23
3	350	45	5	7.66	29.0	4.07	59.27	38.03

With a constant carbonization time of 15 minutes and a constant sample mass of 5 g, it can be seen from Tables 1 and 3 that all the parameters investigated did indeed change, as the carbonization temperature changed. These parameters include fixed carbon content, charcoal yield, moisture content, volatile content, and ash content. This has demonstrated how the factors under consideration rely on the process of carbonization temperature.

The results from the parameters examined when the carbonization duration was changed as shown in Table 2 and 4, but this time, the carbonization temperature was kept constant at 300˚C, and the mass of the sample utilised for all the runs was also kept constant at 5 g. The impacts of carbonization time on the generated peels activated carbon's properties are shown in Table 2 and 4. Similar to what was observed when the carbonization temperature was changed, it was also found in this instance that the parameters under consideration responded to changes in the process of carbonization time. This was a sign that along with carbonization temperature, carbonization time was also the key elements controlling this process [16].

The amount of the carbonised product (charcoal yield) produced from the kinnow and jambhiri peels was observed to decrease when the carbonization duration was increased, as shown in Tables 2 and 4. The severe burning, oxidation, and collapse of the peels' pore structures, which were shown to prevail during prolonged carbonization times, were found to be the cause of this.

According to the findings of the carbonization of Kinnow and Jambhiri peels shown in Tables 1,2,3 and 4, the highest percentage of fixed carbons was produced when the carbonization temperature and duration were 300˚C and 30 minutes for Kinnow peels and 350˚C & 30 minutes for Jambhiri peels, respectively. As a result, these parameters were chosen as the best ones for the process.

Effect of Phosphoric acid as Activating Agent on Adsorption of Dye

The different concentrations of phosphoric acid [17],H_3PO_4(85%) were used to activate the carbon content that was obtained at optimum percentage for Kinnow and Jambhiri peels. The resulting Kinnow peel activated carbon (KPAC) and Jambhiri peel activated carbon (JPAC) were then used to remove dye from the effluent acquired from the cotton dyeing industry. The results of the overall reduction of dye using KPAC and JPAC are as given in Table 5 and 6 for H_3PO_4 treated carbons, respectively.

Table 5: Reduction of dye using different concentrations of H_3PO_4 treated KPAC

S/N	Sample Code	Phosphoric Acid	% Reduction
1	KPAC300	0.5M	75.6
2	KPAC300	1M	89.8
3	KPAC300	1.5M	44.5

*KPAC300–Kinnow peel activated carbon at 300˚C

Table 6: Reduction of dye using different concentrations of H_3PO_4 treated JPAC

S/N	Sample code	Phosphoric Acid	% Reduction
1	JPAC350	0.5M	76.5
2	JPAC350	1M	88.9
3	JPAC350	1.5M	36.4

*JPAC350 – Jambhiri peel activated carbon at 350˚C

From Table 5 and 6, it was discovered that the activated carbon treated with 1M H_3PO_4showed greater percent reduction which indicates that the carbon is good for dye adsorption.

FTIR Analysis of Produced Activated Carbon:

Because the chemical structure of an adsorbent is crucial to understand its adsorption nature, the FT-IR spectra of the developed Kinnow peel activated carbon and Jambhiri peel activated carbon were conducted as a qualitative analysis to gain better insights into the surface functional groups available on the surface of the investigated adsorbent.The widely used technique to find functional groups in samples is the FTIR method [18]. Even in the solid state, the atoms in molecules are never static; they constantly fluctuate around their equilibrium locations. The mass, length, and strength of the bonds that an atom has created all influence the frequency at which it vibrates. The surface functional groups on activated carbon that include oxygen have a significant impact on how the material reacts and behaves.

