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Adsorption of methylene blue dye by carboxymethylated pearl millet (*Pennisetum glaucum*) fibres using fixed bed column method

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The demand for effective, cost-efficient, and environmentally friendly sorbents for dyes and other water contaminants has grown significantly in recent decades. In this study, an underutilised agro waste material Pearl millet straw was used to extract cellulosic fibres. The fibres were modified with monochloroacetic acid (MCA) to impart anionic groups. The effect of carboxymethylation on the physical and chemical properties of PMF was studied using Scanning Electron Microscopy, Fourier Transform Infrared Spectroscopy, and X-ray diffraction (XRD). The adsorption efficiency of native (PMF) and modified PMF (CPMF) in a fixed bed column was studied using breakthrough curves (BTC). Results show that the adsorption efficiency of MB by PMF improves significantly after treatment with MCA. The breakthrough time (t_b) increases as the column bed height is increased. However, the adsorption capacity (q_{exp}) decreases with increase in bed height. These findings indicate that the carboxymethylation of pearl millet fibres, through physicochemical changes and increased functional group availability, is a simple yet effective approach to substantially enhance their adsorption capacity for MB dye, making CPMF a promising material for cationic dye remediation in water treatment applications.

Keywords: Adsorption, Agro waste, Breakthrough curve, Carboxymethylation, Lignocellulosic fibres, Methylene blue

1 Introduction

The contamination of industrial effluents with synthetic dyes presents a significant environmental challenge, particularly in textile and related industries. Among these, methylene blue, a widely used cationic dye, is a notable pollutant due to its persistence, toxicity, and resistance to biodegradation[1,2]. Its presence in wastewater adversely affects aquatic ecosystems by reducing light penetration and introducing toxic substances harmful to human and environmental health. Conventional methods for dye removal, such as chemical oxidation, coagulation, and membrane filtration, are often costly and generate secondary pollutants[3,4]. Use of sorbents is therefore preferred in water remediation. Sorbents may be obtained from natural as well as synthetic sources. Sorbents derived from fossil-based non-biodegradable polymers, are complex and energy-intensive to produce, and they contribute to long-term environmental pollution due to their persistence in landfills and water bodies[5,6].

Among natural sorbents, polysaccharides like cellulose are particularly favoured for their abundant hydrophilic groups and widespread availability in lignocellulosic agricultural waste[7]. Consequently, there is growing interest in sustainable, cost-effective alternatives, including the use of lignocellulosic materials as adsorbents[8]. Derived from renewable biomass, these materials are abundant, eco-friendly, and possess high surface area conducive to effective dye removal. However, the native hydroxyl groups on cellulose chains limit their adsorption capacity due to weak interactions with ionic pollutants such as synthetic dyes. Chemical modification, particularly functionalization with anionic groups, has emerged as a crucial strategy to enhance the surface reactivity and adsorption efficiency of these fibres. Among various methods, carboxymethylation, that is the introduction of carboxymethyl groups (-CH_2COOH), is one of the most effective modifications. This is typically achieved by reacting cellulose with monochloroacetic acid (MCA) in an alkaline medium, often sodium hydroxide, under mild conditions[9-12].

The adsorption process, which involves the binding of dye molecules to the surface of the adsorbent, is governed by mechanisms such as Vander Waals forces, hydrogen bonding, and ionic interactions[13]. Fixed-bed column systems are particularly suited for continuous dye removal processes, where the effluent passes through a packed column of adsorbent material, ensuring high efficiency and scalability for

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industrial applications[14,15]. Breakthrough curve analysis is normally employed for optimizing fixed-bed adsorption systems, determining adsorption capacity, and predicting operational efficiency for large-scale applications.

Breakthrough curves typically exhibit a characteristic sigmoidal (S-shaped) profile with varying degrees of steepness, reflecting the adsorption dynamics in fixed-bed column operations for water and wastewater treatment[16]. The breakthrough curve theory describes the adsorption dynamics in a fixed-bed column system, representing the relationship between effluent concentration (C_t) and influent concentration (C_0) over time[17,18]. The breakthrough point (t_b) is reached when the effluent concentration first becomes detectable, while the saturation point (t_s) occurs when the adsorbent is nearly exhausted[19,20]. Among the key influencing factors, the initial adsorbate concentration, bed height, and flow rate are particularly significant[21].

