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Adsorption of Methylene Blue from Aqueous Solution Using Zinc Chloride-Activated Buckwheat Hull Carbon

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Abstract
Buckwheat hull, an under-utilised agricultural residue, was converted into activated carbon by impregnation with zinc chloride followed by carbonisation. The resulting material was tested for its ability to remove the cationic dye methylene blue from water. Residual dye concentrations were calculated from optical density readings using the linear relationship of Beer's law. Batch experiments showed that removal increased with contact time and adsorbent dose. For a 2 ppm solution the residual concentration fell to 0.32 ppm after 50 min (approximately 84 % removal). Higher initial concentrations required longer contact times to reach comparable efficiency. Temperature elevation from 40 °C to 50 °C improved uptake for most solutions examined, while alkaline conditions favoured adsorption. The study demonstrates that zinc chloride-activated buckwheat hull carbon is a practical, low-cost adsorbent for methylene blue and offers a route for the productive use of an agricultural by-product.

Keywords: Methylene blue; Buckwheat hull; Zinc chloride activation; Adsorption; Residual concentration; Agricultural waste

1. Introduction

Methylene blue ($C_{16}H_{18}ClN_3S$) is a widely used cationic dye in the textile and paper industries. Even at low concentrations it imparts intense colour to water and is known to be toxic to aquatic organisms and humans. Conventional treatment processes such as coagulation or advanced oxidation are often expensive for small-scale or rural applications. Adsorption onto activated carbon remains one of the simplest and most effective methods, yet commercial activated carbon is costly. This has prompted continued interest in the preparation of carbons from abundant agricultural wastes.

Buckwheat (Fagopyrum esculentum) is cultivated in several parts of India and neighbouring countries. After the grain is milled, the hull is usually discarded. The hull contains cellulose, hemicellulose and lignin and therefore possesses the structural requirements of a good carbon precursor. Chemical activation with zinc chloride is known to produce carbons with well-developed porosity because the salt acts as a dehydrating agent and template during carbonisation (Yahya et al., 2015).

Several recent studies have confirmed that agricultural residues activated with $ZnCl_2$ can remove methylene blue efficiently (Das et al., 2025; Luyen, 2023). However, systematic data on buckwheat-hull carbon prepared under simple laboratory conditions are still limited. The present work reports the preparation of such a carbon and evaluates its performance by converting measured optical densities into residual dye concentrations (ppm) so that the results can be expressed in conventional concentration units.

2. Materials and Methods

2.1 Preparation of the adsorbent

Locally collected buckwheat hull was washed thoroughly with distilled water and air-dried. Approximately 250 g of the cleaned material was soaked for 12 h in a zinc chloride solution (2.5 g $ZnCl_2$ in 2.5 L water). After filtration and brief sun-drying the impregnated hull was carbonised in a muffle furnace at 250 °C. The char was ground, washed, and further heated at 350 °C for 30 min in the presence of a dilute $ZnCl_2$ solution. The finished product, designated BWHC, was stored in sealed polythene bags.

2.2 Dye solutions and analytical method

A stock solution of methylene blue (100 ppm) was diluted to give working solutions of 2, 5, 10, 20 and 30 ppm. Optical density (OD) of each solution was measured on a UV-visible spectrophotometer. The OD values of the freshly prepared standards were 0.504 (2 ppm), 0.859 (5 ppm), 1.564 (10 ppm), 1.534 (20 ppm) and 1.654 (30 ppm). These calibration values were used to convert all subsequent residual OD readings into residual concentration according to the relation

$C_t = C_0 \times (OD_t / OD_0)$

where C_t and OD_t are the residual concentration and optical density at time t, and C_0 and OD_0 are the corresponding initial values. Percentage removal was calculated as $[(C_0 - C_t)/C_0] \times 100$.

