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SUSTAINABLE HYDROGEL–CLAY COMPOSITES FOR THE ADSORPTION OF TOXIC METAL IONS FROM WATER

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1. Objective

To evaluate the effect of incorporating 10 wt% Brasgel clay into epichlorohydrin-crosslinked cellulose hydrogels on their structural properties and Cu^{2+} adsorption performance in aqueous solutions, comparing the composite hydrogel (HB10%) with the pure hydrogel (H).

2. Introduction

Water contamination by metal ions



Only 1%
Of the world´s water is available as freshwater.

Main sources

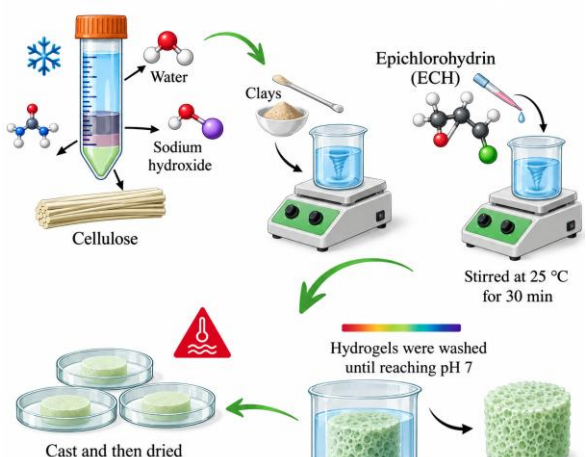


Main sources

Persistence	Bioaccumulation	Biomagnification	Toxicity
They do not degrade naturally.	Accumulation of metals in organisms over time.	Increase in metal concentration across trophic levels.	They cause harm to human health and ecosystems.

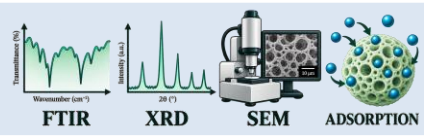
Awang, N. A., Wan Salleh, W. N., Aziz, F., Yusof, N., & Ismail, A. F. (2023)

3. Materials and Methods



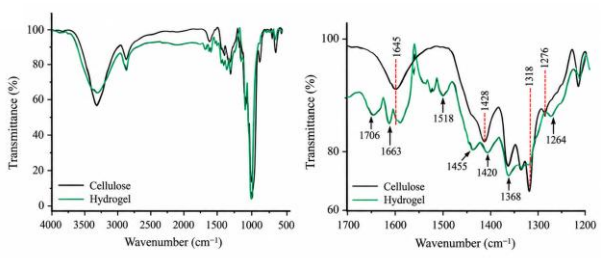
Sample identification	Clay	Clay content (%, relative to cellulose)
H	-	-
HB3%	Brasgel	3
HB5%		5
HB10%		10

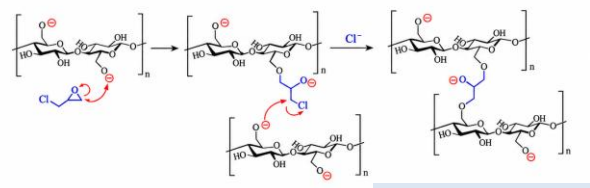
Characterization techniques

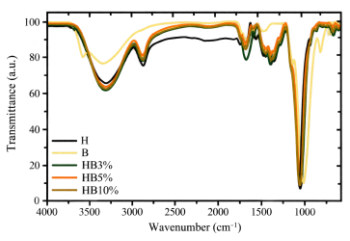


4. Results and Discussions

FTIR



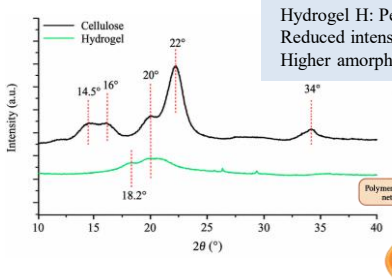




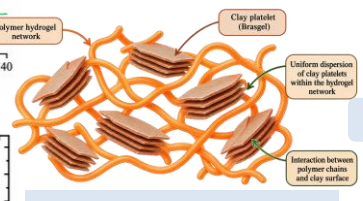
- ✓ **3300 cm⁻¹ (–OH)**
  Decrease in intensity indicates the consumption of hydroxyl groups during crosslinking.
- ✓ **1706 cm⁻¹ (C=O)**
  New band indicating the formation of carbonyl groups after the reaction with ECH.
- ✓ **1276 → 1264 cm⁻¹ (C–O–C)**
  Characteristic shift associated with the formation of ether bonds in the crosslinked network.
- ✓ **1200–900 cm⁻¹**
  Increased intensity due to Si–O–Si vibrations, confirming clay incorporation without the appearance of new bands.

