



Preparation and Evaluation of Activated Carbon from Agricultural Waste for Wastewater Treatment

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Abstract

Agricultural residues are inexpensive lignocellulosic precursors for producing porous activated carbon, allowing waste valorization and low-cost wastewater treatment in a single process. This methodology paper presents a reproducible route for preparing activated carbon from rice husk, sugarcane bagasse and coconut shell by phosphoric-acid-assisted carbonization, followed by physicochemical characterization and adsorption evaluation. The proposed procedure includes precursor cleaning, size reduction, impregnation with H₃PO₄, controlled carbonization at 450 °C, neutral washing, drying and particle-size classification. Product quality is assessed by yield, moisture, ash, iodine number, pH, surface functional groups, morphology and, where available, BET surface area. Adsorptive performance is evaluated with methylene blue as a model wastewater contaminant using batch tests, kinetic fitting and Langmuir/Freundlich equilibrium models. The design also includes regeneration over repeated cycles. The approach is based on activated-carbon studies reported up to 2015 and is suitable for laboratory-scale comparison of agricultural wastes. Illustrative results show how precursor composition and activation conditions can influence carbon yield, porosity and pollutant uptake; these values are provided only to demonstrate data treatment and must be replaced by experimentally measured values in an actual study.

Introduction

Activated carbon (AC) is widely used in water and wastewater treatment because its highly developed pore network and chemically active surface can retain dyes, phenolic compounds, metals and other contaminants. The cost of commercial AC, however, can restrict routine use in small treatment systems. Agro-industrial residues offer an alternative carbon source because they are renewable, locally available and often require disposal. Reviews by Bhatnagar and Sillanpää (2010) and Alslaibi et al. (2013) show that agricultural by-products can be converted into effective adsorbents through physical or chemical activation, while the resulting surface area and pore structure depend

strongly on precursor composition, impregnation ratio, temperature and residence time.

A wide range of residues has been investigated. Oil-palm wood and shell produced porous carbons capable of methylene-blue removal (Ahmad et al., 2007; Tan et al., 2008), while oil-palm empty fruit bunches were optimized for phenol adsorption (Alam et al., 2009). Rice husk activated with ZnCl₂ has also shown substantial dye removal (Sharma and Uma, 2010). These studies demonstrate that both the agricultural precursor and activation route influence carbon yield, surface chemistry and adsorption capacity. The present methodology therefore compares

three commonly available wastes—rice husk, sugarcane bagasse and coconut shell—under the same H_3PO_4 activation protocol so that precursor effects can be assessed directly.

Literature Review

Chemical activation is frequently preferred at laboratory scale because activation and carbonization can occur at lower temperatures than many physical-activation routes. Prahas et al. (2008) produced jackfruit-peel AC with H_3PO_4 and reported that activation temperature and impregnation ratio substantially affected pore structure and surface chemistry. Foo and Hameed (2012a) similarly prepared coconut-husk AC and demonstrated that activation agent, impregnation ratio and energy input-controlled yield and adsorption behavior. Their orange-peel study also showed that K_2CO_3 -assisted activation could generate high surface area and strong methylene-blue uptake (Foo and Hameed, 2012b).

Adsorption performance has been evaluated with both organic and inorganic pollutants. Groundnut-hull AC was effective for eosin adsorption and followed equilibrium/kinetic

behavior that could be described using conventional adsorption models (Bello et al., 2012). Sugarcane-bagasse AC was reported as a low-cost adsorbent for cationic dyes (Farahani et al., 2011). Date-pit AC has been investigated for metal removal, including aluminum (Al-Muhtaseb et al., 2008), while a broader review of date-palm biomass concluded that such residues can adsorb dyes, metals and phenolic compounds (Ahmad et al., 2012).

Pore development is not governed by a single activation pathway. Corn-cob carbons prepared by chemical and physical activation displayed different sorption characteristics, emphasizing the need to characterize both surface chemistry and pore structure (Każmierczak et al., 2013). Across agricultural precursors, carbonization temperature, impregnation ratio and activating atmosphere determine fixed-carbon content, yield and adsorption capacity (Alslaibi et al., 2013). Consequently, an evaluation protocol should combine basic proximate tests, iodine number, microscopy/FTIR and an adsorption test rather than judging the material from yield alone.

