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(Original Research)

Unlocking The Rice Straw Agro-Waste Potential As Activated Carbon: Pyrolysis And Characterization

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Abstract

Rice growing is widely practiced in the northern Indian region known as the Indo-Gangetic Plain. A significant amount of rice straw is burned in the field due to the absence of a waste management system. To boost its economic value, Rice straw is converted into activated charcoal, which can subsequently be used for wastewater treatment, metal extraction, air purification, and other applications. The purpose of this work was to produce porous activated carbon particles from RS waste using a chemical activation procedure that included 40% orthophosphoric acid. The process of synthesizing porous carbon particles involves three steps: (i) carbonization; (ii) chemical impregnation; and (iii) activation treatment. Variations were made to the activation temperature, residence time, and activating agent concentration to attain the best possible approach for the activation treatment. Activated carbon was characterized using different techniques, such as XRD, FTIR, FESEM and EDX. Experimental results showed that this approach is effective at producing porous carbon particles. ACs synthesized were carbonaceous and amorphous in form, as determined by X-ray diffraction studies. FTIR revealed the presence of functional groups that are good for adsorption, such as hydroxyl, carbonyl, amines, aromatic, and others. Scanning electron micrographs showed that activated carbon has a compact and porous structure. When comparing the activated carbon to the original rice straw, EDX demonstrates the increased carbon content. The optimal conditions determined are 700°C, a ratio of 1:3, and a duration of 90 minutes. The results of the investigation show that the agricultural wastes used in the evaluation may serve as low-cost sources of material for the production of local ACs, thereby addressing the issue of disposing of agricultural wastes.

Key Words	Rice straw, Activated carbon, Impregnation ratio, Activation temperature, Time duration

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1. INTRODUCTION

The wastes generated represent a problem for disposal, in contrast to industrialized nations where agricultural wastes are transformed into raw materials for a range of industrial uses. The current situation is different in India, where wastes are burned and disposed of in open fields, producing greenhouse gasses like carbon dioxide (CO_2) that contaminate the air (Koul et al. 2022). Wastes are sometimes disposed of in urban and rural regions, contaminating water sources (Singh et al. 2022). When wastes are disposed of on land, organic matter breaks down and generates methane (CH_4) emissions, which contaminates both the air and the land. The methods employed to handle agricultural waste harm the environment, and occasionally the wastes themselves serve as carriers of pathogenic organisms, endangering the health of the general public (Kour et al. 2023).

Around the globe, activated carbon (AC) is known as a popular and widely used adsorbent for water and wastewater treatment. The oldest known adsorbent for water filtration is charcoal, which is the precursor to contemporary AC (Parlayıcı et al. 2024). The term "activated carbon" is commonly used to describe materials composed of carbon that have a well-developed interior pore structure (Serafin et al. 2024). It, often called activated charcoal, has various definitions by different authors throughout the literature. A chemical study or structural formula is insufficient to adequately characterize this class of porous carbonaceous materials (Li et al. 2022). It can also be defined as a black carbonaceous substance that is solid, tasteless, microcrystalline, non-graphitic, and has a very porous structure. Some researchers describe AC as a porous carbon material featuring carbon atoms that are separated by countless tiny pores opened during the activation process (Ale et al. 2022). In summary, AC consists of black carbon-rich porous materials with a large surface area formed by carbonizing substances with high elemental carbon content. (Tetteh et al. 2024). A range of carbonaceous-rich materials, including wood (Yustanti et al. 2021), lignite (Ahmad et al. 2022), coal, and coconut shell (Spencer et al. 2024) are used to make AC. The versatility of AC is evident in its wide range of functional groups present on its surface, high surface area, high porosity, and a well-developed interior pore structure with micro-, meso-, and macropores. These properties make AC an ideal material for a variety of applications, primarily in the environmental field (Singh et al. 2023; Pavlenko et al. 2022). The one-step method of manufacturing AC, often referred to as chemical activation, occurs when activating chemical agents are present. It provides several benefits; Firstly, materials can usually be activated at a lower temperature and in a shorter time. Secondly, the influence of the chemicals can encourage the growth of pores in the carbon structure (Maazouzi et al. 2023, Lan et.al. 2023). Carbon yields from chemical activation are often higher than those from physical activation.

Typically, activated carbon is made from materials that contain a lot of element carbon (Serafin et al. 2024). Two significant sources for activated carbon preparations are biomass that contains lignocellulosic components, such as agricultural wastes and coal (Singh et al. 2023, Gayathiri et al. 2022). Commercial activated carbon is made from nonrenewable and costly precursors, such as wood, coal, peat, lignite, and petroleum wastes (Sosa et al. 2023). However, agricultural byproducts like Marula nutshell (Mkungunugwa et al.,2021), cereals (Ukanwa et al. 2019), corn cobs (Sahu et al. 2024, Medhat et al. 2021), almond shells (Singh et al. 2024, Bicil et al. 2021), rice husk (Kumari et. al. 2023, Bilal et.al. 2021) rice straw (Charoensook et al. 2021, Vunain et al. 2021) and bamboo (Duan et al. 2023) have been successfully used to create affordable activated carbon. Thus, as a sustainable waste management approach, RS (Rice Straw) were utilized in this work to generate AC through chemical activation. The conversion of unwanted agricultural waste into high-value adsorbents is one of the major economic and environmental benefit processes by utilizing these agricultural wastes as AC precursors. In this way, less activated carbon will be imported from outside markets, and using agro-waste residues as precursors for AC synthesis will support the economic growth of the nation (Heidarinejad et al. 2020).

