IOCE 2026 Conference

The 1st International Online Conference on Environments

02–04 March 2026 | Online

Chaired by Prof. Dr. Sergio Ulgiati

environments MDPI

Advancing Low-Cost Biosorbent Engineering: Optimized Alkaline Activation of Agro-Industrial Biomass for Enhanced Removal of Cationic Dyes

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INTRODUCTION & AIM

The discharge of dye-containing industrial effluents represents a significant environmental concern due to the persistence and stability of synthetic dyes in aquatic systems. Methylene blue (MB), a widely used cationic dye, is frequently selected as a model pollutant because of its high stability and resistance to biodegradation. Adsorption processes are considered among the most effective treatment approaches owing to their operational simplicity and high removal efficiency; however, the economic limitations associated with conventional adsorbents motivate the development of alternative low-cost materials derived from biomass residues.

Agricultural wastes represent a promising resource due to their abundance, renewability, and chemical functionality. Nevertheless, untreated biomasses generally exhibit limited adsorption performance because of poor porosity and low availability of active surface groups. Chemical activation is therefore required to enhance adsorption properties, although activation efficiency strongly depends on operating parameters.

The objective of this work was to optimize NaOH chemical activation conditions for two lignocellulosic wastes — spent coffee grounds (SCG) and date pits (DP) — using a full factorial experimental design. The study further aimed to correlate activation conditions with structural characteristics and adsorption performance toward methylene blue through kinetic, isotherm, and thermodynamic analyses.

METHOD

Experimental design and activation procedure

Chemical activation was performed using sodium hydroxide under a full factorial 2^3 design considering three independent variables:

- NaOH concentration: 0.2–0.7 M
- activation time: 1.5–5.5 h
- activation temperature: 22–50 °C

The response variable selected for optimization was the maximum adsorption capacity (Qm). After treatment, samples were washed to neutral pH, dried, ground, and sieved. Adsorption experiments were carried out in batch mode using methylene blue solutions and UV–Vis quantification at 665 nm.

Figure 1. Response optimization chart obtained using Minitab.

Characterization and adsorption studies

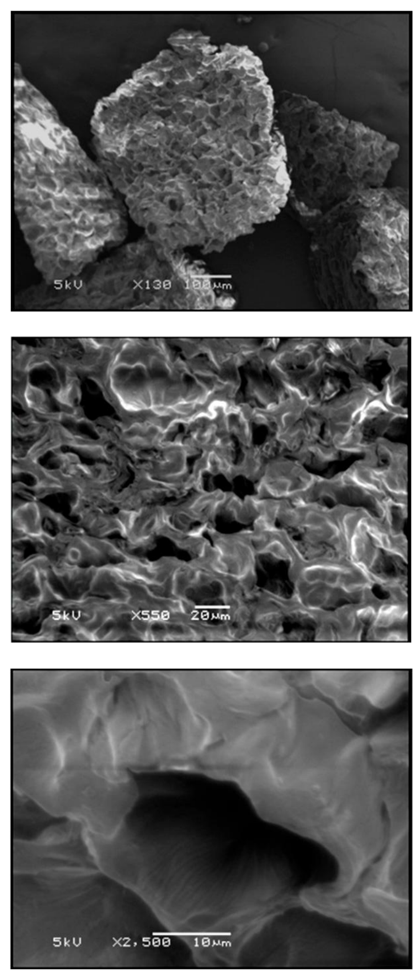
The optimized biosorbent was characterized using:

- BET/BJH analysis (surface area and porosity)
- SEM (surface morphology)
- FTIR spectroscopy (functional groups)
- pHpzc determination (surface charge behavior)

Operational parameters studied included solution pH, adsorbent dosage, contact time, and temperature. Adsorption kinetics were evaluated using pseudo-first-order (PFO), pseudo-second-order (PSO), Elovich, and intraparticle diffusion models, while equilibrium data were fitted using Langmuir, Freundlich, and Sips isotherms.

RESULTS & DISCUSSION

Figure 2. SEM images of SCG_3.



The factorial design highlighted a clear difference between the two investigated biomasses. The statistical model obtained for date pits was not significant, while the spent coffee grounds (SCG) model showed strong predictive capability. Activation time and temperature were identified as the main controlling factors, whereas NaOH concentration had a negligible influence within the tested range.