2.3 Batch adsorption procedure

Fifty millilitres of dye solution were placed in a reagent bottle together with a weighed quantity of BWHC (normally 0.1 g). The bottles were agitated on a rotary shaker for the required time, after which the suspension was centrifuged and the supernatant OD recorded. Contact times of 2-90 min, adsorbent doses of 0.05-0.40 g, temperatures of 40 °C and 50 °C, and both acidic and alkaline pH conditions were examined.

3. Results and Discussion

3.1 Effect of contact time

Residual concentrations calculated from the measured optical densities are listed in Table 1. For the 2 ppm solution the residual concentration dropped from 0.82 ppm after 2 min to 0.32 ppm after 50 min, corresponding to a removal of nearly 84 %. A similar pattern was observed for the 5 ppm solution, where residual concentration reached 0.68 ppm (86 % removal) at 50 min. At higher initial concentrations the residual values remained elevated for longer periods, indicating that the available surface sites became progressively occupied. Figure 1 illustrates the decline in residual concentration with time, while Figure 2 shows the corresponding removal efficiencies for the lower concentration range.

Table 1. Residual concentration of methylene blue (ppm) calculated from optical density measurements (adsorbent dose = 0.1 g)

Time (min)	2 ppm	5 ppm	10 ppm	20 ppm	30 ppm
2	0.82	2.31	8.10	19.35	19.28
5	0.62	2.08	6.91	16.62	21.39
10	0.79	2.62	8.54	13.90	24.45
15	0.64	2.41	8.12	19.35	22.22

30	0.72	1.55	7.05	17.41	26.95
50	0.32	0.68	2.55	10.08	24.23
60	0.37	1.82	2.78	8.93	26.99
90	0.50	1.89	8.08	1.76	28.22