The proper use and management of agricultural waste can have a positive impact on the environment by reducing trash and removing pollutants from water sources (Wang et al. 2021). Since agricultural waste products are plentiful and renewable, many research groups are interested in producing economical AC from them. The generation of useful goods made from agricultural waste, such as AC, can increase its utilization, minimize waste byproducts, and provide money to needy areas (Mishra et al. 2024). Techniques that reduce overall treatment costs by using natural, indigenous products are particularly appealing because they lessen reliance on imported chemicals for water treatment, require minimal transportation, and offer suitable, localized solutions for water quality problems (Lotfy et al. 2023). In India, stubble burning is also a big concern. We can use agro waste for the preparation of adsorbent (Tokas et al. 2021). The highly effective approach for the treatment of wastewater is nanomaterials loaded with adsorbents, because in this method agro waste is efficiently used for the preparation of adsorbent. It has been found that the loading of nanoparticles on their surface increases their removal capacity and also decrease air pollution which is caused by burning of agro waste (Moosavi et al. 2020 ;Hoang et al. 2024). In this way the problem of solid waste and waste water treatment can be solved together (Karić et al. 2022).

In this research, RS is explored as an agricultural waste to produce porous AC via the chemical activation process with 40% orthophosphoric acid at varying temperatures, residence times, and impregnation ratios. The qualities of the AC produced are greatly influenced by the physical and chemical characteristics of the starting material as well as the activation method employed. Furthermore, the impregnation ratio, activation period, and temperature all have an impact on the properties of the generated carbon (Heidarinejad et al. 2020).

2. Material and methods

2.1. Chemicals and raw materials

The following chemicals were bought from Sigma-Aldrich: ethanol, HCl, NaOH, and phosphoric acid (H_3PO_4). All reagents used were of analytical grade. The *Pusa Basmati* 1121 RS was collected from Ratia, Haryana, India. Measurements of ash content, pH, and moisture content in RS were conducted following the standards ASTMD2866-11, ASTMD3838-23, and ASTMD2867-23 methods respectively.

2.2. Synthesis of AC

As per the protocol followed by Singh et al. 2021, ACs were produced using a chemical activation method. Fig. 1 Shows the whole process of synthesis of AC from RS by using chemical activation method. The RS was thoroughly cleaned of contaminants using deionized water before being oven-dried for three hours at 80°C. A 0.2 mm sieve was used to screen the ground granular RS. Next, the powdered RS was placed in a tubular furnace with a 200 mL/min argon flow, set to 400°C for 60 minutes at a ramp rate of 20°C/min. The carbonization process for carbonaceous raw materials is typically carried out at temperatures below 800°C in the absence of oxygen (Gayathiri et al. 2022).The carbonized rice straw (CRS) was then soaked for 24 hours at room temperature in the phosphoric acid (H_3PO_4) activation reagent solution using (wt.%) ratios of 1:1, 1:2, 1:3, and 1:4. The pyrolysis was carried out in an argon atmosphere for 60, 90 and 120 minutes, respectively, at 500, 600 and 700°C. Various types of biomass were selected and analyzed under different production parameters, including chemical impregnation temperature and time, carbonization temperature, and heating rate. The study revealed that different biomass wastes require specific operating conditions for effective activation (Danish & Ahmad, 2018). Therefore, the selection of necessary parameters such as type of chemical activator, impregnation ratio and time, carbonization temperature and time involved in the synthesis of activated carbon is important to produce activated carbon with a high specific surface area (Gayathiri et al. 2022).Table 1 shows a list of ACs under diverse conditions. After being cleansed with deionized water, all of the samples were dried for 24 hours at 110°C.

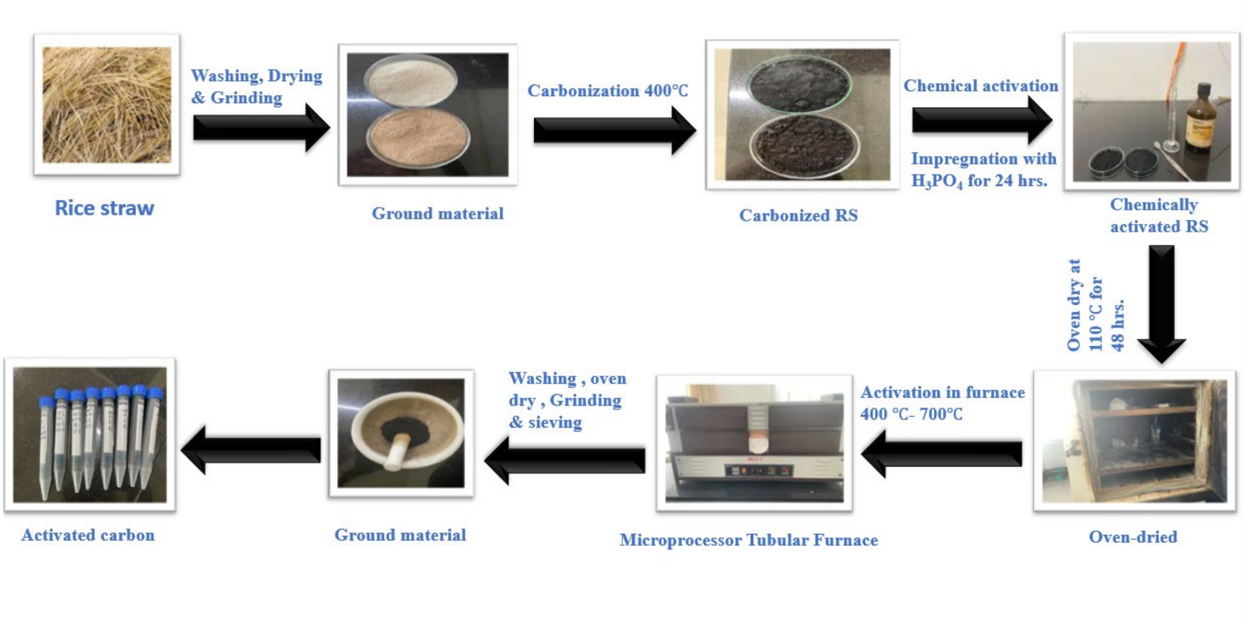


Fig. 1: Whole process of synthesis of AC by chemical activation method using RS agro-waste.

