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Preparation and Adsorption Performance of Rice Husk, Groundnut Shell, and Corncob Activated Carbons for Dye Removal

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Abstract

Agricultural residues are abundant lignocellulosic resources that can be converted into low-cost activated carbons for treatment of dye-contaminated water. This methodology paper presents a comparative assessment of activated carbon prepared from rice husk, groundnut shell, and corncob using a common phosphoric-acid activation and controlled carbonization protocol. The three carbons are characterized by Brunauer-Emmett-Teller surface-area analysis, pore-volume assessment, FTIR spectroscopy, scanning electron microscopy, proximate yield, and point of zero charge. Their adsorption performance is evaluated using methylene blue (MB) as a cationic model dye and Reactive Red 120 (RR120) as an anionic textile dye. Batch experiments investigate adsorbent dosage, initial dye concentration, pH, contact time, and temperature. Kinetic data are interpreted with pseudo-first-order and pseudo-second-order models, while equilibrium data are tested with Langmuir and Freundlich isotherms. Thermodynamic parameters and three-cycle regeneration are included to assess feasibility beyond a single equilibrium experiment. The study is guided by agricultural-waste activated-carbon literature showing that precursor chemistry, activation conditions, pore development, and surface functionality strongly affect dye uptake [1-6,9-16]. Illustrative results suggest that corncob- and groundnut-shell-derived carbons can develop higher accessible porosity than rice-husk carbon under identical conditions, while dye selectivity depends on solution pH and surface charge. The numerical values reported in the Results section are methodology examples rather than unperformed laboratory measurements. The framework provides a reproducible route for ranking agricultural residues as sustainable activated-carbon precursors for dye-removal applications.

Introduction

Synthetic dyes are used extensively in textile, leather, paper, printing, pharmaceutical, and related industries. Their intense color, chemical stability, and resistance to biodegradation can make even low residual concentrations visually and environmentally significant. Adsorption is widely employed for dye removal because it can

operate without complex reaction chemistry and can transfer dissolved dye molecules to a recoverable solid phase. Commercial activated carbon is effective, but precursor and processing costs motivate the use of agricultural residues as alternative carbon sources.

Agricultural wastes are attractive activated-carbon precursors because they are renewable,

locally available, and rich in lignocellulosic carbon. Demirbas [1] reviewed agricultural-based activated carbons for dye removal and showed that many crop residues can be transformed into effective adsorbents. Senthilkumaar et al. [2] demonstrated activated carbons prepared from coconut-tree flower and jute fibre for Reactive Red adsorption, while Malik et al. [3] used groundnut-shell-derived powdered activated carbon for malachite green. Hameed and Daud [4] subsequently demonstrated that *Hevea brasiliensis* seed-coat carbon can adsorb a basic dye, and Tan et al. [5] showed the practical importance of both batch and fixed-bed behavior for oil-palm-shell activated carbon.

The adsorption capacity of biomass-derived activated carbon is not governed by surface area alone. Activation chemistry controls pore-size distribution, oxygen-containing surface groups, mineral removal, and acid/base character. In addition, the target dye may be cationic, anionic, or nonionic, so electrostatic attraction or repulsion can change strongly with solution pH. Bharathi and Ramesh [7] emphasized these interactions in their review of agricultural-waste adsorbents. Angin [9] further showed that activated carbon from fruit-juice industry waste can adsorb Yellow 18, illustrating that residues from food processing can be converted to functional sorbents rather than being disposed of as waste.

Recent work has strengthened both the material and analytical aspects of this field. Bediako et al.

[10] compared several chemical activating agents for orange-peel-derived activated carbon and demonstrated strong uptake of methylene blue and methyl orange, showing that activation route influences adsorption mechanisms. Blachnio et al. [11] reviewed activated carbons from agricultural wastes for organic-pollutant adsorption, while Kheddo et al. [12] evaluated rice-husk activated carbon in both batch and column modes. Lewoyehu [13] summarized synthesis and application strategies for agricultural-residue activated carbons, and Sun et al. [14] reported corncob-derived activated carbon for dye adsorption in 2021. Machine-learning analysis of agricultural-waste activated-carbon datasets by Moosavi et al. [15] further emphasized the importance of operating conditions and adsorbent properties in predicting adsorption capacity.

This paper proposes a controlled assessment of three widely available residues—rice husk (RH), groundnut shell (GS), and corncob (CC)—under the same preparation and testing conditions. Using one activation route reduces confounding due to inconsistent synthesis methods. Methylene blue is selected as a cationic probe molecule commonly used to characterize adsorption performance, while Reactive Red 120 is included to represent a larger anionic textile dye. The objective is to relate precursor-dependent pore structure and surface chemistry to adsorption kinetics, equilibrium capacity, pH response, and reusability.