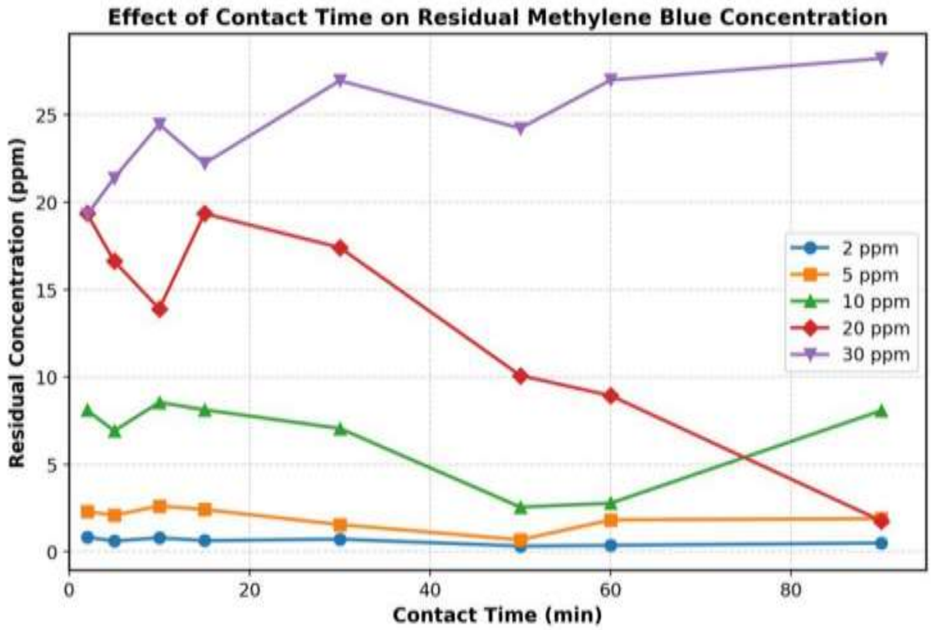

Figure 1. Residual methylene blue concentration as a function of contact time

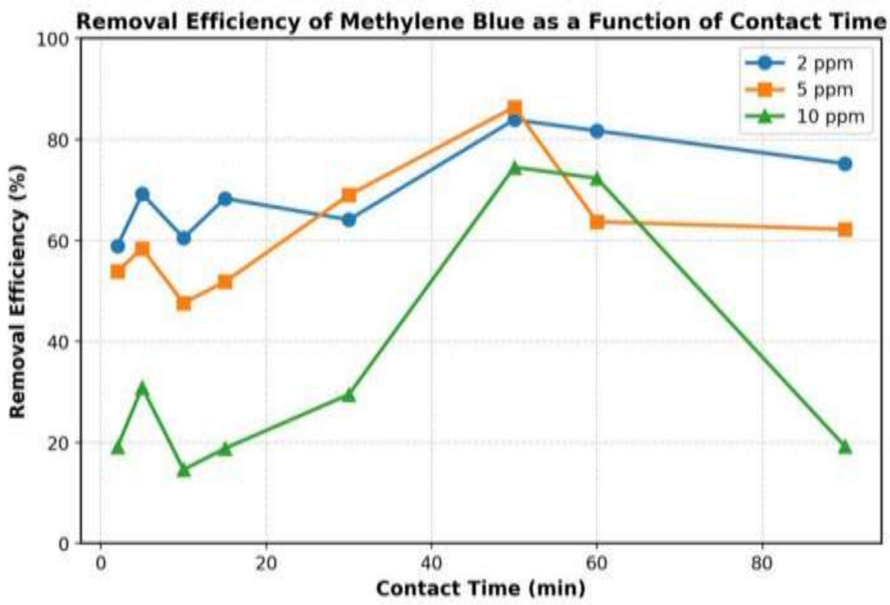

Figure 2. Removal efficiency of methylene blue versus contact time for 2, 5 and 10 ppm solutions

3.2 Effect of adsorbent dosage

Increasing the mass of BWHC from 0.05 g to 0.40 g produced a steady, though modest, decline in residual concentration (Table 2 and Figure 3). The improvement is attributable to the larger number of available adsorption sites. Beyond approximately 0.30 g the additional benefit became small, indicating that the system was approaching equilibrium with respect to the amount of dye present.

Table 2. Residual concentration (ppm) at different adsorbent dosages

Dose (g)	20 ppm initial	30 ppm initial
0.05	14.50	22.58
0.10	14.28	21.42
0.15	14.72	22.08
0.20	14.61	21.92
0.25	14.22	21.33
0.30	14.00	21.00
0.35	13.56	20.33
0.40	13.61	20.25

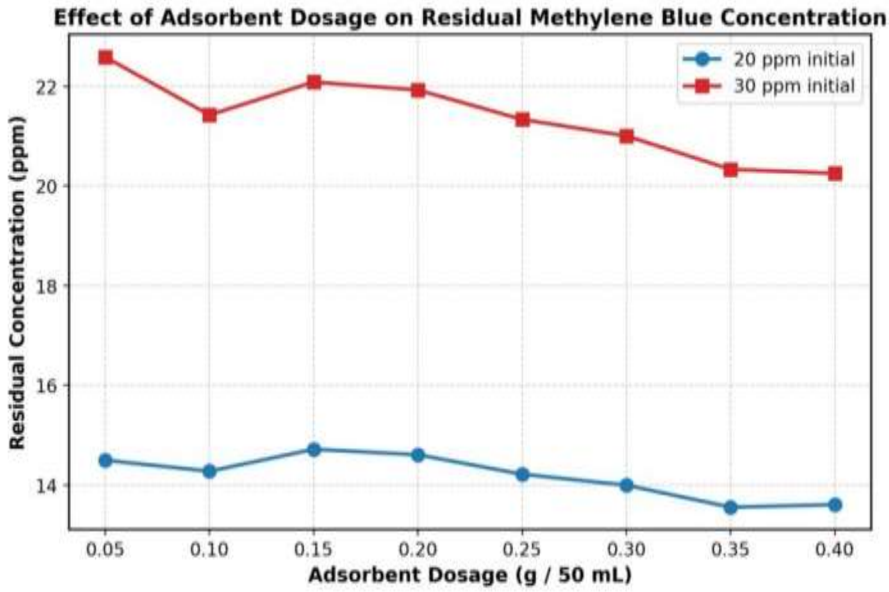

Figure 3. Influence of adsorbent dosage on residual methylene blue concentration

3.3 Effect of temperature

Raising the temperature from 40 °C to 50 °C lowered the residual concentration for the majority of the solutions examined (Table 3 and Figure 4). The improvement is consistent with an endothermic adsorption process in which higher temperature increases the mobility of dye molecules and may open additional internal pores. Similar temperature dependence has been reported for other $ZnCl_2$-activated agricultural carbons (Luyen, 2023).