Table 1: List of ACs prepared by chemical activation method.

Sr. No.	Samples	Activation method
1.	RS 1	Chemical activation using 40% orthophosphoric acid at a 1:1 impregnation ratio, at 500 ˚C temperature, at 60 min time
2.	RS 2	Chemical activation using 40% orthophosphoric acid at a 1:2 impregnation ratio, at 500 ˚C temperature, at 60 min time
3.	RS 3	Chemical activation using 40% orthophosphoric acid at a 1:3 impregnation ratio, at 500 ˚C temperature, at 60 min time
4.	RS 4	Chemical activation using 40% orthophosphoric acid at a 1:4 impregnation ratio, at 500 ˚C temperature, at 60 min time
5.	RS 5	Chemical activation using 40% orthophosphoric acid at a 1:3 impregnation ratio, at 600 ˚C temperature, at 60 min time
6.	RS 6	Chemical activation using 40% orthophosphoric acid at a 1:3 impregnation ratio, at 700 ˚C temperature, at 60 min time
7.	RS 7	Chemical activation using 40% orthophosphoric acid at a 1:3 impregnation ratio, at 600 ˚C temperature, at 90 min time

8.	RS 8	Chemical activation using 40% orthophosphoric acid at a 1:3 impregnation ratio, at 600 ˚C temperature, at 120 min time

2.3. Characterization of AC

2.3.1. Determination of AC Yield

The precursors and ACs synthesized were weighed using a Mettler electronic balance. Every sample that was prepared had its carbon yield calculated in accordance with the protocol by Njewa et al. 2022. For every prepared sample, the yield of carbon was computed as

$$Y=\frac{Wf}{Wo}\times 100 \qquad (1)$$

Wf is the final weight of activated carbon when carbonization is complete, W0 is the initial weight of impregnated precursors, and Y is the carbon yield in this equation.

2.3.2. Determination of the Ash Content

The amount of ash was calculated using the ASTMD2866-11 technique. To find the percentage of ash content, a silica crucible was heated to 900°C for an hour in a furnace. After cooling in a desiccator, it was weighed once again. After that, 1 g of the AC sample was put into a crucible with a lid, and it was heated to 900°C for an hour. A silica crucible that had been heated in a furnace for an hour at 900°C was used to calculate the percentage of ash content. Once the crucible and its contents had cooled in the desiccator, they were weighed again. The weight of the incombustible residues determines the amount of ash in the AC samples.

The following equation was used to determine the percentage of ash content.

$$Ash\ content\ (\%) = \frac{C-D}{C-B}\times 100 \qquad (2)$$

Where B is the weight of the crucible in grams, C is the weight of the crucible with the sample in grams, and D is the weight of the crucible after weight loss in grams.

2.3.3.Determination of pH and Conductivity

The ASTMD3838-23 standard test protocol was used to measure the pH and conductivity was measured according to Rostamian et al. 2018. A beaker with one gram of weighed sample was filled with 100 milliliters of distilled water, and the mixture was stirred for an hour. After allowing the samples to stabilize, the pH was determined. The pH and conductivity were measured with a cyberscan (EUTECH) meter.

2.3.4.Determination of moisture content

The moisture content of AC samples was evaluated in accordance with ASTMD2867-23. After weighing, a small quantity of AC was put in a petri dish. It was evenly distributed throughout the plate. Then, for 1.5 hours, it was heated at a temperature between 105 and 110 ˚C. Throughout the heating period, the petri dish was kept open. After being heated, the petri dish was removed and left to cool in a desiccator. The dried sample was weighed once it was cooled (Singh et al. 2024, Ashtaputrey and Ashtaputrey; 2020)

$$M\ \% = \frac{100(B-F)}{B-G} \qquad (3)$$

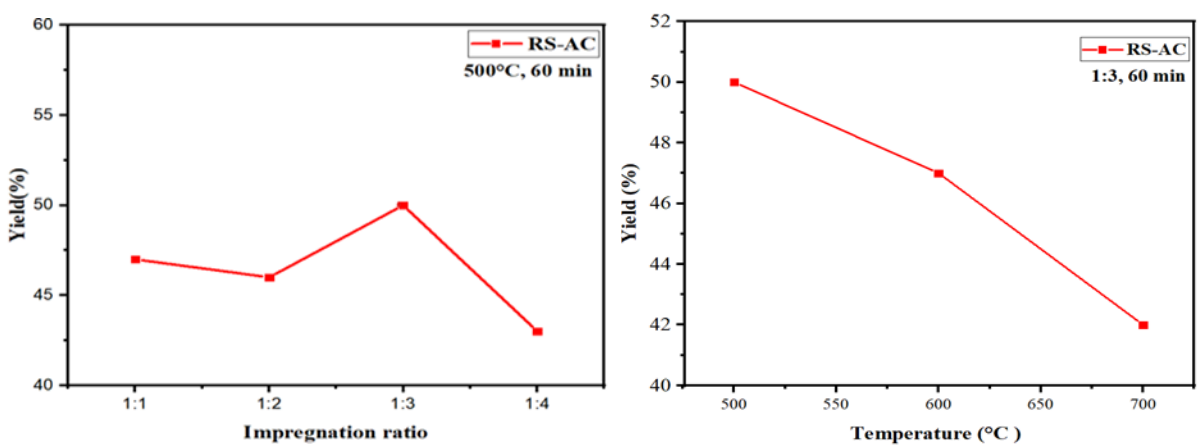
where G is the weight of the Petri dish, F is the weight of the Petri dish plus the dried sample, and B is the weight of the original sample plus the Petri dish.