Table 1. Selected pre-2016 studies supporting the proposed methodology.

Precursor / focus	Activation / evaluation	Key methodological implication
Jackfruit peel	H_3PO_4 ; BET and surface chemistry	Impregnation ratio and temperature control pore development (Prahas et al., 2008).
Rice husk	$ZnCl_2$; methylene blue	Agro-waste AC can be evaluated by adsorption capacity and process variables (Sharma and Uma, 2010).
Coconut / orange residues	Microwave chemical activation	Activation conditions strongly affect yield and adsorption performance (Foo and Hameed, 2012a,b).
Oil-palm residues	Physical/chemical activation; dye/phenol tests	Different lignocellulosic precursors generate different porosity and capacities (Ahmad et al., 2007; Alam et al., 2009).
Groundnut hull / bagasse	Batch dye adsorption	Kinetic and equilibrium models are appropriate for treatment evaluation (Bello et al., 2012; Farahani et al., 2011).

Research gap and methodological objective. Many reports evaluate a single precursor under different conditions, which makes direct precursor-to-precursor comparison difficult. The proposed study standardizes particle size, impregnation ratio, carbonization temperature and adsorption conditions for three wastes, and then compares carbon quality and wastewater-treatment performance using the same analytical criteria.

Methodology

1. Study Design and Materials

Three agricultural wastes—rice husk (RH), sugarcane bagasse (SB) and coconut shell (CS)—are collected from clean local sources. Analytical-grade phosphoric acid (H_3PO_4), hydrochloric acid, sodium hydroxide and methylene blue are used. Distilled/deionized water is used for washing and solution preparation. The experiment is conducted in triplicate for each precursor. Commercial activated carbon may be included as a benchmark control.

Step-by-Step Preparation of Activated Carbon

1. Pre-cleaning: Remove stones and foreign matter. Wash each residue repeatedly with tap water and finally with distilled water.
2. Drying and size reduction: Oven-dry at 105 °C to constant mass. Grind and sieve to approximately 0.5–1.0 mm.
3. Chemical impregnation: Mix dry precursor with 40–50% H₃PO₄ solution at a 1:1 dry precursor:acid mass ratio. Stir thoroughly and allow to impregnate for 12 h.
4. Carbonization/activation: Drain excess liquid, place the impregnated precursor in covered ceramic crucibles and heat in a muffle furnace to 450 °C. Maintain 450 °C for 60 min. Where inert gas is available, a low nitrogen flow is preferred.
5. Cooling and washing: Cool in a desiccator. Wash first with warm distilled water; use dilute NaOH only, if necessary, then continue washing until the filtrate reaches approximately pH 6.5–7.0.
6. Final drying and sizing: Dry at 105 °C for 12 h, grind gently and sieve to 150–300 µm for batch adsorption tests. Store in airtight containers labeled RHAC, SBAC and CSAC.
7. Yield calculation: Activated-carbon yield (%) = (dry mass of final AC / dry mass of initial precursor) × 100.



Figure 1. Stepwise preparation, characterization and wastewater-evaluation workflow.

Table 2. Controlled preparation variables for direct precursor comparison.

Variable	Proposed condition	Reason for control
Particle size	0.5–1.0 mm before activation	Reduces mass-transfer differences during impregnation.
H ₃ PO ₄ impregnation	1:1 (w/w), 12 h	Common chemical-activation basis for all precursors.
Carbonization	450 °C, 60 min	Promotes carbonization while limiting excessive burn-off.
Final adsorbent size	150–300 µm	Provides reproducible batch adsorption kinetics.