The current study addresses two pressing environmental concerns: the reliance on synthetic sorbents and the open burning of agricultural residues. Specifically, it explores the potential of utilizing pearl millet fibres (PMF) an underutilized agro-waste as a sustainable alternative for dye removal from wastewater. The central hypothesis is that chemical modification of PMF via carboxymethylation using monochloroacetic acid (MCA) can significantly enhance their adsorption capacity for methylene blue, a common cationic dye found in textile effluents. The study also strives to optimise the column parameters for maximising the adsorption of MB in a fixed bed column system.

2 Materials and Methods

2.1 Extraction of Fibres from PMS

Sun-dried stalks of pearl millet (*Pennisetum glaucum*) were obtained from farms in the state of Haryana. The stalks of pearl millet were mechanically crushed to open the structure and treated with 4% NaOH for 90 min at 1:40 material to liquor ratio (MLR), at 90 °C, to extract the fibres. The extracted fibres were thoroughly washed with tap water, followed by drying till constant weight as stated in the process proposed by Yadav *et al.*[22]

2.2 Carboxymethylation of PMF with Monochloroacetic Acid (MCA)

The PMF were treated with 4 different concentrations of MCA and two different concentrations of NaOH respectively, to determine the concentrations of reagents that yielded maximum functionalization. The efficiency of modification was assessed by evaluating the water absorbency of treated samples.

For carboxymethylation, solutions of four different concentrations of MCA, namely 25 %, 50 %, 75%, and 100 % on the weight of fibres (o.w.f.) were prepared by stirring the mixture with a magnetic stirrer for 5 min at 500 rpm. 2 g samples of wetted PMF were added to each beaker. Solutions containing 50 % NaOH were prepared. Four ml of the alkali solution was added to each of the beakers containing PMF and MCA. The samples were stirred with a glass rod and kept at room temperature for 24 h, neutralized with 2 % acetic acid, hot washed, cold washed, filtered and drained. The fibres were centrifuged followed by drying at room temperature.

2.3 Testing and Characterization of Fibres

To study the effect of chemical modification, the raw fibres (PMF) and carboxymethylated fibres (CPMF) were characterised in terms of morphology, chemical composition and crystallinity.

2.3.1 Morphological Analysis

The morphological analysis of fibres was carried out by scanning electron microscope (SEM Zeiss EVO18, Germany) at 20 kV accelerating voltage. The working distance was kept within the range of 7.5 to 8.5 mm from the sample. Dried fibres were carefully mounted on a carbon tape for SEM analysis.

2.3.2 Chemical Analysis

Chemical bonds and functional groups in the treated and untreated fibres were analysed using Fourier transform infrared (FTIR) spectrophotometry, using NICOLET iS50 FT-IR. The instrument operates on the principle of directing IR radiation at certain frequencies to the sample and recording the output in the form of wavelengths because of vibrations of the chemical bonds.

2.3.3 Crystallinity Analysis

X-ray diffraction was used to examine the crystalline structure of fibres (X'Pert PRO, Netherlands, 40 kV, 40 mA). The diffraction intensities were recorded between 10° and 50° (2Θ angle range). The diffraction peak intensity (I_{002}) corresponding to primary crystalline plane (002) at 22.8° and the peak intensity at 18.0° connected to the amorphous fraction of cellulose (I_{am}) were used to

quantify the degree of crystallinity in terms of the crystallinity index (CrI) using the following formula.

$$\mathrm{CrI} = \left(\frac{I_{002} - I_{am}}{I_{002}}\right) \times 100\% \quad \ldots (1)$$

where Crl is the Crystallinity Index, I_{002} is diffraction peak intensity at 22.8° and I_{am} is the peak intensity at 18°.

2.4 Studies on Continuous Adsorption Study of Methylene Blue

Methylene blue (MB) procured from Thermo Fisher Scientific India Private Limited was used as the adsorbate. The molecular weight of MB, an aromatic heterocyclic basic dye, is 319.85 g mol^{-1},[23]. The dye has the maximum absorbance (λ_{max}) at 664 nm. A standard curve of MB was plotted using UV-visible spectrophotometer (Model: UV-2450, Shimadzu-1900i Co., Japan).

All other chemicals used were of analytical grade. Deionised (DI) water was used for all experiments.