3. Results and Discussions

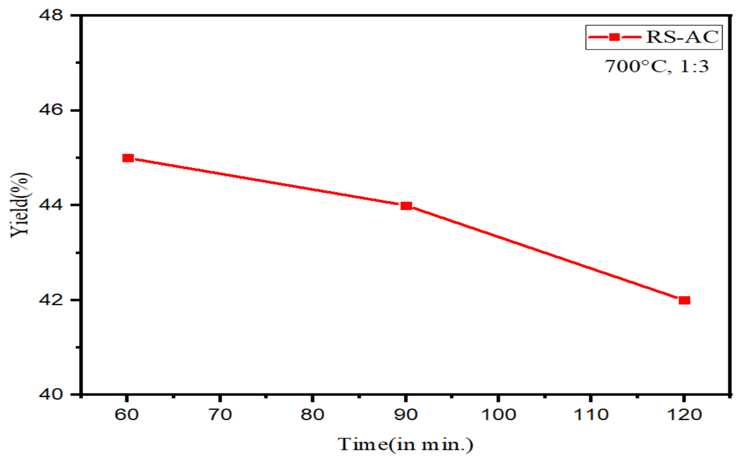
3.1. Physicochemical Properties

3.1.1. Carbon Yield

The prepared AC from the RS demonstrated that the percentage of carbon yield drops down as the impregnation ratios increase from 1:1 to 1: 4 (Figure 2). The reason behind the decline in carbon output is the release of volatiles from the sample. One possible explanation for the reduced carbon production at greater impregnation ratios could be that the surplus activation agents are enhancing carbon burn-off. The yields of ACs made from RS using various methods are significantly influenced by the activation temperature. Regardless of the precursor, raising the activation temperature usually results in a significant drop in the yield of AC. This is because during activation at high temperatures, pore opening, and biomass gasification cause the emission of volatile compounds (Gao et al. 2011). Furthermore, when carbonization times rise, AC yields are reduced. For instance, the AC output drops from 45% to 42% when the carbonization period was increased from 60 to 120 min at 600 °C and an impregnation ratio of 1:3. Results are concurrent with similar findings (Uner and Bayrak, 2018).



(a) (b)

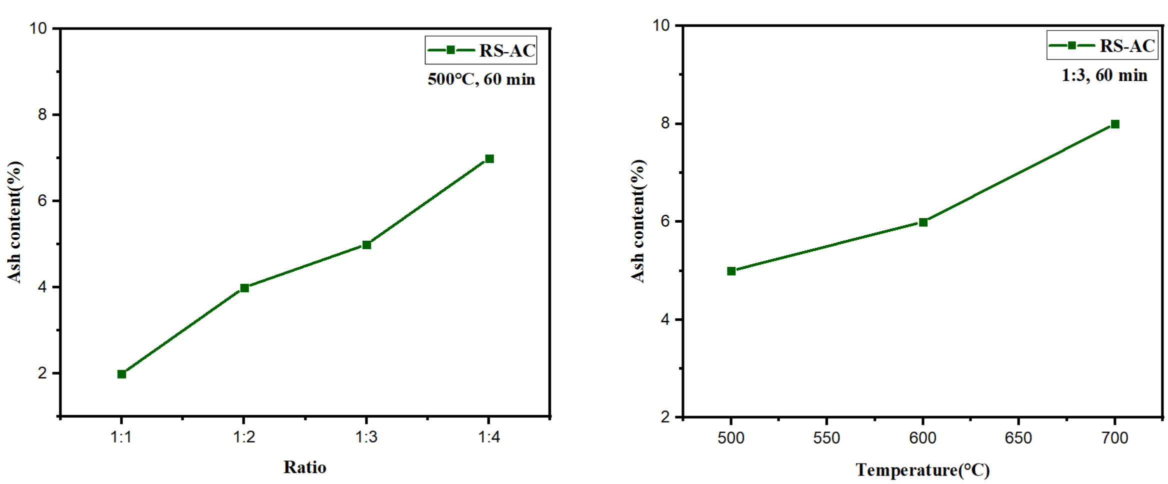


(c)

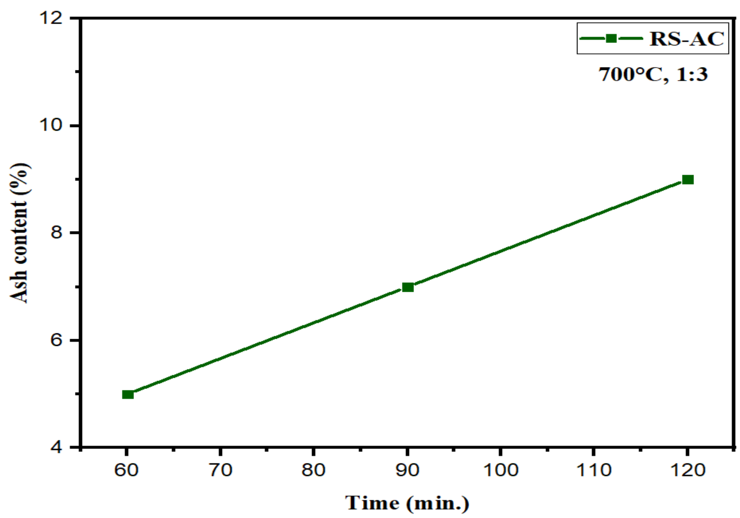
Fig. 2 The effect of the (a) impregnation ratio (b) activation temperature (c) time on carbon yield

3.1.2. Ash content

The findings demonstrated that raising the carbonization temperature from 500 to 700°C, impregnation ratios from 1: 1 to 1: 4, and time from 60 to 120 minutes for precursor, enhanced the ash content to produce the AC (Fig. 3). It was observed that the ash content of AC samples increased with increasing activation temperature. A rising temperature-induced high degree of biomass burn-off may be linked to the rise in ash content. High ash content degrades AC's mechanical strength and impairs its ability to absorb substances (Ashtaputrey and Ashtaputrey; 2020). However, several researchers have discovered comparable findings in the literature on ACs obtained from agricultural waste (Njewa et al. 2022).



