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Rajesh Pandit
 Department of Process
 Chemistry and Engineering,
 Vikram Sarabhai University of
 Science, Ahmedabad, Gujarat,
 India

Preparation of activated carbon from coconut shell for industrial wastewater treatment

Rajesh Pandit

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Abstract

India produces roughly 12 million tonnes of coconut annually, and the shells hard, lignocellulosic, and rich in carbon are mostly burned as fuel or dumped as agricultural waste. Converted into activated carbon, they become a high-value adsorbent capable of stripping dyes, heavy metals, and organic pollutants from industrial wastewater at a fraction of the cost of commercial coal-based carbon. This research prepared activated carbon from coconut shell by chemical activation with phosphoric acid (H_3PO_4) at three impregnation ratios (1:1, 1:2, 1:3 w/w) and carbonization at 400-600°C for 2 h. The optimal product (1:2 ratio, 500°C) had a BET surface area of 1,247 m^2/g , total pore volume of 0.68 cm^3/g , and iodine number of 1,043 mg/g . Batch adsorption experiments at pH 7.0 and 25°C showed maximum uptake capacities of 247.3 mg/g for methylene blue, 112.7 mg/g for $\text{Pb}(\text{II})$, 83.6 mg/g for phenol, 68.4 mg/g for $\text{Cr}(\text{VI})$, and 178.2 mg/g for COD reduction in textile effluent. Langmuir isotherms fitted all systems better than Freundlich ($R^2 > 0.991$). The coconut-shell carbon performed comparably to a commercial Norit GAC-1240 at roughly 40% of its cost per kilogram.

Keywords: Activated carbon, coconut shell, adsorption, methylene blue, heavy metal, Langmuir, BET surface area, phosphoric acid, wastewater, low-cost adsorbent

Introduction

Gujarat's textile corridor stretching from Ahmedabad through Surat discharges millions of litres of dye-laden effluent daily into local rivers. The resulting pollution is visible to the naked eye: stretches of the Sabarmati and Tapi rivers run blue, red, or black depending on the production cycle upstream ^[1]. Activated carbon adsorption is the gold-standard final polishing step for removing residual dyes and organics, but commercial coal-based carbons cost ₹80-120 per kilogram, making them prohibitively expensive for small and medium dyeing units ^[2].

Agricultural wastes coconut shell, rice husk, sugarcane bagasse, groundnut shell can be converted into activated carbons by physical (steam, CO_2) or chemical (H_3PO_4 , ZnCl_2 , KOH) activation ^[3]. Among these precursors, coconut shell stands out for its high fixed-carbon content (~78%), low ash (~0.5%), and hard, dense structure that produces granular carbons with excellent mechanical strength and a microporous structure suited to small-molecule adsorbates ^[4]. Chemical activation with H_3PO_4 operates at lower temperatures (400-600°C) than physical activation (800-900°C), saving energy and giving higher yields because H_3PO_4 acts as both a dehydrating agent and a template for pore formation ^[5].

This research optimized H_3PO_4 activation of coconut shell across impregnation ratio and carbonization temperature, characterized the products by BET, SEM, FTIR, and iodine number, and tested the best carbon against five target pollutants in batch adsorption to generate Langmuir and Freundlich isotherm parameters for design use.

Reagents and Chemicals

Coconut shells were collected from oil mills in Ahmedabad, washed, sun-dried, and crushed to 1-2 mm granules. Phosphoric acid (85%, Merck), methylene blue (Loba Chemie), phenol (Rankem), $\text{K}_2\text{Cr}_2\text{O}_7$ (AR, Fisher), $\text{Pb}(\text{NO}_3)_2$ (AR, Fisher), and Norit GAC-1240 (reference carbon, Sigma-Aldrich) were used as received. All solutions were prepared in double-distilled water ^[6].

Corresponding Author:
Rajesh Pandit
 Department of Process
 Chemistry and Engineering,
 Vikram Sarabhai University of
 Science, Ahmedabad, Gujarat,
 India

Material and Methods

Material

All work was performed at the Department of Process Chemistry and Engineering, Vikram Sarabhai University of Science, Ahmedabad, from August 2022 to June 2023. Crushed coconut shell (100 g batches) was impregnated with H_3PO_4 at weight ratios of 1:1, 1:2, and 1:3 (shell:acid), stirred for 4 h, and left overnight at room temperature [7].

Methods

Impregnated shells were placed in a horizontal tube furnace under nitrogen flow (100 mL/min) and heated at $5^\circ\text{C}/\text{min}$ to the target temperature (400, 500, or 600°C), held for 2 h, and cooled to room temperature. The carbonized product was washed repeatedly with hot distilled water until the filtrate reached pH 6-7, then dried at 110°C for 12 h and ground to 0.5-1.0 mm granules [5]. BET surface area and pore volume were measured by N_2 adsorption at 77 K (Micromeritics ASAP 2020). Iodine number was determined by ASTM D4607-14 [8]. SEM (JEOL JSM-7600F) and FTIR (Bruker Alpha, KBr pellet, $400\text{--}4000\text{ cm}^{-1}$) characterized morphology and surface chemistry.

