New trends and strategies in the chemistry of advanced materials with relevance in biological systems, technique and environmental protection

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APPLICATION OF FULL FACTORIAL DESIGN FOR ADSORPTION OF METHYLENE BLUE DYE FROM AQUEOUS SOLUTION

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Introduction

- The application of the adsorption process for wastewater treatment has proven to be an effective method and has generated the development of new materials used as adsorbents.
- The application of magnetic nanocomposites as adsorbent materials in solving environmental problems has recently received great attention.

Aim

- Synthesis and characterization of new magnetic nanocomposite.
- Performing a full factorial design experiment to model and optimize the removal of Methylene Blue (MB) dye by adsorption on magnetic nanocomposite (NMC).
- The termodinamic studies of the adsorption of MB onto NMC.

Experimental

Synthesis of magnetic nanocomposite (NMC)

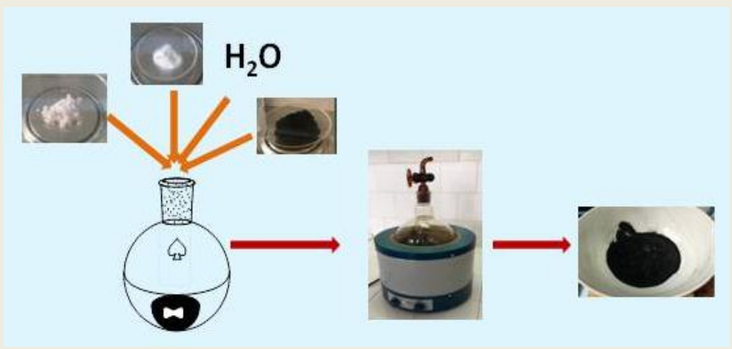
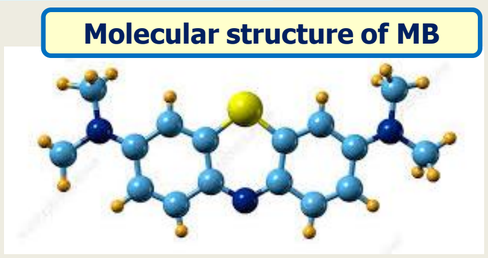


Table 1. Composition of magnetic nanocomposite

Sample	Mass $Fe(NO_3)_3 \cdot 9H_2O$ (g)	Mass $C_6H_{14}N_4$ (g)	Mass carbon (g)	Mass Ratio
NMC	10.4693	2.0358	8.00	1:4

Molecular structure of MB



Characterization of magnetic nanocomposite (NMC)