(a) (b)



(c)

Fig. 3 The effect of the (a) impregnation ratio (b) activation temperature (c) time on ash content

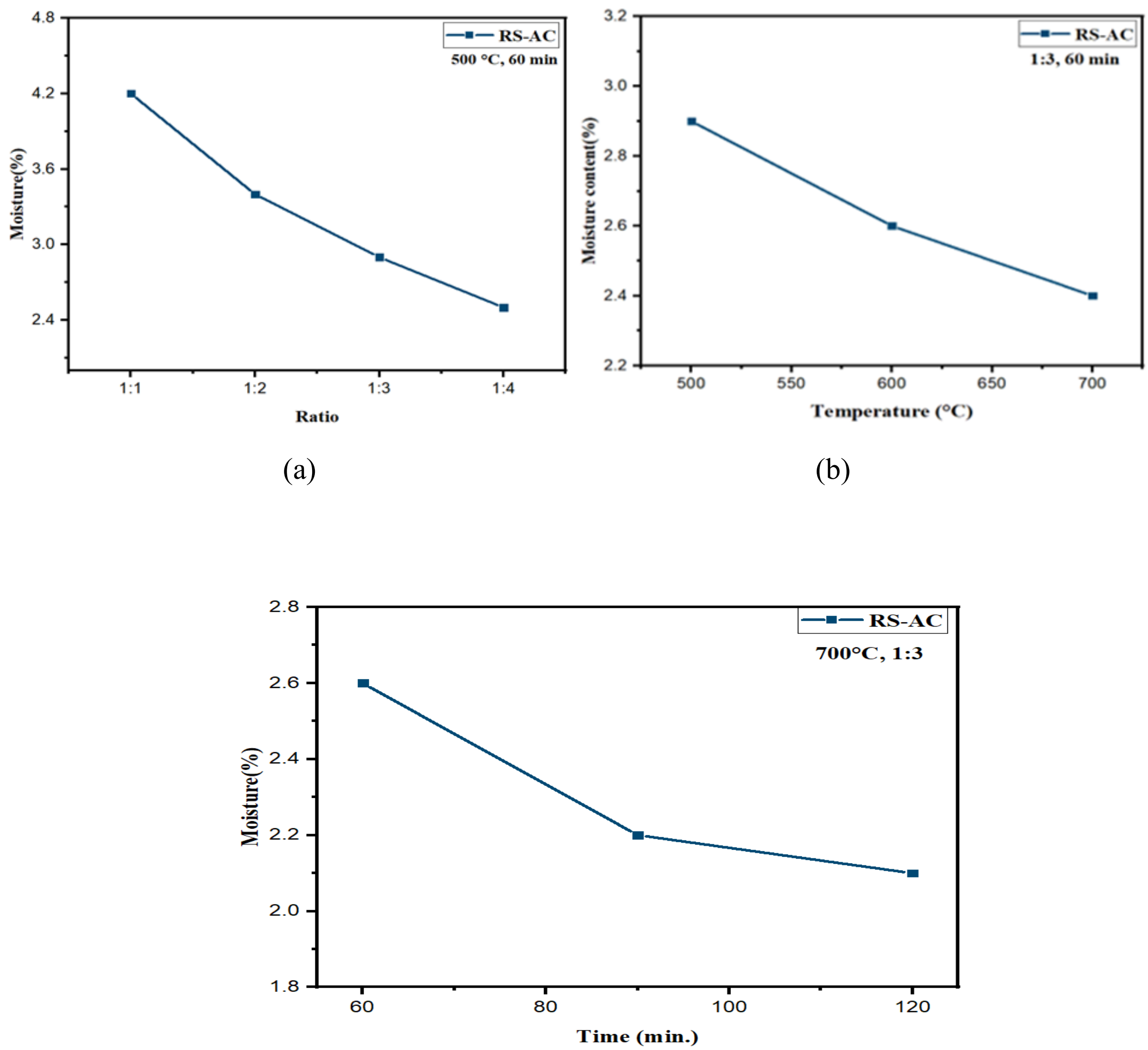
3.1.3. pH and conductivity

All of the synthesized ACs had somewhat acidic pH, ranging from 6 to 6.5. ACs having pH values ranging from 6 to 8 are found suitable for the majority of applications (Ashtaputrey and Ashtaputrey; 2020). As a result, the investigated ACs may be suitable for the majority of aqueous solution-adsorption applications.

The ACs had EC values ranging from 0.09 to 0.29 dS m^{-1}. The EC of all the ACs was low, indicating a low concentration of ions that are soluble in water in the produced samples (Rostamian et al. 2018).

3.1.4. Moisture content

To evaluate the prepared AC's appropriateness for adsorption research, its moisture content was measured. The findings show that when the impregnation ratios were increased from 1:1 to 1:4 and the carbonization temperature rose from 500°C to 700°C, the moisture content of the generated AC samples dropped. Additionally, as the activation time increases, the moisture content drops. This result could be linked to the elimination of volatile components, which reduces the carbon yield and moisture content of the produced AC samples (Njewa et al. 2022).