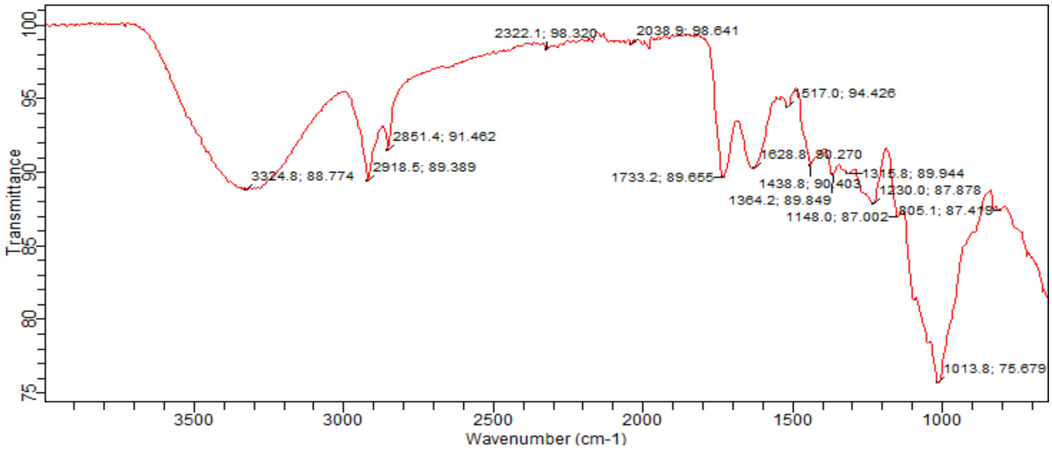

Fig 1: FTIR spectra of Kinnow Peel Activated Carbon.

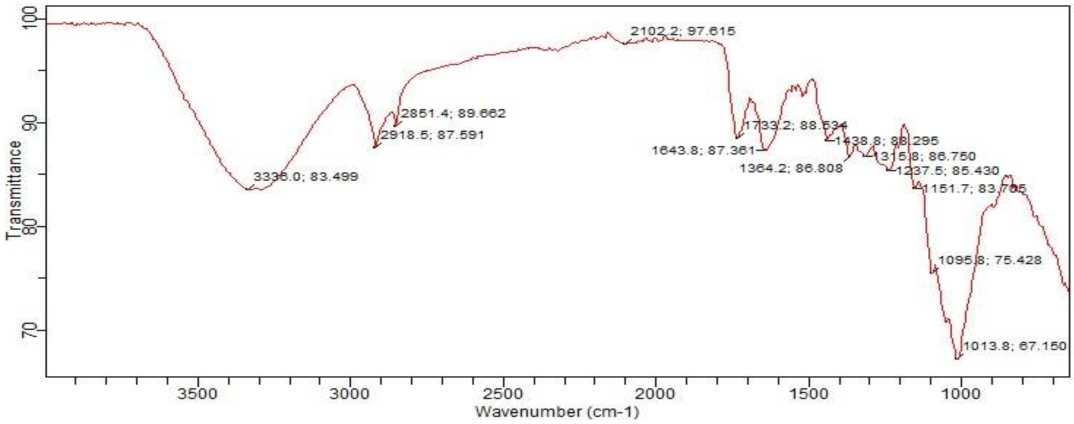

Fig 2: FTIR spectra of Kinnow Peel Activated Carbonafter adsorption.

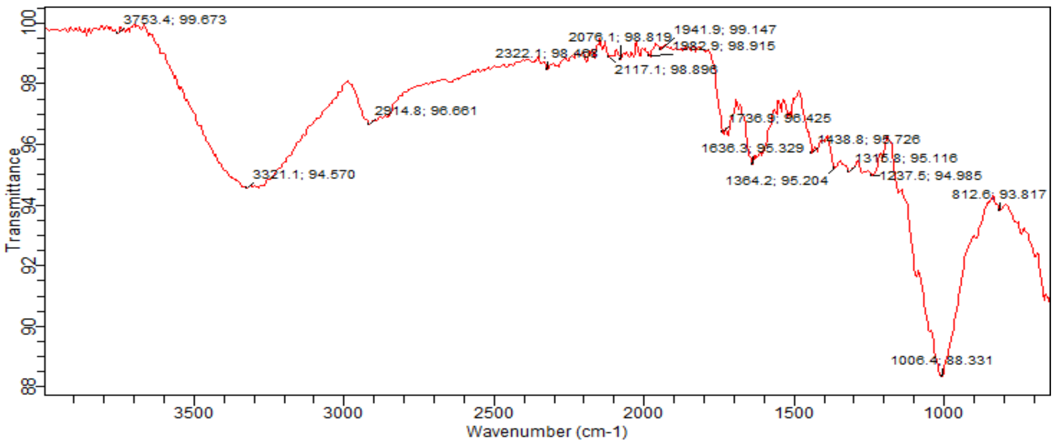

Fig 3: FTIR spectra of Jambhiri Peel Activated Carbon.