2.4.1 Fixed Bed Column Adsorption

Fibres were packed into a syringe of 20 ml volume (2 cm diameter and 7 cm height) and compressed with a plunger to maintain a fixed bed height. Five ml of water was added slowly into the syringe to initiate a uniform flow of influent. After the fibres swell and expand after absorbing the water, they were compressed again to maintain the bed height. The packed syringe and a burette of 25 ml volume was clamped to a burette stand in such a manner that the two were completely aligned with each other with no gap between the burette nip point (outlet) and the syringe top point (inlet). This was done to minimize the contact time between the water drop and the bed of adsorbent packed in the column (syringe).

The concentration of MB in the influent was maintained at 100mg/l. The flow rate was kept at 5 ml/min and the bed height was 1 cm. In case of PMF, the effluent was collected for 1.5 h at 10 min. interval. In case of CPMF, the effluent was collected for 3 h at 10 min. intervals and for the next 3 h at 20 min. intervals. The amount of dye remaining in the filtered solution was determined spectrophotometrically.

2.4.2 Effect of Bed Height on Adsorption of MB

In this study, the initial concentration of MB in the influent was 100mg/l, the flow rate was 5 ml/min, and the bed height was varied to 1 cm, 2 cm, 3 cm. The weight of fibres needed to obtain these bed heights was 0.5g, 1 g and 1.5 g respectively. In the case of a 1 cm bed height of CPMF, the effluent was collected at every 10-min intervals for 3 h and then every 20 min for 3 h. For bed height of 2 cm, the effluent was collected at 10-min intervals for 5 h and then every 20 min for the next 4 h. For the bed height of 3 cm, the effluent was collected at 10-min intervals for 7 h and then every 20 min for the next 5 h as the time taken to reach the exhaustion point increased with increasing bed height.

2.4.3 Effect of Flow Rate on Adsorption of MB

To study the effect of flow rate on adsorption, the initial concentration of MB in the influent was 100mg/l, the bed height was 3 cm, and the flow rate was varied at 3, 5, and 7 ml/min. In case of 3 ml/min flow rate, the effluent was collected at 10-min intervals for 8 h and then every 20 min for next 6 h. In the case of 5 ml/min flow rate, the effluent MB dye solution was collected at 10-min intervals for 7 h and then every 20 min for 5 h. For 7 ml/min flow rate, the effluent was collected at 10-min intervals for 3 h and then every 20 min for 5 h. This was done as the time taken to reach the exhaustion point decreased with increasing flow rate.

3 Results and Discussion

To determine the performance of PMF and carboxymethylated fibres, their surface and morphological analysis was carried out by scanning electron microscope (SEM). Fourier Transform Infrared (FTIR) analysis was carried out to detect the chemical bonds and functional groups after carboxymethylation. X-ray diffraction analysis was carried out to study the effect of carboxymethylation on the crystallinity of fibres. Results are presented below.

3.1 Morphological Analysis

The fibrous mass obtained after treatment of pearl millet stalks with alkali comprises of long bundled fibres Fig. 1 (a) and some fibrillated fibres, Fig. 1 (b). Fibres obtained after carboxymethylation display a significantly different fibre morphology. The fibre bundles are opened, showing gaps and voids between fibres, indicating the removal of binding material lignin that holds the fibres together, Fig. 1 (c). Fibrillated fibres are also obtained for treated native fibres, Fig. 1 (b, d).

3.2 FTIR Analysis

The chemical modification of pearl millet fibres (PMF) via carboxymethylation was confirmed

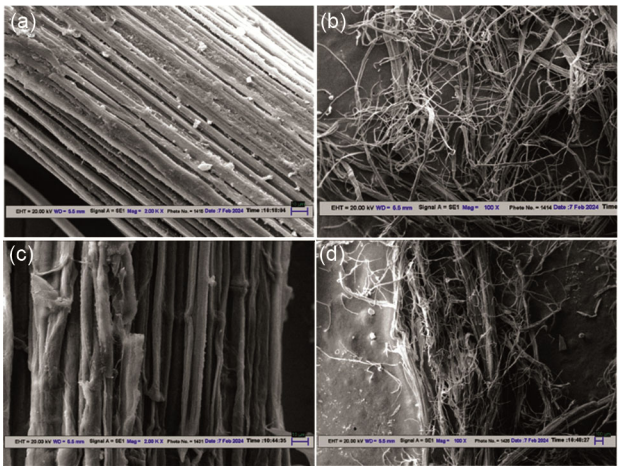

Fig. 1 — SEM micrographs (a) PMS fibre bundles, (b) PMS unicellular fibres, (c) CPMS fibres, and (d) CPMS unicellular fibres

through Fourier-transform infrared spectroscopy (FTIR). Figure 2 presents the FTIR spectra of native PMF and carboxymethylated PMF (CPMF).