(a) (b)

(c)

Fig. 4 The effect of (a) impregnation ratio (b) activation temperature (c) time on moisture content

3.2. Analytical characterization

3.2.1. Fourier Transforms Infrared (FTIR)

FTIR spectroscopy was used to investigate samples of raw and activated RS to look for functional groups, as indicated in Fig. 5. Using infrared spectra, the cellulose, hemicellulose, and lignin structures of RS were assessed. The region of 4000 – 400 cm^{-1} was detected in an FTIR spectrum. The fingerprint region, indicative of the substance, was found to be below 1500 cm^{-1} (Malik et al. 2022). The FTIR spectrum's broadband at 3000–3500 cm^{-1} was typically associated with cellulose's hydroxyl group (O–H) stretching (Dhull et al. 2023) (Fig. 4a, b, c). Additionally, it was noted that the absorbance intensity dropped in the activated RS scenario. As the temperature rose from 500 to 700 °C during the pyrolysis process, the intensity of the peak at approximately 3427 cm^{-1} rapidly reduced from the biomass of RS, indicating that the organic O–H group broke down at higher temperatures. The band around 2920, 2854 and 1384 cm^{-1} was caused by the axial deformation of C–H bonds, which are present in lignin, hemicellulose, and cellulose; the C=C group has been seen at 1633 to 1638 cm^{-1}; C-O stretching caused the region of hemicellulose and cellulose between 1200 and 1100 cm^{-1} to attain its maximum value at 1170 cm^{-1}; peak appears in activated RS samples but is absent in raw RS samples (Kumari et al. 2022). It intensifies from 1:1 to 1:4. The absorption band at 1038 to 1055 cm^{-1} is C–O–C stretching of lignin, cellulose, and hemicellulose; bending vibrations of O-P-O are responsible for the tiny peaks at 672 to 676 cm^{-1} in activated RS. Bands at 460–520 cm^{-1} are allocated to Si–O–Si and Si–O–C cross-linking bonds. In raw RS samples, the peak is located at 471 cm^{-1} and is less intense. In activated RS samples, the peak was found at 493–494 cm^{-1} and becomes more intense from 1:1 to 1:4 (Borges et. al. 2023, El-Gawad et. al. 2023, Sakhiya et. al. 2023, Singh et. al. 2022, Phoung & Loc; 2022).

3.2.2. X-ray diffraction (XRD)

The X-ray diffraction method is a useful technique for comprehending the structure and crystallinity of activated RS and RS biomass. The raw biomass and activated RS results of the XRD spectra are shown in Fig. 6. The two sharp, fine peaks at 2θ values around 22° and 24° proved the presence of crystalline structure in the biomass caused by the cellulose. The raw RS exhibits a single, less intense peak at 22°, while RS1, RS2, RS3, and RS4 show distinct peaks at 15°, 23°, 24°, 26°, and 27°. In contrast, RS5, RS6, RS7, and RS8 display peaks at 13°, 14°, 18°, 22°, 24°, 26° and 27°. Pyrolysis of the RS biomass resulted in the breaking of the crystallinity structure's distinct, narrow peaks and the pyrolysis-produced biochars have a larger, less intense peak. The cellulose crystalline structure of the biomass persisted in the biochar despite partial degradation at low temperatures. These peaks, however, were not stable and entirely disappeared as the pyrolysis temperature increased. So, it may be concluded that the biochar was found amorphous based on the XRD spectra (Biswas et. al. 2024).

3.3.3. SEM-EDX

SEM was used to analyze the surface morphology of raw RS and activated RS at different magnifications, as illustrated in Fig. 7. The surface of the raw material (RS) was rougher, less porous, and has some cracks (Sakhiya et al. 2023). The morphological structure of the activated RS differs significantly as a result of the activation process (Abd-Elhamid et al. 2020). Rice straw activation releases volatile materials, creating a compact and porous structure (Sakhiya et al. 2023). The activated RS has an average particle size of 4 nm, indicating that the material falls within the mesoporous range.

Figure 7 displays EDX (energy dispersive X-rays) spectra of Raw and Activated RS at 700°C, 1:3, and 90 minutes, revealing chemical contents. The composition of raw RS is primarily organic as evidenced by its high C (47.5%) and O (46.8%) content with varying amounts of inorganic (K (0.7%) and Cl (0.3%)) material. Carbonization of rice straw in an inert atmosphere resulted in activated RS, which was validated by increasing the carbon content (61.4%) (Abd-Elhamid et al. 2020). A decrease in oxygen concentration signifies the elimination of carboxylic functional groups and the development of aromatic ring structures with activation (Qin et al. 2020). This is caused by the dissipation of hydrogen or oxygen functional groups (Park et al. 2019).

4. Conclusion

From a waste agricultural product rice straw, an affordable, effective, and environment-friendly activated adsorbent was synthesized. The experimental results demonstrate that the present method is very effective in producing porous carbon particles. To determine the ideal conditions for the greatest surface area and micropores, an H_3PO_4 chemical activation was done at various activation temperatures and times. It has been noted that the physicochemical and structural properties of the biochar vary with variations in the impregnation ratio, pyrolysis temperature, and time. The oxygen and hydrogen content dropped and a greater degree of carbonization took place at high temperatures. It is also feasible to control the porosity by adjusting the temperature, time, and amount of activation agent orthophosphoric acid. The product's capacity of adsorption is directly impacted by changes in porosity. The optimal conditions of activated RS were 700°C temperature, a ratio of 1:3, and a time duration of 90 minutes. Overall, activated RS is a carbon-rich substance, including various applications that is the treatment of wastewater, the manufacturing of super-capacitors, the fertilization of soil, and catalytic support. The current research shows the potential of rice straw utilization in the synthesis of good quality activated carbon for application in addition to a decrease in rice straw burning which leads to a decrease in air pollution and improved soil health and farmer’s economic conditions.