Table 3. Residual concentration (ppm) at 40 °C and 50 °C (dose = 0.1 g)

Initial conc.	40 °C	50 °C
2 ppm	0.22	0.34
5 ppm	0.50	0.69
10 ppm	1.34	0.88
20 ppm	3.56	2.56
30 ppm	4.68	4.72

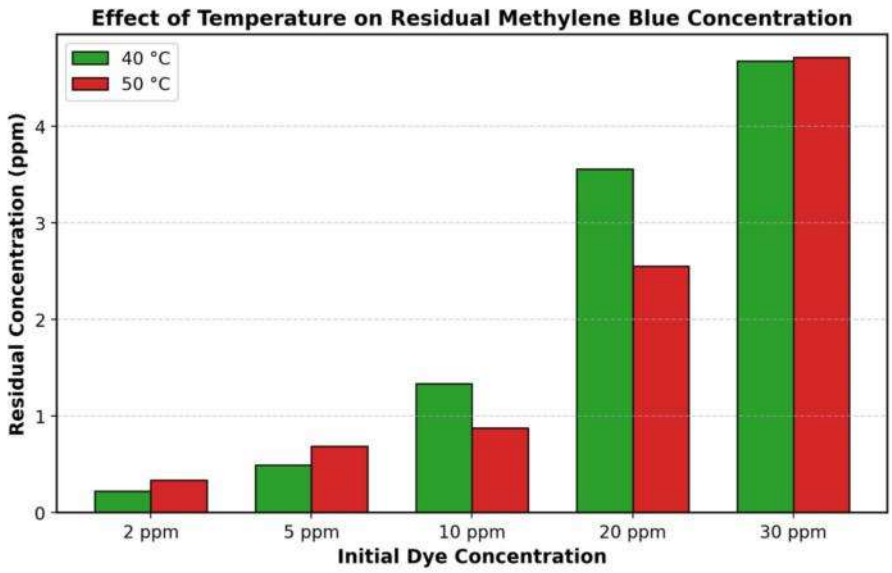

Figure 4. Residual methylene blue concentration at 40 °C and 50 °C

3.4 Effect of pH

When the pH of a 10 ppm solution was lowered by addition of dilute HCl the residual OD remained high (approximately 1.24-1.26). In contrast, addition of dilute NaOH reduced the residual OD to about 0.17. The cationic dye is therefore more readily adsorbed under alkaline conditions, when the carbon surface carries a net negative charge. This electrostatic preference is typical of methylene blue adsorption on activated carbons derived from biomass (Yetgin & Amlani, 2024).

3.5 General observations

The data as a whole indicate that BWHC is effective at low to moderate dye concentrations. At higher loadings the residual concentrations remain appreciable, suggesting that the adsorption capacity of the present preparation is limited. Further optimisation of the $ZnCl_2$ impregnation ratio and carbonisation temperature would be expected to improve performance, as has been demonstrated for related agricultural precursors (Jung et al., 2025).

4. Conclusion

Zinc chloride-activated carbon prepared from buckwheat hull removes methylene blue from aqueous solution under simple laboratory conditions. Residual concentrations calculated from optical density measurements show that removal is favoured by longer contact times, higher adsorbent doses, moderately elevated temperature and alkaline pH. The method utilises an agricultural residue that would otherwise be discarded and requires only common laboratory reagents and equipment. The results encourage further work on surface characterisation, isotherm modelling and regeneration of the spent carbon.

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