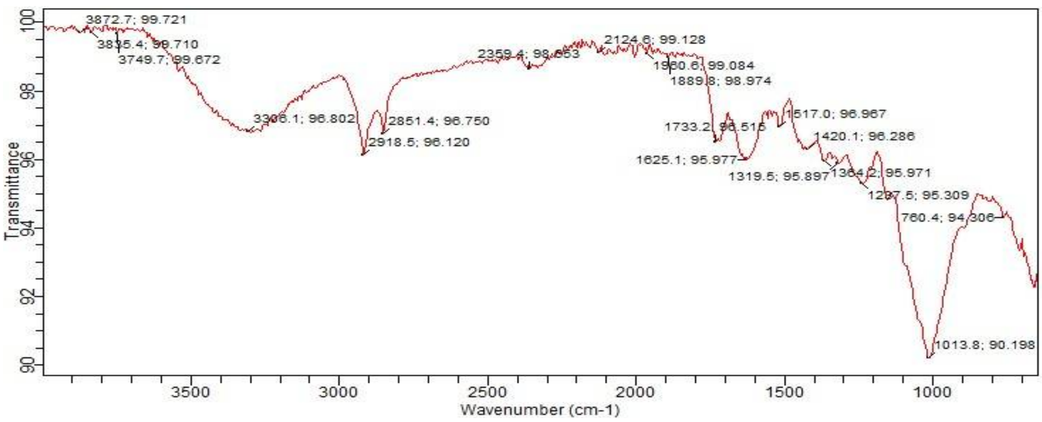

Fig 4: FTIR spectra of Jambhiri Peel Activated Carbonafter adsorption

In order to determine the mechanism of adsorption of dye ions on the adsorbents, FTIR spectra were taken before and after the adsorption of congo red dye as seen in Figure 1 to 4. The spectrum shows the presence of peaks as follows: 3324 cm^{-1} (KPAC) and 3321 cm^{-1}(JPAC) which corresponds to stretching vibrations of C-H and N-H, O-H stretching mode of carboxylic acid and O-H band of alcohol.The band at 2918 cm^{-1} and 2914cm^{-1} shows O-H stretching mode (carboxylic acid).A new band was found at 2102cm^{-1} in fig 2 which shows stretching vibration of C≡C and band at 2851 in fig 4 shows stretching vibration of C-H(aldehyde hydrogen), O-H(carboxylic acid).The band at 1517cm^{-1}which corresponds to symmetric vibration of C=C (aromatic ring), N-H bending in 2˚ amine, asymmetric stretching of NO_2(aromatic nitro) has found to be diminished in fig 2 and band at 812cm^{-1} in fig 3 has also diminished after adsorption. The peak at 1628cm^{-1} in fig 1has shifted to 1643cm^{-1} in fig 2. During adsorption process, there were some interactions between the peels and dye ions in aqueous phase, consequently resulting in shifts of characteristic peaks. As peels were activated with phosphoric acid , the sharp adsorption band between 900 and 1200cm^{-1} may be attributed to the presence of phosphorus species.

CONCLUSION

The results obtained from this work was carried out to develop an adsorbent from Kinnow and Jambhiri peel for use in wastewater treatment, specifically for adsorption of dye, have revealed that the optimum carbonization temperature and time for the preparation of Kinnow peel activated carbon(KPAC) and Jambhiri peel activated carbon (JPAC) with fixed content were 300˚C, 30 min and 350˚C, 30min, respectively. Activated carbonfrom Kinnow and Jambhiri peels have been successfully prepared by chemical activation method using Phosphoric acid as an activating agent. In addition, KPAC and JPAC with 1.0 M phosphoric acid was found to be the best among the treated ones because it has relatively highest percentage reduction of congo red dye investigated in this work. The prepared adsorbent was then characterized using FT-IR technique.

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