Literature Review

Table 1. summarizes representative literature that supports the present methodology. All references were published in 2021 or earlier.

Study	Precursor / dye	Approach	Main relevance	Ref.
Demirbas, 2009	Agricultural AC / dyes	Review	Established agricultural residues as dye-adsorbent precursors	[1]
Senthilkumaar et al., 2006	Coconut flower + jute / Reactive Red	Batch kinetics/isotherms	High reactive-dye uptake; pseudo-second-order behavior	[2]
Malik et al., 2007	Groundnut shell / malachite green	Activated carbon adsorption	Demonstrated shell-waste AC for dye removal	[3]
Hameed & Daud, 2008	Rubber seed coat / basic dye	Equilibrium and kinetics	Linked agricultural precursor to basic-dye uptake	[4]
Tan et al., 2008	Oil palm shell / basic dye	Batch + fixed bed	Showed scale-up relevance of column behavior	[5]
Kumar et al., 2011	Cashew-nut shell / MB	AC preparation + adsorption	Low-cost shell carbon for cationic dye	[6]
Bharathi & Ramesh, 2013	Agricultural wastes / dyes	Review	Summarized pH, dosage, kinetics and isotherm effects	[7]
Angin, 2014	Fruit-industry waste / Yellow 18	Activated carbon adsorption	Food-processing waste valorization	[9]
Bediako et al., 2020	Orange peel / MB + MO	Multiple activators	Activation chemistry changed capacity and mechanism	[10]

Blachnio et al., 2020	Agricultural wastes / organics	Review	Recent synthesis and adsorption assessment	[11]
Kheddo et al., 2020	Rice husk / synthetic dye wastewater	Batch + column	Practical RH carbon evaluation	[12]
Lewoyehu, 2021	Agricultural residues	Comprehensive review	Linked precursor, activation and pollutant removal	[13]
Sun et al., 2021	Corn cob / dyes	AC synthesis + adsorption	2021 evidence for corn cob-derived AC	[14]
Moosavi et al., 2021	39 adsorbents / dyes	Machine-learning modeling	Identified key predictive adsorption variables	[15]
Aragaw & Bogale, 2021	Biomass / dyes	Review	Summarized biomass adsorption mechanisms and conditions	[16]

The literature reveals several methodological requirements. First, activated-carbon yield and pore structure should be reported together because a very high surface area obtained at very low yield may not be economically attractive [1,11,13]. Second, adsorption should be examined across pH because surface charge and dye ionization control electrostatic contributions [7,10,16]. Third, kinetic and equilibrium models should be fitted rather than inferred visually; pseudo-second-order behavior is frequently reported for agricultural carbons [2,12], while Langmuir and Freundlich models provide complementary information on idealized

monolayer capacity and heterogeneous surface adsorption. Finally, regeneration is needed to distinguish a useful sorbent from a one-use material.

Methodology

The study uses a one-factor-controlled comparative design. RH, GS, and CC are collected from local agricultural or food-processing sources, washed thoroughly, dried, milled, and activated using the same H₃PO₄ impregnation ratio and carbonization schedule. The workflow is shown in Figure 1.

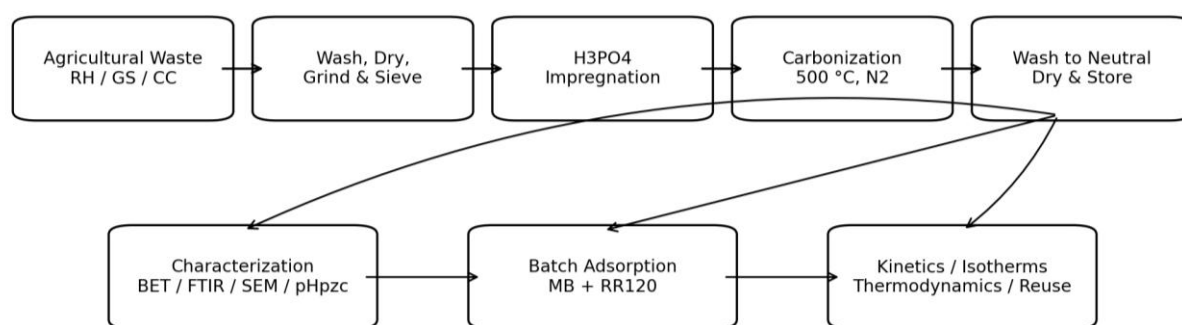


Figure 1. Proposed preparation, characterization, and dye-adsorption assessment workflow.