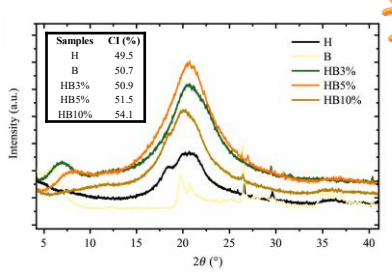
(Ferreira et al., 2025a).

XRD



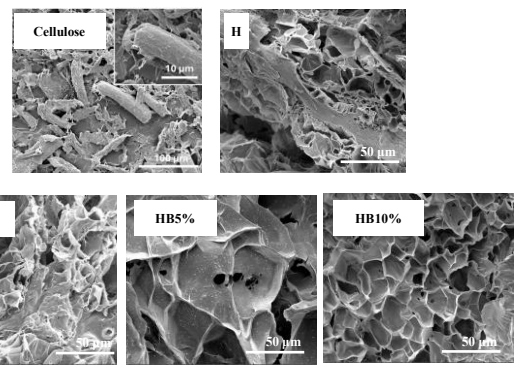
Cellulose I: Peaks at 16° (110) and 22° (200).
Hydrogel H: Peak at 22.3° (200) → Cellulose II.
Reduced intensity at 16° and 31°.
Higher amorphous fraction after crosslinking.





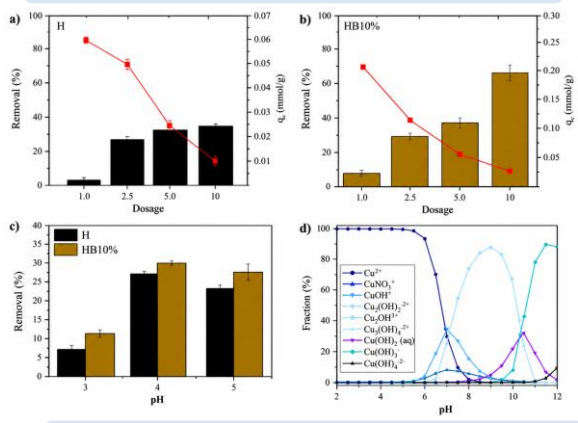
The CI increased from 49.5% (H) to 54.1% (HB10%), while the reduced peak intensity suggests changes in the diffraction profile associated with clay dispersion and interactions within the hydrogel network (Aljeboree, Hussein, et al., 2025).

SEM

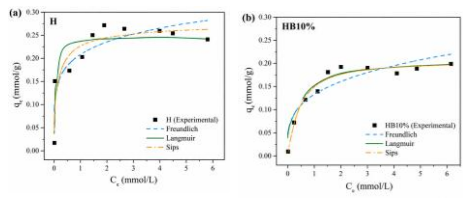


Adsorption

Solid-to-liquid ratio (S/L) and effect of pH



Cu^{2+} Adsorption Isotherm

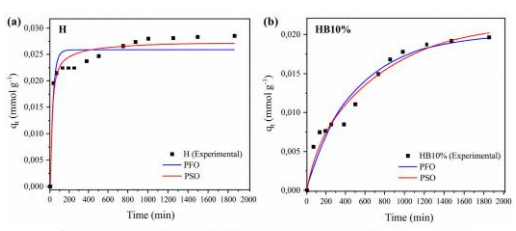


Model	Parameter	H	HB10%
Langmuir	q_e (mmol/g)	0,243 ± 0,012	0,215 ± 0,008
	K_L (L/mmol)	55,00 ± 26,79	2,17 ± 0,40
	Adj. R^2	0,81377	0,96957
	AIC	-88,9	-90,3
Freundlich	K_F	0,207 ± 0,013	0,132 ± 0,009
	1/n	0,169 ± 0,043	0,276 ± 0,053
	Adj. R^2	0,77516	0,85805
	AIC	-64,9	-75,6
Sips	q_m (mmol/g)	0,293 ± 0,081	0,209 ± 0,014
	K_s (mmol/g)	3,39 ± 4,91	2,47 ± 0,83
	n_s	0,48 ± 0,27	1,12 ± 0,24
	Adj. R^2	0,80419	0,96651
	AIC	-86,5	-88,7