A key distinction between the two samples is the emergence of a prominent absorption band around 1730 cm^{-1} in CPMF, which corresponds to the C=O stretching vibration of newly introduced carboxylic acid or ester groups. This feature provides strong evidence of successful carboxymethylation. Additionally, a band near 1600 cm^{-1} in CPMF, absent or weak in native PMF, can be assigned to asymmetric stretching of the carboxylate (COO^{-}) group, further confirming the incorporation of carboxymethyl groups. The region between 1000–1150 cm^{-1}, associated with C–O and C–O–C stretching vibrations of cellulose and its derivatives, shows increased intensity and minor shifts in CPMF, suggesting alterations in the cellulose backbone due to etherification.

Collectively, the spectral changes validate the chemical modification of PMF through carboxymethylation with monochloroacetic acid (MCA), resulting in enhanced anionic character and altered surface functionality. These modifications are expected to significantly improve the fibres' dye adsorption performance, particularly for cationic species like methylene blue.

3.3 Crystalline Structure

The crystallinity of PMF and CPMF was assessed using X-ray diffraction (XRD), Fig. 3. Both samples exhibited characteristic diffraction peaks near $2\theta \approx 16.5°$ and 22.5°, which correspond to the typical crystalline regions of cellulose I, associated with the

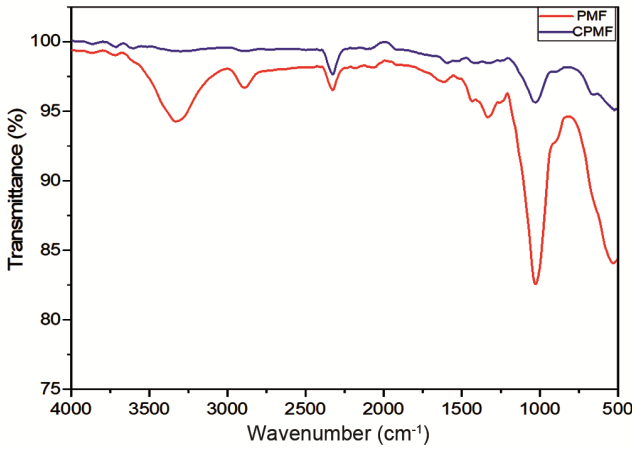

Fig. 2 — FTIR spectrum of native and carboxymethylated pearl millet fibres

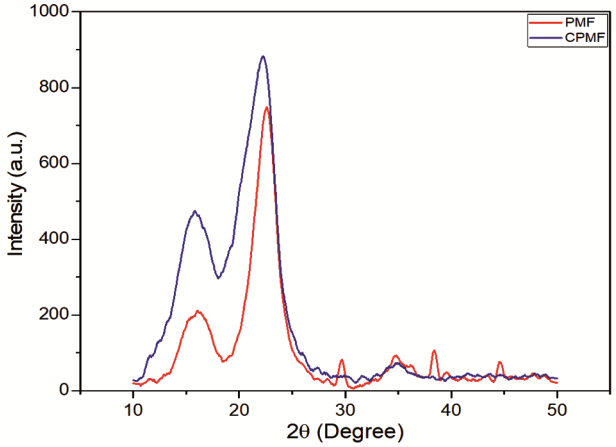

Fig. 3 — XRD spectra of PMF and CPMF

(110) and (200) planes, respectively[24]. The crystallinity of PMF and CPMF was calculated according to equation 2, and it showed 86.7 % and 66.67 % respectively. The observed reduction in crystallinity after carboxymethylation is attributed to the incorporation of bulky carboxymethyl group, which disrupt intermolecular hydrogen bonding, and hinder the tight packing of cellulose chains, thereby increasing the amorphous content. The reduction in crystallinity can be also due to the opening of structure and greater surface area for interaction.

3.4 Adsorption of MB by Fixed Bed Column

The adsorption of MB on PMF and CPMF was studied with the help of breakthrough curves (BTC). The breakthrough curves (MB-100 mg/L, 5 ml/min and bed height-1 cm) for PMF and CPMF are shown in Fig. 4. These curves represent the ratio of effluent concentration (Ct) to initial concentration (Co) as a function of time, allowing insight into the dynamic

adsorption behaviour of the materials under fixed-bed conditions. The adsorption parameters calculated from the breakthrough curves, namely the breakthrough time (t_b), the concentration at breakpoint (C_b), breakpoint volume (V_b), the exhaustion time (t_e), exhaustion point concentration (C_e), and exhaustion point volume (V_e), total amount of MB dye adsorbed (m_{ad}) and experimental adsorption capacity (q_{exp}) are reported below Fig. 4.