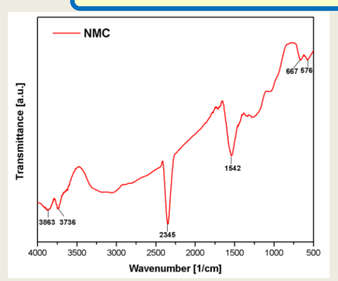


Fig.1. IR Spectrum

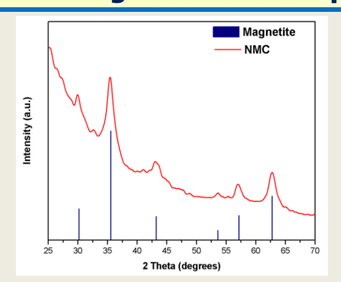


Fig.2. XRD pattern

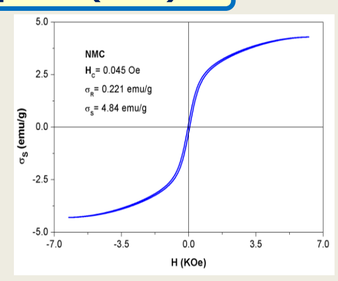


Fig.3. Magnetization curve

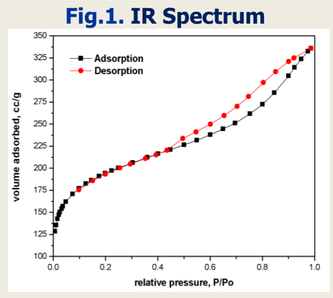


Fig.4. N_2 adsorption-desorption isotherm

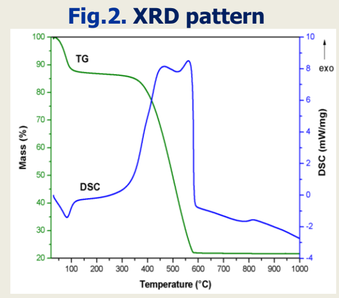


Fig.5. TG-DSC curve

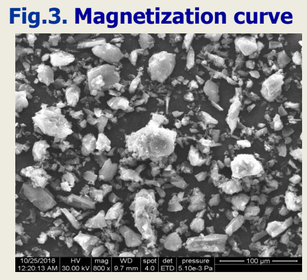


Fig.6. SEM analysis

Results and Discussions

Matrix Design

Table 2. The working conditions specific to each experiment and the experimental results obtained

Nr	pH	Dye concentration (mg/L)	Adsorbent dose (g/L)	Temp (°C)	R^* (%)
1	2.5	20	0.25	25	96.80
2	12.5	20	0.25	25	98.43
3	2.5	250	0.25	25	17.80
4	12.5	250	0.25	25	63.23
5	2.5	20	3	25	97.18
6	12.5	20	3	25	98.87
7	2.5	250	3	25	90.26
8	12.5	250	3	25	93.05
9	2.5	20	0.25	60	97.61
10	12.5	20	0.25	60	99.04
11	2.5	250	0.25	60	20.06
12	12.5	250	0.25	60	77.43
13	2.5	20	3	60	97.84
14	12.5	20	3	60	99.30
15	2.5	250	3	60	97.17
16	12.5	250	3	60	98.49
17	7.5	135	1.625	42.5	99.27
18	7.5	135	1.625	42.5	99.46
19	7.5	135	1.625	42.5	98.24

Tabel 3. The influence of operational parameters on adsorption efficiency

Level	pH	Conc.	NMC dosage	Temp.
-1	76.84	98.13	71.3	81.95
1	90.98	69.67	96.52	85.87
Slope	7.07	-14.22	12.61	1.96
Effect	significant	strong	strong	low

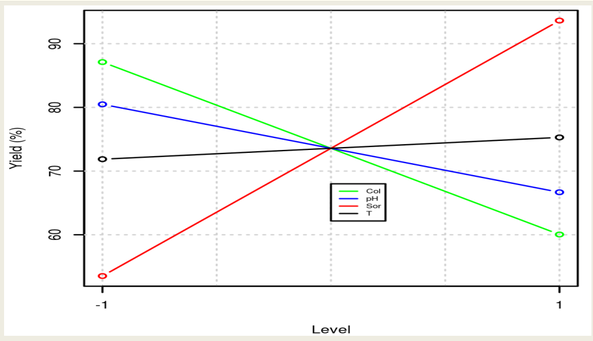


Fig. 7. The effects of four operational parameters on the MB adsorption efficiency

Interaction Analysis

Fig. 8. Interaction effect plot for AO7 dye removal

Statistical and regression analysis

The regression equation

$$R = 75.3 + 12.2\,pH - 0.323\,Col - 2.36\,Sor + 0.00474\,pH*Col + 0.428\,pH*Sor + 0.108\,Col*Sor - 1.00\,pH*pH$$

Table 4. Regression data for the quadratic model with interaction

	Value	Std Errors	t value	Pr(>|t|)
intercept	75.333	1.6690	45.15	0.000
b_1 :pH	12.2120	0.4274	28.57	0.000
b_2:Col	-0.3227	0.0066	-48.85	0.000
b_3:Sor	-2.3591	0.5513	-4.28	0.000
b_5:pH*Col	0.0047	0.0006	7.74	0.000
b_6:pH*Sor	0.4284	0.5125	8.36	0.000
b_8:Col*Sor	0.1084	0.0022	48.65	0.000
b_{12}:pH*pH	-1.0027	0.0269	-37.15	0.000