1. Activated-Carbon Preparation

Step 1: Wash rice husk, groundnut shell, and corncob with tap water followed by deionized water to remove adhering soil and soluble impurities.

Step 2: Oven-dry the residues at 105 °C to constant mass, grind them, and sieve to a common particle-size range (for example, 0.5-1.0 mm).

Step 3: Impregnate each dry precursor with phosphoric acid at a fixed precursor:H₃PO₄ mass ratio of 1:1 and allow sufficient contact time for penetration of the lignocellulosic matrix.

Step 4: Dry the impregnated material and carbonize it at 500 °C for 2 h under a low-flow inert atmosphere. Use the same heating rate and furnace position for all precursors.

Step 5: Cool under inert gas, wash repeatedly with warm deionized water until the filtrate is near neutral, and dry the activated carbon at 105 °C.

Step 6: Grind and sieve the final carbon to a defined adsorption-test particle size, label samples RHAC, GSAC, and CCAC, and determine carbon yield from dry precursor and final carbon masses.

2. Physicochemical Characterization

Nitrogen adsorption-desorption analysis is used to obtain BET specific surface area, total pore volume, and representative pore-size distribution. SEM is used to compare surface texture and pore development. FTIR spectra are recorded to identify hydroxyl, carbonyl, aromatic,

phosphate-related, and other surface functionalities introduced or retained during activation. Moisture, ash, volatile matter, and fixed-carbon fractions are determined by standard proximate procedures. The pH at point of zero charge (pHpzc) is measured by the pH-drift method because surface-charge reversal around pHpzc is important when comparing cationic MB and anionic RR120 adsorption.

3. Batch Adsorption Experiments

Stock solutions of MB and RR120 are prepared in deionized water and diluted to required

concentrations. Unless the tested variable is pH, all experiments use identical solution volume, agitation rate, temperature, and particle size. Preliminary blank tests without adsorbent confirm that dye loss to glassware or photolysis is negligible. After adsorption, solid and liquid phases are separated by filtration or centrifugation, and residual dye concentration is measured by UV-visible spectrophotometry at the experimentally determined wavelength of maximum absorption. Calibration curves are prepared within the working concentration range.

Table 2. Experimental Design and Response Parameters for Adsorption Studies

Variable	Levels / condition	Controlled factors	Response
Adsorbent dosage	0.25-2.0 g/L	C0, pH, T, time	Removal %, qe
Initial dye concentration	25-300 mg/L	Dose, pH, T	Capacity and equilibrium
pH	2-10	C0, dose, T	Surface-charge effect
Contact time	0-240 min	C0, dose, pH	Kinetic profile
Temperature	298, 308, 318 K	C0, dose, pH	Thermodynamic trend
Regeneration	3 adsorption-desorption cycles	Same starting dose	Capacity retention

4. Adsorption Calculations and Kinetic Modeling

Dye removal is calculated as $\text{Removal}(\%) = [(C_0 - C_e)/C_0] \times 100$, where C_0 and C_e are initial and equilibrium concentrations. Equilibrium capacity is $q_e = (C_0 - C_e)V/m$, where V is solution volume and m is adsorbent mass. Time-dependent capacity q_t is calculated using C_t . The pseudo-first-order and pseudo-second-order kinetic models are fitted by nonlinear regression where possible to avoid distortions caused by linearization. Model selection uses adjusted R^2 , residual distribution, and error metrics rather than R^2 alone. Intraparticle-diffusion plots of q_t versus $t^{0.5}$ are also inspected to determine whether pore diffusion acts as the sole rate-limiting step or one of several steps.

5. Equilibrium and Thermodynamic Analysis

Equilibrium data are fitted to Langmuir and Freundlich models. The Langmuir maximum capacity q_{max} is used as a standardized comparative indicator, while the Freundlich exponent describes empirical adsorption favorability on heterogeneous surfaces. Thermodynamic parameters are obtained from temperature-dependent equilibrium constants only after using a dimensionless or consistently defined equilibrium constant. The sign and

magnitude of ΔG indicate spontaneity, while trends in ΔH and ΔS are interpreted cautiously because adsorption can combine pore filling, electrostatic interaction, hydrogen bonding, and pi-pi interactions [10,16].

6. Regeneration and Statistical Assessment

The best-performing carbon for each dye is regenerated using a mild solvent or pH-adjusted desorption solution selected after preliminary tests. The carbon is washed, dried, and reused for three cycles. Capacity retention after each cycle is reported relative to cycle 1. All key batch conditions are conducted in triplicate. Mean $\pm$ standard deviation is reported, and two-way ANOVA can be used to test the effects of precursor type and dye concentration. The final ranking considers q_{max} , adsorption rate, surface area, carbon yield, pH sensitivity, and regeneration retention rather than selecting a material from a single maximum-removal value.