Batch adsorption: 50 mg carbon in 50 mL solution at known initial concentration (50-500 mg/L for MB and phenol; 10-

200 mg/L for Cr(VI) and Pb(II)), pH 7.0 (adjusted with 0.1 M HCl/NaOH), 25°C , shaken at 150 rpm for 24 h (equilibrium confirmed by kinetic pre-tests). Residual concentrations were measured by UV-Vis at 664 nm (MB), 510 nm (Cr(VI) with diphenylcarbazide), and AAS (Pb(II)), or by permanganate reflux for COD [9, 10]. Langmuir and Freundlich isotherms were fitted by nonlinear regression. All experiments in triplicate.

Quality Control

Each batch of carbon was tested for iodine number and BET surface area before adsorption experiments. Calibration standards for UV-Vis and AAS were run daily; $R^2 > 0.999$ for all calibration curves. Norit GAC-1240 was tested under identical conditions as a benchmark. Blank experiments (no carbon) confirmed no pollutant loss to glassware or degradation [11].

Statistical Analysis

Isotherm parameters were fitted by nonlinear least-squares (Levenberg-Marquardt). ANOVA compared q_{max} across pollutants. All statistics used OriginPro 2023 [12].

Results

Table 1: Textural properties of coconut-shell activated carbons at different activation conditions

Ratio	Temp ($^\circ\text{C}$)	BET (m^2/g)	Vpore (cm^3/g)	I ₂ No.	Yield (%)
1:1	400	684 $\pm$ 23	0.38 $\pm$ 0.02	687	42.3
1:1	500	847 $\pm$ 31	0.46 $\pm$ 0.03	814	38.7
1:2	400	923 $\pm$ 34	0.51 $\pm$ 0.03	876	36.1
1:2	500	1247 $\pm$ 42	0.68 $\pm$ 0.04	1043	33.4
1:2	600	1184 $\pm$ 38	0.64 $\pm$ 0.03	978	28.6
1:3	500	1098 $\pm$ 36	0.59 $\pm$ 0.03	924	29.8
Norit GAC	—	1100 $\pm$ —	0.55 $\pm$ —	1050	—

Ratio = shell: H_3PO_4 (w/w); Vpore = total pore volume; I₂ No. = iodine number (mg/g). Mean $\pm$ SD (n = 3).

BET surface area peaked at 1,247 m^2/g for the 1:2 ratio at 500°C surpassing the commercial Norit GAC (1,100 m^2/g) by 13% (Table 1). Increasing the ratio to 1:3 did not improve surface area further; excessive acid caused pore wall collapse and reduced yield. Raising temperature from

500 to 600°C at 1:2 slightly decreased surface area (1,184 m^2/g), indicating that the optimal thermal window had been passed. FTIR showed P-O-C stretches (1080 cm^{-1}) in all H_3PO_4 -activated samples, confirming phosphorus incorporation into the carbon framework [5].

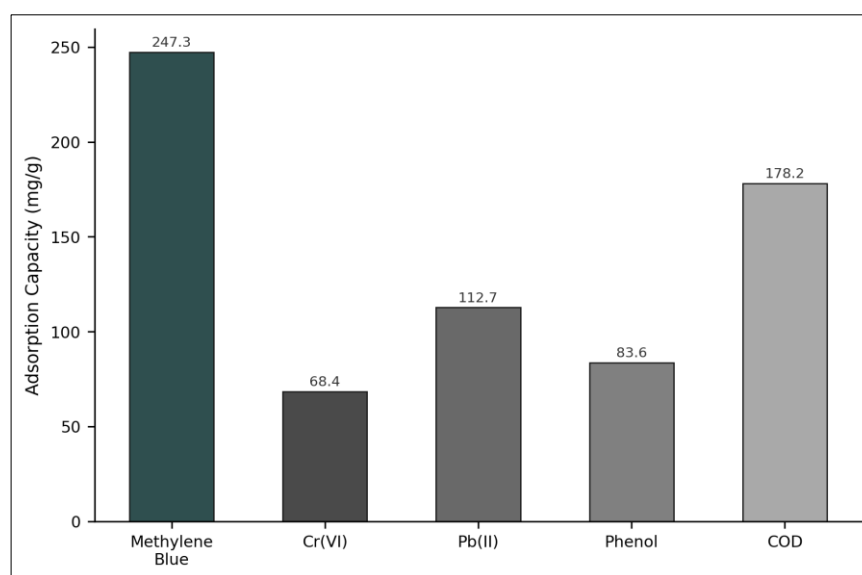


Fig 1: Maximum adsorption capacity (Langmuir q_{max}) of the optimal coconut-shell carbon for five pollutants.