The breakthrough curves obtained for PMF and CPMF are significantly different. For PMF no breakthrough point could be detected while (t_e) was observed at 90 min. In contrast, CPMF exhibits a breakthrough (t_b) at ~200 min, and exhaustion (t_e), after ~350 min. This considerable shift in breakthrough and exhaustion times for CPMF indicates a marked improvement in adsorption performance following carboxymethylation.

The enhanced performance of CPMF can be attributed to the successful introduction of carboxymethyl groups, which increase the density of anionic functional sites on the fibre surface. These sites facilitate stronger electrostatic interactions with the cationic MB dye, resulting in prolonged adsorption activity and higher uptake capacity. This observation aligns with the SEM data which shows the opening of fibre bundles, the FTIR data, which confirms the incorporation of carboxylate groups, and XRD data, which suggest structural reorganization conducive to adsorption.

The broader and more gradual slope of the CPMF breakthrough curve indicates a more uniform and deeper penetration of dye molecules into the adsorbent bed, suggesting better column utilization. In contrast, the steeper curve for PMF implies rapid saturation and limited diffusion, consistent with a lower number of available binding sites. Since CPMF showed much better adsorption as compared to PMF, the next two set of adsorption experiments were carried out using only CPMF.

Sample	t_b (min)	V_b (ml)	C_b (mg/l)	t_e (min)	V_e (ml)	C_e (mg/l)	m_{ad} (mg)	q_{exp} (mg/g)
PMF	-	-	-	90	421	97.5	13.1	26.2
CPMF	90	414	4.86	360	1696	96.9	107.6	215.2

3.4.1 Effect of Bed Height on Adsorption of MB

The effect of column bed height (1 cm, 2 cm, 3 cm) on adsorption capacity of CPMF (MB-100mg/l, flow rate -5 ml/min) was studied. The BTC for the said parameters is plotted in Fig. 5 and corresponding adsorption parameters are reported in Table 1. As the column bed height increases from 1 cm to 3 cm, the breakthrough time (t_b) increases from 90 min to 210 min to 380 min respectively. However, the experimental adsorption capacity q_{exp} (mg/g) per gram of adsorbent decreased with increasing bed height, from 215.2 mg/g at 1 cm to 183.94 mg/g at 3 cm. This can be attributed to mass transfer limitations and non-uniform utilization of the deeper adsorbent layers during the shorter contact time per unit volume of dye solution.

The breakthrough time in a fixed-bed column adsorption increases with increasing bed height because a longer bed provides more adsorbent (fibres), which offers a greater number of adsorption sites. A longer bed extends the contact time between the fluid and adsorbent, enhancing the interaction. When the bed height of the fibrous adsorbent in a fixed-bed column is increased, the theoretical capacity should increase indefinitely due to more available adsorption sites. However, when increased beyond a certain limit, practical limitations may arise from channelling and uneven flow, compaction, mass transfer limitations, and non-ideal flow patterns[25]. Therefore, there is a need to use optimum bed height in fixed bed columns.

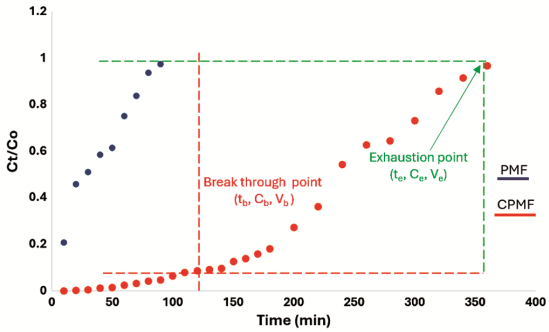

Fig. 4 — BTC and adsorption parameters for adsorption of MB by PMF and CPMF

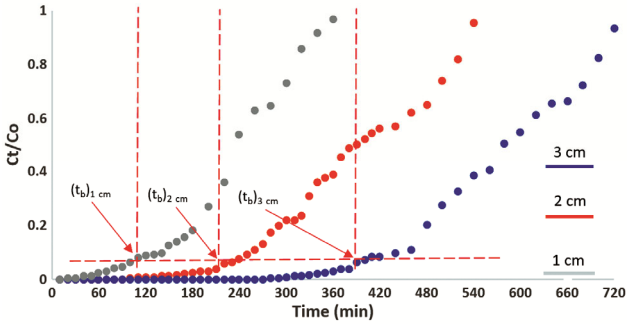

Fig. 5 — BTC of the MB for varying column bed height of CPMF

Table 1 — Effect of column height and flow rate on adsorption of MB by CPMF.