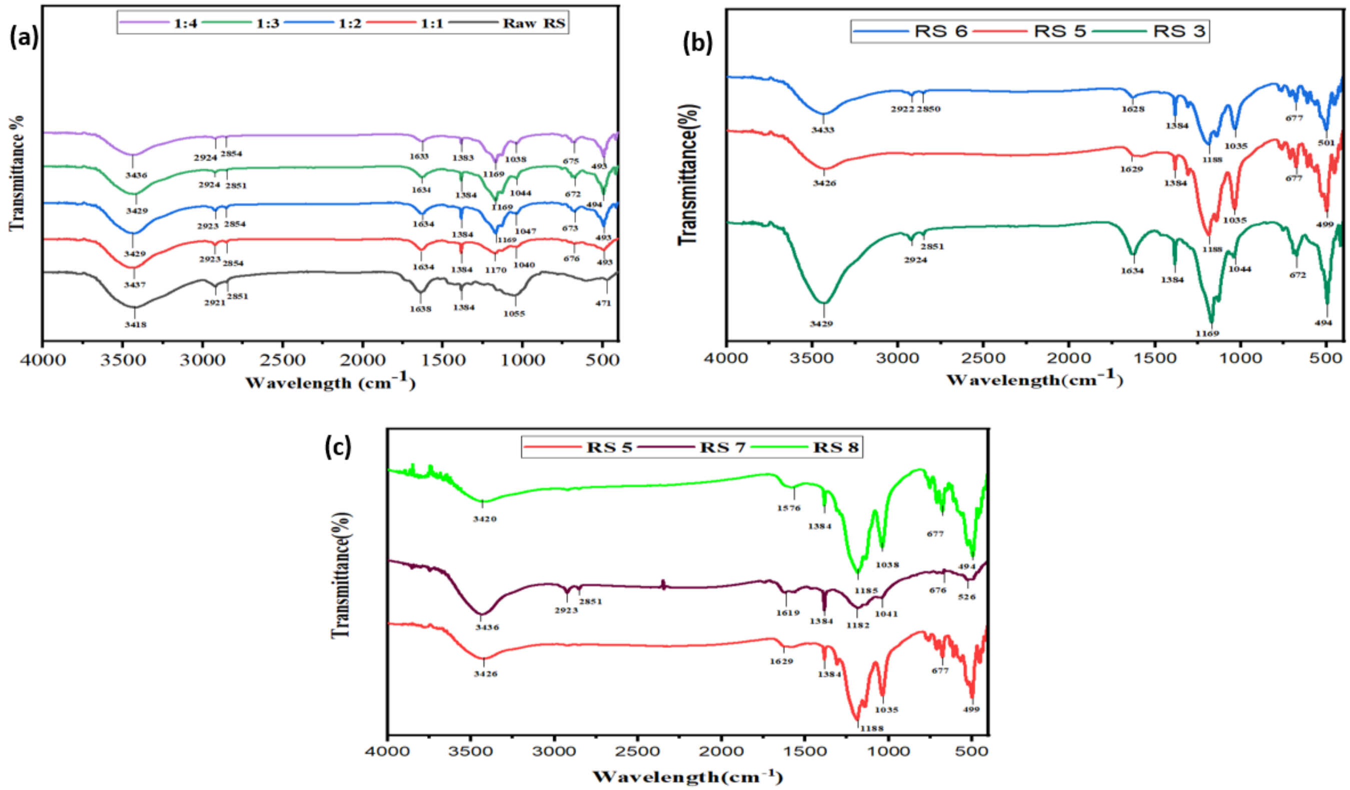


Fig. 5 FTIR analysis at different conditions (a) impregnation ratio (b) activation temperature (c) time