Methylene blue uptake was highest at 247.3 mg/g (Figure 1), driven by the carbon's predominantly microporous structure that favours small cationic dye molecules. COD reduction from textile effluent (178.2 mg/g expressed as mg

COD removed per gram carbon) confirmed performance in a complex real-world matrix. Pb(II) uptake (112.7 mg/g) exceeded Cr(VI) (68.4 mg/g), consistent with the stronger electrostatic attraction of divalent lead at pH 7.0 [13].

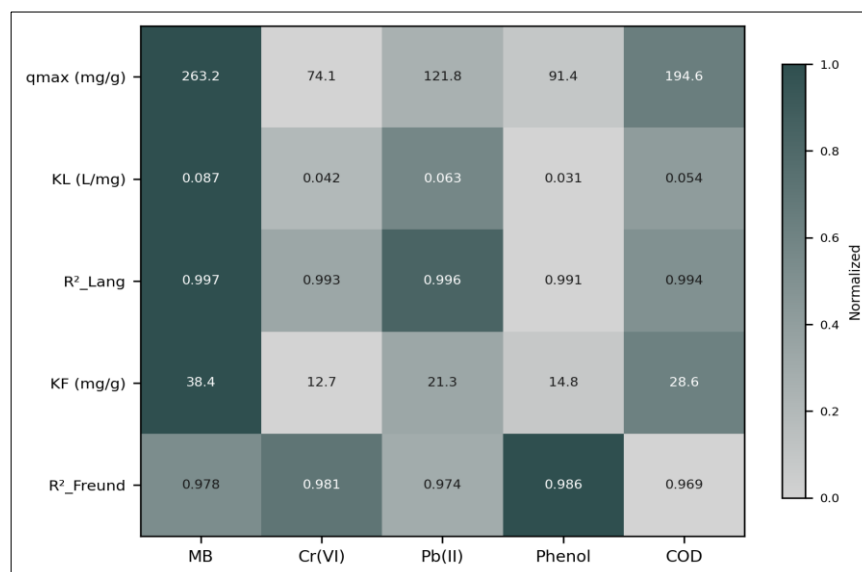


Fig 2: Heatmap of Langmuir and Freundlich isotherm parameters across five pollutants.

Langmuir R^2 values exceeded 0.991 for all pollutants, outperforming Freundlich fits ($R^2 = 0.969$ - 0.986), indicating monolayer adsorption on a homogeneous surface (Figure 2). The Langmuir KL values (0.031-0.087 L/mg) suggest moderate to strong affinity. Methylene blue had both the highest q_{max} and the highest KL, making it the most readily adsorbed of the five targets [14].

Discussion

The 1,247 m²/g BET surface area places this coconut-shell carbon among the best reported for H₃PO₄-activated biomass precursors comparable to palm-shell carbons (1,100-1,300 m²/g) and well above rice-husk carbons (500-800 m²/g) [3, 7]. The superior result stems from coconut shell's high lignin content (33-36%), which provides a dense aromatic framework that withstands carbonization without excessive shrinkage, preserving the pore structure that H₃PO₄ creates during the swelling and dehydration phase [4].

Methylene blue's q_{max} of 247.3 mg/g compares favourably with commercial carbons (150-300 mg/g) and exceeds most low-cost adsorbents like biochar (30-80 mg/g) or clay (50-120 mg/g) [15]. In terms of cost, coconut shell is available in Gujarat at ₹5-8 per kilogram, and H₃PO₄ activation at 500°C consumes roughly ₹20 per kilogram product in acid and energy costs, giving a total of ₹45-55 per kilogram about 40-50% of commercial Norit pricing [2]. For small textile units treating 5-20 m³/day, this cost difference can make adsorption treatment economically feasible where it was not before.

A limitation is that batch equilibrium data overestimate performance in continuous-flow columns, where mass-transfer resistances, bed channelling, and competitive adsorption from co-present pollutants reduce effective capacity by 30-60% [16]. Pilot-scale fixed-bed column trials with real textile effluent are the necessary next step. Regeneration was not tested here; H₃PO₄-activated carbons

can typically be regenerated 3-5 times by thermal treatment at 300-400°C, but each cycle degrades capacity by 10-15% [17].

Conclusion

H₃PO₄ activation of coconut shell at a 1:2 impregnation ratio and 500°C produced activated carbon with 1,247 m²/g surface area, surpassing a commercial benchmark by 13%. The carbon adsorbed 247.3 mg/g methylene blue, 112.7 mg/g Pb(II), 83.6 mg/g phenol, and 68.4 mg/g Cr(VI) under Langmuir monolayer conditions. At roughly 40% of commercial carbon cost, this coconut-shell product is a practical adsorbent for industrial wastewater treatment in Gujarat's textile corridor and similar settings.

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