The optimal activation conditions were determined as **0.2 M NaOH, 5.5 h activation time, and 22 °C**, producing the optimized material SCG3 with a maximum adsorption capacity of **140.23 mg·g⁻¹**. This value confirms the effectiveness of mild alkaline treatment in enhancing adsorption performance without excessive structural degradation.

BET analysis revealed a specific surface area of **37.44 m²·g⁻¹** and a mesoporous structure (average pore diameter ≈ 4.39 nm), suitable for methylene blue diffusion. SEM observations showed a heterogeneous porous morphology facilitating mass transfer toward adsorption sites. FTIR analysis confirmed the presence of oxygenated functional groups (–OH, C=O, C–O), while the pHpzc value (5.42) indicates that adsorption is favored at neutral–alkaline pH due to electrostatic attraction between negatively charged surface sites and cationic dye molecules.

Adsorption performance increased with pH and contact time, reaching equilibrium after approximately **4 h**. The optimal adsorbent dosage was **1 g·L⁻¹**, balancing removal efficiency and adsorption capacity. Increasing temperature reduced adsorption capacity, indicating an exothermic process confirmed by thermodynamic analysis ($\Delta G° < 0$, $\Delta H° < 0$), consistent with a spontaneous and predominantly physical adsorption mechanism.

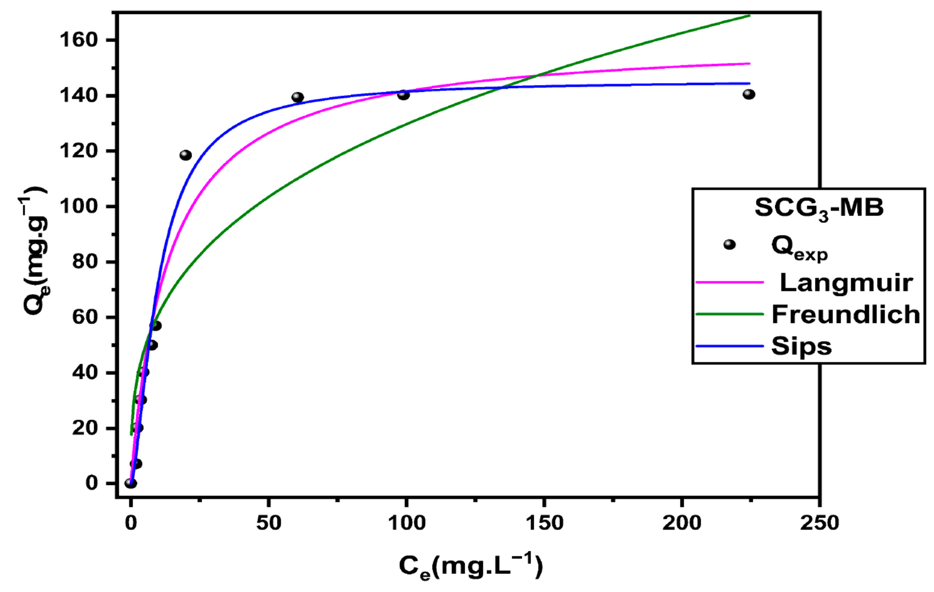


Figure 3. Nonlinear fitting of the MB adsorption isotherm onto SCG_3.

Kinetic data were best described by the pseudo-second-order model ($R^2 \approx 0.96$), while intraparticle diffusion indicated a two-step adsorption mechanism. Equilibrium was accurately fitted by the Sips model ($R^2 \approx 0.99$), confirming heterogeneous surface adsorption. SCG3 showed competitive adsorption capacity compared with other chemically activated biomasses.

CONCLUSION

Optimization of NaOH activation conditions significantly improved the adsorption performance of spent coffee grounds toward methylene blue. The optimized material (SCG3) showed high adsorption capacity, favorable surface chemistry, and suitable porous morphology. Adsorption proceeded through a spontaneous and exothermic mechanism governed primarily by physical interactions and heterogeneous surface adsorption behavior.

The combination of factorial design optimization, material characterization, and adsorption modeling confirms the potential of chemically activated spent coffee grounds as a low-cost and sustainable adsorbent for dye-contaminated wastewater treatment.

FUTURE WORK / REFERENCES

Future research will focus on regeneration and reuse cycles, treatment of real industrial effluents, and scale-up evaluation for practical implementation.

1. *Bounaas, Meryem, et al. "Optimization of NaOH Chemical Treatment Parameters for Biomass-Based Adsorbents in Cationic Dye Removal." Processes 13.12 (2025): 3932.*