Table 5. ANOVA results for MB removal efficiency (%)

Source	DF	SumSq	MeanSq	F value	Pr(>F)
Regression	7	9226.6	1318.1	1326.91	0.000
Residual error	3	3.0	1.0		
Total	10	9229.6			

Estimation of the response surface

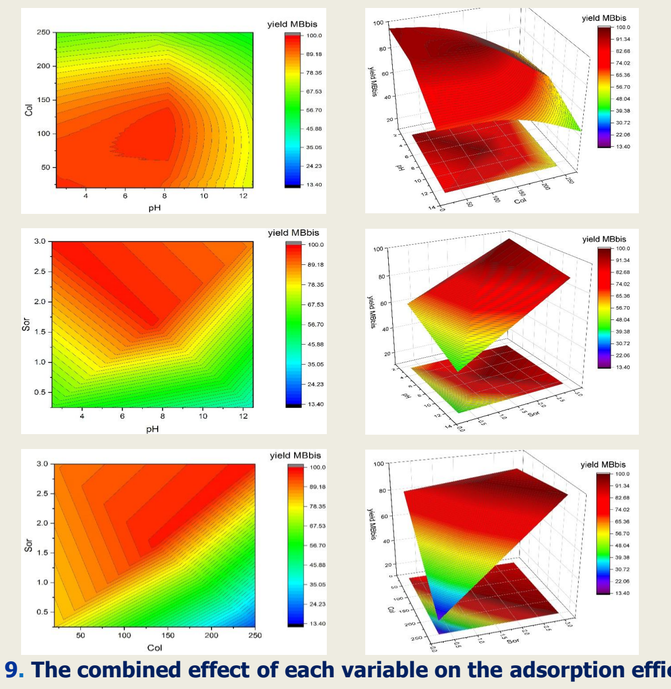


Fig. 9. The combined effect of each variable on the adsorption efficiency

Thermodynamic parameters

Dye	Sorbent	Temp. (°C)	ΔG^0 (J/mol)	ΔH^0 (J/mol)	ΔS^0 (J/mol·K)
MB	NMC	25	-2184.94	-2160.34	7.33
		40	-2294.93		
		55	-2404.91		

Thermodynamic study of adsorption

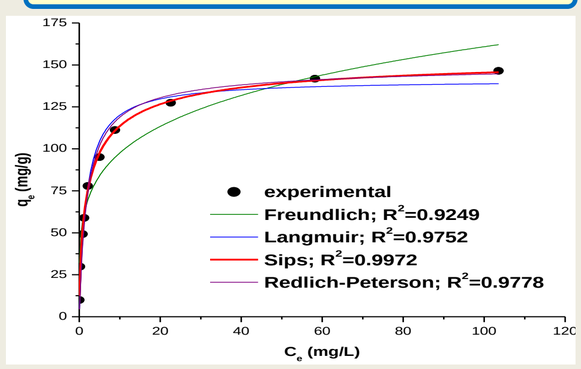


Fig. 10. Correlation of experimental data with adsorption isotherms

Table 6. Parameters of adsorption isotherms for adsorption of MB dye on NMC

Model	Parameter	Pollutant MB
Freundlich	K_F (mg/g(mg/L)$^{-1/n}$)	59.12
	n	4.60
	χ^2	190.37
Langmuir	q_m (mg/g)	141.17
	K_L (L/mg)	0.567
	χ^2	62.79
Sips	q_m (mg/g)	**157.58**
	K_S ((mg/L)$^{-1/n}$)	0.414
	n	1.50
	χ^2	**8.16**
Redlich-Peterson	K_{RP} (L/g)	76.11
	α_{RP} (mg/L)$^{-\beta}$	0.565
	β	0.981
	χ^2	64.44

Conclusions

- New magnetic adsorbent was successfully synthesized by a facile combustion method.
- The results obtained from the application of the full factorial design method showed that the statistical approach is an appropriate tool for optimizing the operational parameters, in order to obtain a maximum efficiency of removal of dyes from aqueous solutions.