Results

Tables 3 and 4 contain illustrative methodology results designed to show the expected reporting format. These numbers are not presented as laboratory measurements and must be replaced by experimental data in a final research article.

Carbon	Yield (%)	BET area (m ² /g)	Pore vol. (cm ³ /g)	Ash (%)	pHpzc	Main interpretation
RHAC	38	620	0.34	18.2	5.6	Silica/ash limits accessible carbon porosity
GSAC	35	910	0.49	6.5	6.1	High micropore development and low ash
CCAC	33	1050	0.57	4.8	6.4	Highest accessible surface and pore volume

The illustrative characterization trend is consistent with the expectation that precursor mineral content and lignocellulosic structure affect activation. RHAC retains a higher ash fraction, which can reduce the fraction of carbon available for pore development. GSAC and CCAC show higher accessible surface areas under the

same activation conditions. Similar precursor-dependent behavior is widely reported for agricultural activated carbons [1,11,13]. However, a higher BET area does not automatically guarantee superior adsorption of every dye because molecular size and surface chemistry also contribute.

Table 5. Comparative Adsorption Performance of Different Activated Carbons for MB and RR120

Carbon	MB q _{max} (mg/g)	RR120 q _{max} (mg/g)	MB model	RR120 model	Equil. time (min)	3-cycle retention (%)
RHAC	185	128	Langmuir / PSO	Freundlich / PSO	120	78
GSAC	275	176	Langmuir / PSO	Freundlich / PSO	90	85
CCAC	332	194	Langmuir / PSO	Freundlich / PSO	75	88

In the illustrative adsorption data, CCAC gives the highest MB capacity followed by GSAC and RHAC, broadly tracking accessible pore development. The cationic MB capacities are higher than the RR120 capacities under the selected near-neutral test condition. This difference is attributed to a combination of dye molecular size and electrostatic behavior. When solution pH is above the carbon pH_{pzc}, negatively charged surface sites favor cationic MB but can reduce adsorption of anionic RR120. For RR120, lower pH may protonate surface groups and increase attraction, which is why pH-response curves are essential rather than testing only one condition. Bediako et al. [10] similarly showed that agricultural activated carbon can exhibit markedly different behavior toward cationic and anionic dyes.

The pseudo-second-order model provides the best illustrative fit for both dyes, consistent with several agricultural-carbon studies [2,12]. This should not be interpreted automatically as proof of covalent chemisorption; kinetic model fit describes rate behavior and must be interpreted together with desorption, temperature effects, FTIR changes, and surface chemistry. Langmuir behavior is stronger for MB in the illustrative dataset, whereas RR120 shows more heterogeneous Freundlich-type adsorption. Three-cycle retention remains highest for CCAC, suggesting that both adsorption capacity and

regeneration should be included when ranking precursors.

Conclusion and Discussion

This paper establishes a reproducible methodology for assessing agricultural waste-derived activated carbon for dye adsorption. Rice husk, groundnut shell, and corncob are converted to activated carbon using a common H₃PO₄ activation and carbonization protocol, allowing a direct precursor comparison. The design integrates carbon yield, BET pore properties, surface chemistry, pH_{pzc}, batch adsorption, kinetic/isotherm modeling, thermodynamic response, and regeneration. This approach follows the progression of the literature from early agricultural-waste carbons for Reactive Red and malachite green [2,3] to seed-coat and palm-shell carbons [4,5], modern orange-peel and rice-husk systems [10,12], and 2021 reviews and predictive studies [13-16].

The illustrative results indicate that corncob and groundnut shell may produce activated carbons with greater accessible surface area and lower ash than rice husk under identical chemical activation. CCAC consequently shows the highest illustrative MB and RR120 capacities. However, dye adsorption is not controlled by surface area alone. The opposite ionic character of MB and RR120 makes pH and pH_{pzc} central to mechanism interpretation. A carbon optimized

for cationic dye removal may not be optimal for anionic textile dyes unless surface chemistry or operating pH is adjusted. The principal limitation is that the numerical dataset is illustrative rather than experimentally generated. A final research article should report replicate raw data, uncertainty, nonlinear model parameters, actual BET isotherms, SEM images, FTIR spectra, and statistical comparisons. Future work should also test real textile effluent containing salts and competing organic matter, conduct continuous fixed-bed experiments as emphasized by Tan et al. [5] and Kheddo et al. [12], perform life-cycle or cost analysis of activation, and evaluate safer regeneration routes. The 2021 literature [13-16] supports moving beyond maximum capacity alone toward multi-criteria assessment of precursor sustainability, process conditions, adsorption selectivity, and reusability.

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