2. Characterization of the Prepared Carbon

Moisture and ash are determined gravimetrically using standard oven/furnace procedures. Bulk density and slurry pH are measured as routine quality indicators. Iodine number is used as a simple proxy for micropore-related adsorption. BET N₂ surface area is measured where instrumentation is available. FTIR spectra (approximately 4000–400 cm^{−1}) are recorded to identify O-containing functional groups and changes after activation, while SEM is used to observe pore opening and surface morphology. The combined characterization approach is consistent with agricultural-waste AC studies that link pore/surface properties to adsorption performance (Ahmad et al., 2007; Foo and Hameed, 2012a; Kaźmierczak et al., 2013).

3. Batch Wastewater-Treatment Test

Methylene blue (MB) is selected as a model-colored organic pollutant because it is widely used to compare activated carbons in the literature (Tan et al., 2008; Sharma and Uma, 2010). Prepare a 1000 mg/L stock solution and dilute to 25–200 mg/L. For the screening test, add 0.10 g AC to 100 mL of 50 mg/L MB in 250 mL flasks, adjust pH to 7.0, agitate at 150 rpm and 25 ± 2 °C, and collect samples at 0, 15, 30, 60, 90 and 120 min. After filtration/centrifugation, measure residual MB by UV-Vis spectrophotometry at the previously established λ_{max}.

Removal efficiency and equilibrium adsorption capacity are calculated as: Removal (%) = [(C₀ – C_t)/C₀] × 100; and q_e = [(C₀ – C_e)V]/m, where C₀, C_t and C_e are dye concentrations (mg/L), V is solution volume (L), and m is dry adsorbent mass (g). Adsorbent dose (0.25–2.0 g/L), initial

concentration (25–200 mg/L) and pH (3–11) are then varied one factor at a time while other variables remain fixed. All tests are performed in triplicate with an adsorbate blank.

4. Kinetics, Equilibrium and Regeneration

Time-dependent data are fitted to pseudo-first-order and pseudo-second-order kinetic models. Equilibrium data obtained at different initial concentrations are fitted to Langmuir and Freundlich isotherms. Model selection is based on R^2 together with agreement between experimental and calculated q_e . These models are routinely used in agricultural-waste AC studies (Tan et al., 2008; Bello et al., 2012). For regeneration, MB-loaded AC is washed and desorbed with dilute ethanol/water or another validated eluent, dried, and reused for at least three adsorption cycles. Regeneration efficiency

is reported as retained removal capacity relative to cycle 1.

5. Quality Control and Statistical Analysis

Each preparation and adsorption experiment is performed in triplicate. Results are reported as mean $\pm$ standard deviation. One-way ANOVA followed by an appropriate post-hoc comparison ($p < 0.05$) is used to compare RHAC, SBAC and CSAC. Calibration curves must meet a predefined linearity criterion (e.g., $R^2 \geq 0.995$). Mass balance, blank samples and duplicate instrumental readings are used to identify analytical error. The protocol intentionally standardizes preparation conditions so that observed differences can be attributed primarily to precursor composition rather than inconsistent processing.

Table 3. Evaluation parameters and acceptance/interpretation criteria.

Parameter	Method	Interpretation
Carbon yield (%)	Gravimetric	Higher yield improves material efficiency but does not by itself prove adsorption quality.
Ash / moisture	Gravimetric	Lower ash and controlled moisture generally improve reproducibility.
Iodine number	Iodometric adsorption test	Higher value indicates greater small-pore adsorption potential.
BET / SEM / FTIR	Instrumental characterization	Confirms pore development, morphology and surface functional groups.
MB removal and q_e	UV-Vis batch adsorption	Primary wastewater-treatment performance indicators.
Regeneration	Repeated adsorption-desorption	Assesses practical reuse and loss of capacity.

Results

Important note: the numerical values below are an illustrative methodology-validation dataset, included only to demonstrate calculation,

Table 4. Illustrative physicochemical characteristics of activated carbons (mean $\pm$ SD, $n = 3$).