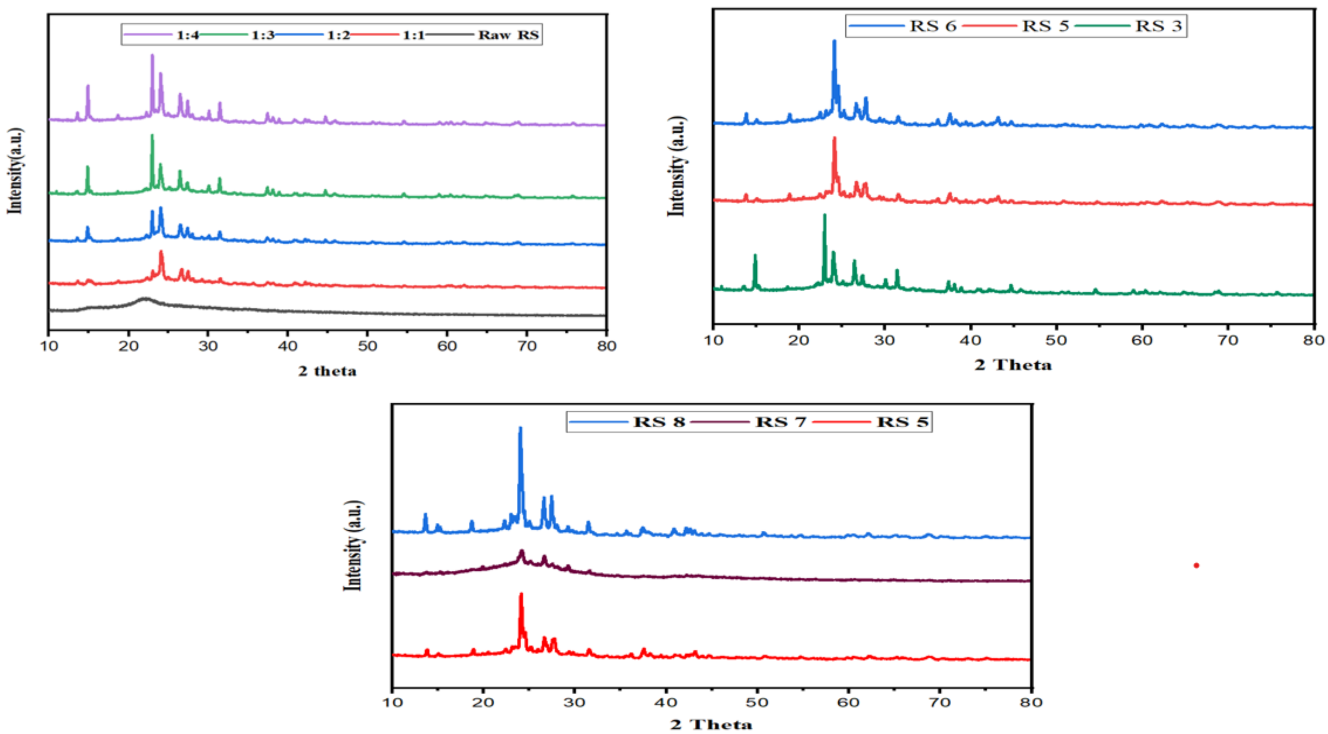


Fig. 6 XRD pattern at diverse conditions (a) impregnation ratio (b) activation temperature (c) time

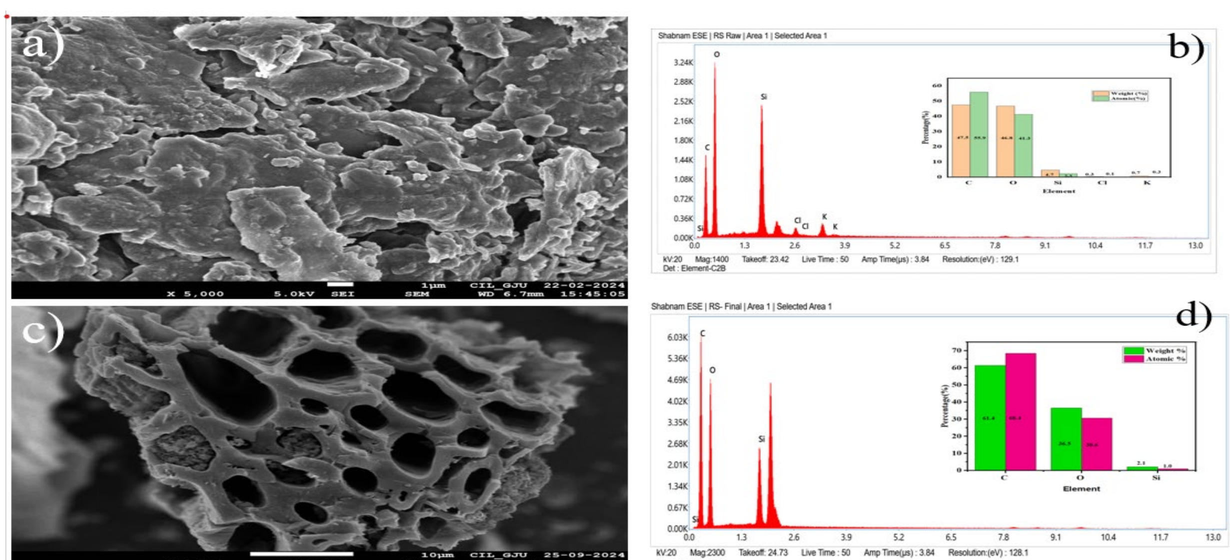


Fig. 7 SEM micrograph of different adsorbents (a) Raw RS (b) EDX spectrum of Raw RS (c) Activated carbon (d) EDX spectrum of activated carbon

Table 2: Comparison of the characteristics of the activated carbons with reported values in the literature.

Precursor	Activator	Activation Temp. (°C)	Time (min.)	Carbon Yield (%)	Ash content(%)	Moisture content (%)	XRD	FTIR	FESEM EDX	Reference
Coffee Grounds	**H_3PO_4**	**500**	**60**	**40 ± 3.4**	**35 ± 8**	**---**	**---**	**C-H, C-C, C=O, C=C,**	**mesoporous**	**Ferraz et al. 2019**
Almond shell	**H_3PO_4**	**600**	**---**	**---**	**---**	**---**	**---**	**COO−, C=O, C-H,**	**Microporous surface**	**Rai et al. 2018**
Rice husk	**NaOH**	**650**	**---**	**---**	**---**	**---**	**Amorphous**	**---**	**Mesoporous surface**	**Mullick et al. 2018**
Wood Apple Fruit Shell	**$ZnCl_2$**	**500**	**60**	**---**	**3.82**	**7.75**	**---**	**---**	**honey comb like structure.**	**Ashtaputrey & Ashtaputrey, 2020**
Rice Husk	**H_3PO_4**	**500**	**90**	**41**	**30.8**	**4:1 ± 0:18**	**Amorphous**	**C=O, OH, C-O, C≡C**	**Microporous**	**Njewa et al. 2022**
Potato Peels	**H_3PO_4**	**500**	**90**	**47**	**33.3**	**4:4 ± 0:24**	**Amorphous**	**C≡C, C=O, C-O, OH**	**Microporous**	**Njewa et al. 2022**
Rice Straw	**H_3PO_4**	**700**	**90**	**44**	**7**	**2.2**	**Amorphous**	**C≡C, C-O-C, C-O, OH, Si–O–C**	**Mesoporous**	**This study**

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