Color scale – Adj. R^2 (Higher = Better fit): 0,75 Poorer fit – 1,00 Better fit
Color scale – AIC (Lower = Better fit): -91 Better fit – -64 Poorer fit

Overall, the results show that the system is well described by the Langmuir model, with an additional contribution from heterogeneity in the modified material. This behavior is common in hybrid materials, in which the incorporation of an inorganic phase alters the energetic distribution of adsorption sites (Teğin et al., 2023).

Cu^{2+} Adsorption Kinetics



Kinetic model	Parameters	H (Cu^{2+})	HB10% (Cu^{2+})
PFO (Pseudo-first-order)	q_e (mmol g⁻¹)	0,026 ± 0,001	0,021 ± 0,001
	k_1 (min⁻¹)	0,041 ± 0,010	0,0019 ± 0,0003
	Reduced chi-square (χ^2_ν)	6,20 × 10⁻⁶	2,65 × 10⁻⁶
	R^2	0,896	0,946
	Adjusted R^2	0,888	0,941
	AIC	-152,9	-167,0
PSO (Pseudo-second-order)	q_e (mmol g⁻¹)	0,027 ± 0,001	0,027 ± 0,002
	k_2 (g mmol⁻¹ min⁻¹)	2,30 ± 0,64	0,068 ± 0,022
	Reduced chi-square (χ^2_ν)	3,31 × 10⁻⁶	2,37 × 10⁻⁶
	R^2	0,944	0,951
	Adjusted R^2	0,940	0,947
	AIC	-163,4	-168,4

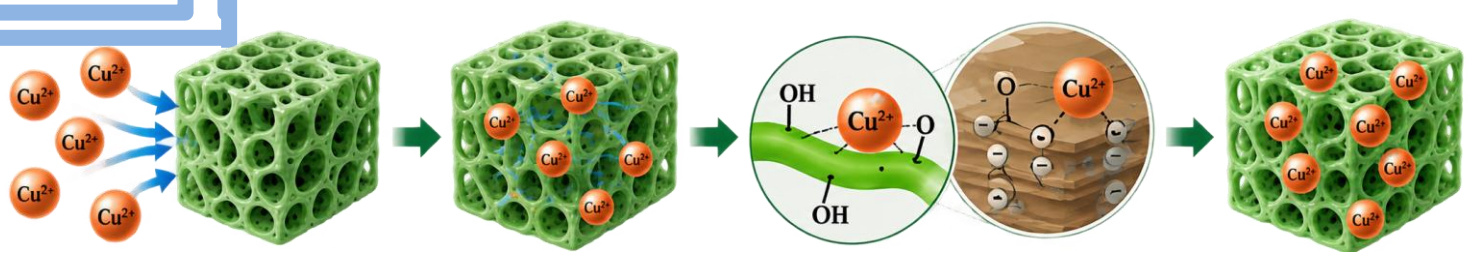
R^2 / Adjusted R^2 (Higher value = better fit): 0,50 Poorer fit – 1,00 Better fit
AIC (Lower value = better fit): -185 Better fit – -145 Poorer fit

PSO provided the best kinetic fit for both H and HB10%, indicating that the adsorption rate is governed by the availability of active sites in the clay-modified matrix.

5. Conclusions

- FTIR: Confirmed crosslinking.
- XRD: Revealed partial conversion from cellulose I to cellulose II.
- SEM: Revealed a porous, three-dimensional network.
- Brasgel clay incorporation: Significantly modified the hydrogels' structural and adsorption properties, promoting more organized and interconnected networks.

1. MAMAH, S. C. et al. *J. Water Process Eng.*, 62, 105394, 2024.
2. OUGHLIS-HAMMACHE, F. et al. *Materials*, 17, 3594, 2024.



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