Parameter	RHAC	SBAC	CSAC
Yield (%)	38.6 $\pm$ 1.4	34.9 $\pm$ 1.2	31.7 $\pm$ 1.1
Ash (%)	12.8 $\pm$ 0.6	7.4 $\pm$ 0.4	4.8 $\pm$ 0.3
Iodine number (mg/g)	612 $\pm$ 18	735 $\pm$ 21	824 $\pm$ 25
BET surface area (m ² /g)	540 $\pm$ 24	710 $\pm$ 31	865 $\pm$ 35
pH (1% slurry)	6.7 $\pm$ 0.1	6.6 $\pm$ 0.1	6.8 $\pm$ 0.1

tabulation and interpretation. They are not presented as measured laboratory data and should be replaced by actual experimental observations in a completed research study.

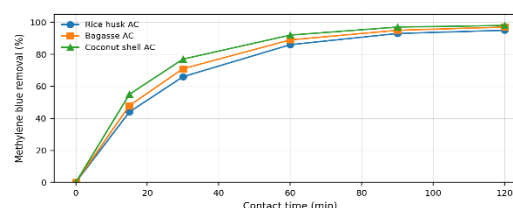


Figure 2. Illustrative contact-time profile for methylene-blue removal by the three activated carbons.

Table 5. Illustrative adsorption-model and regeneration results.

Metric	RHAC	SBAC	CSAC
Equilibrium MB removal (%)	95.0	97.0	98.0
Langmuir q_{max} (mg/g)	118	156	184
Langmuir R^2	0.982	0.991	0.995

Pseudo-second-order R^2	0.989	0.995	0.997
Capacity retained after cycle 3 (%)	84	88	91

The illustrative pattern suggests that coconut-shell carbon would provide the highest iodine number, BET area and MB adsorption capacity, whereas rice-husk carbon would give the highest mass yield but greater ash content. Such a trade-off is plausible because precursor mineral content and lignocellulosic composition influence burn-off, pore development and surface chemistry. Published studies likewise show that strong adsorption performance can be obtained from oil-palm shell, coconut husk, orange peel, rice husk, bagasse and other residues when activation conditions are appropriately selected (Tan et al., 2008; Sharma and Uma, 2010; Farahani et al., 2011; Foo and Hameed, 2012a,b).

A better fit to the pseudo-second-order model would indicate that adsorption rate is strongly associated with the availability of surface sites, although model fit alone should not be interpreted as proof of a single chemical mechanism. A high Langmuir fit can be used to compare apparent monolayer capacity, while the Freundlich model remains useful for heterogeneous surfaces. Regeneration results should be interpreted together with carbon loss and desorption efficiency. Literature on groundnut-hull and other agricultural carbons demonstrates that reusability can materially affect practical treatment value (Bello et al., 2012).

Conclusion and Discussion

The proposed methodology provides a standardized framework for preparing and evaluating activated carbon from agricultural wastes for wastewater treatment. A common H_3PO_4 impregnation and 450 °C carbonization route allows rice husk, sugarcane bagasse and coconut shell to be compared under identical processing conditions. The most informative evaluation combines carbon yield with ash/moisture content, iodine number, BET/SEM/FTIR characterization, batch adsorption capacity, kinetic/isotherm modeling and regeneration. This multidimensional assessment is necessary because a precursor that gives a high carbon yield may not provide the highest surface area or pollutant uptake. Pre-2016 literature consistently supports the use of agricultural residues as practical AC precursors and shows that activation conditions strongly govern adsorption performance (Bhatnagar and

Sillanpää, 2010; Ahmad et al., 2012; Alslaibi et al., 2013). For an actual laboratory study, the illustrative data in Section 7 should be replaced with measured values, and the optimum activation temperature/impregnation ratio should be refined through a factorial or response-surface experiment if resources permit. Wastewater matrices should also be tested after the model-dye stage because dissolved salts, natural organic matter and competing contaminants can suppress adsorption. Nevertheless, the method is sufficiently detailed for reproducible laboratory work and provides a defensible basis for comparing low-cost activated carbons produced from locally available agricultural waste.

References

- Ahmad, A.L., Loh, M.M. & Aziz, J.A. (2007). Preparation and characterization of activated carbon from oil palm wood and its evaluation on methylene blue adsorption. *Dyes and Pigments*, 75(2), 263–272. DOI: 10.1016/j.dyepig.2006.05.034.
- Al-Muhtaseb, S.A., El-Naas, M.H. & Abdallah, S. (2008). Removal of aluminum from aqueous solutions by adsorption on date-pit and BDH activated carbons. *Journal of Hazardous Materials*, 158(2–3), 300–307. DOI: 10.1016/j.jhazmat.2008.01.080.
- Alslaibi, T.M., Abustan, I., Ahmad, M.A. & Foul, A.A. (2013). A review: production of activated carbon from agricultural byproducts via conventional and microwave heating. *Journal of Chemical Technology & Biotechnology*, 88(7), 1183–1190. DOI: 10.1002/jctb.4028.
- Ahmad, T., Danish, M., Rafatullah, M., Ghazali, A., Sulaiman, O., Hashim, R. & Ibrahim, M.N.M. (2012). The use of date palm as a potential adsorbent for wastewater treatment: a review. *Environmental Science and Pollution Research*, 19(5), 1464–1484. DOI: 10.1007/s11356-011-0709-8.
- Alam, M.Z., Ameen, E.S., Muyibi, S.A. & Kabbashi, N.A. (2009). The factors affecting the performance of activated carbon prepared from oil palm empty fruit bunches for adsorption of phenol. *Chemical Engineering Journal*, 155(1–2), 191–198. DOI: 10.1016/j.cej.2009.07.033.
- Bhatnagar, A. & Sillanpää, M. (2010). Utilization of agro-industrial and municipal waste materials as potential adsorbents for water treatment—A

review. Chemical Engineering Journal, 157(2-3), 277-296. DOI: 10.1016/j.cej.2010.01.007.

Bello, O.S., Fatona, T.A., Falaye, F.S., Osuolale, O.M. & Njoku, V.O. (2012). Adsorption of eosin dye from aqueous solution using groundnut hull-based activated carbon: kinetic, equilibrium, and thermodynamic studies. Environmental Engineering Science, 29(3), 186-194. DOI: 10.1089/ees.2010.0385.

Farahani, M., Abdullah, S.R.S., Hosseini, S., Shojaeipour, S. & Kashisaz, M. (2011). Adsorption-based cationic dyes using the carbon active sugarcane bagasse. Procedia Environmental Sciences, 10, 203-208. DOI: 10.1016/j.proenv.2011.09.035.

Foo, K.Y. & Hameed, B.H. (2012a). Coconut husk derived activated carbon via microwave induced activation: effects of activation agents, preparation parameters and adsorption performance. Chemical Engineering Journal, 184, 57-65. DOI: 10.1016/j.cej.2011.12.084.

Foo, K.Y. & Hameed, B.H. (2012b). Preparation, characterization and evaluation of adsorptive properties of orange peel based activated carbon via microwave induced K₂CO₃ activation.

Bioresource Technology, 104, 679-686. DOI: 10.1016/j.biortech.2011.10.005.

Kaźmierczak, J., Nowicki, P. & Pietrzak, R. (2013). Sorption properties of activated carbons obtained from corn cobs by chemical and physical activation. Adsorption, 19, 273-281. DOI: 10.1007/s10450-012-9450-y.

Prahas, D., Kartika, Y., Indraswati, N. & Ismadji, S. (2008). Activated carbon from jackfruit peel waste by H₃PO₄ chemical activation: pore structure and surface chemistry characterization. Chemical Engineering Journal, 140(1-3), 32-42. DOI: 10.1016/j.cej.2007.08.032.

Sharma, Y.C. & Uma. (2010). Optimization of parameters for adsorption of methylene blue on a low-cost activated carbon. Journal of Chemical & Engineering Data, 55(1), 435-439. DOI: 10.1021/jc900408s.

Tan, I.A.W., Ahmad, A.L. & Hameed, B.H. (2008). Adsorption of basic dye using activated carbon prepared from oil palm shell: batch and fixed bed studies. Desalination, 225(1-3), 13-28. DOI: 10.1016/j.desal.2007.07.005.