Variable	t_b, min	V_b, ml	C_b, mg/l	t_e, min	V_e, ml	C_e, mg/l	m_{ad}, mg	q_{exp}, mg/g
Bed Column Height, cm								
1 cm	90	414	4.86	360	1696	96.90	107.6	215.2
2 cm	210	1035	3.8	540	2667	95.55	191.27	191.27
3 cm	380	1840	4.1	720	3490	93.55	183.94	183.94
Flow rate, ml/min								
3	700	2050	4.9	1260	3710	96.54	303	202
5	380	1840	4	720	3490	93.55	275	191
7	130	900	4.8	480	3340	99.34	184	123

Table 2 — Adsorption capacity reported for various biomass for MB dye.

Materials	Adsorption capacity, mg/g	References
Waste watermelon rind	113.5	[27]
Pinecone	55.68	[28]
NaOH modified husk	101.3	[16]
Biochar and kaolin	20.06	[29]
PMF fibres	28.5	Present Study
CPMF	215.2	Present Study

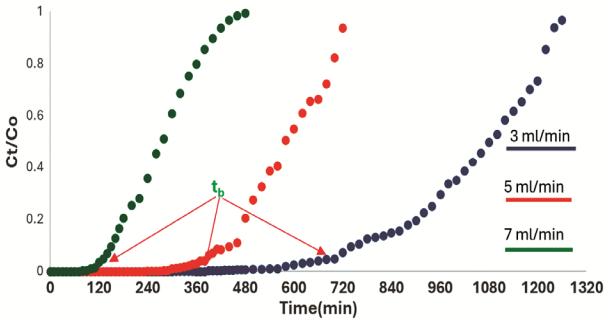

Fig. 6 — BTC for adsorption of MB by CPMF at variable flow rates

3.4.2 Effect of Flow Rate of Influent on adsorption of MB

The effect of variable flow rate (3, 5, and 7 ml/min) on the adsorption of MB by CPMF (MB-100mg/l, bed height- 3 cm) is shown in Fig. 6 and corresponding data is reported in Table 1. When the influent flow rate increases from 3 mL/min to 7 mL/min, both t_b and t_e decrease substantially — from 700 to 130 min and 1260 to 480 min, respectively. This decline indicates faster saturation of the bed due to reduced residence time, limiting the opportunity for dye molecules to interact with available binding sites. Correspondingly, the q_{exp} dropped from 202 mg/g at 3 mL/min to 123 mg/g at 7 mL/min. The reduced contact time at higher flow rates restricts intra-particle diffusion and adsorption equilibrium, resulting in incomplete dye uptake. As the flow rate increases, the breakthrough curve becomes steeper, resulting in a decrease in both the breakthrough time and adsorption capacity. Other researchers have reported similar findings in dye adsorption studies[26].

Comparative data for adsorption of MB by other fibres reported in literature is shown in Table 2. CPMF shows the best adsorption of MB indicating that carboxymethylated PMS fibres can serve as an effective adsorbent for cationic dyes.

4 Conclusion

The study shows that treatment with MCA causes significant modification in the morphology as well as chemical and fine structure of PMF. The carboxymethylation process not only improves the accessibility of active binding sites but also causes structural loosening of the cellulose matrix, enhancing dye diffusion into the fibre interior. The hydroxyl groups on cellulose are substituted by carboxymethyl groups, which significantly increase the fibre's hydrophilicity, ion-exchange capacity, and affinity for cationic dye molecules such as methylene blue. The breakthrough curves for fixed bed column adsorption of MB by CPMF show that adsorption of MB is affected significantly by the height of the fixed bed column as well as the rate of influent flow to the column. The results confirm that lower flow rates and greater bed heights improve MB dye removal in fixed-bed columns using CPMF, although these benefits are counterbalanced by reductions in specific uptake capacity q_{exp}. Optimization of these parameters is essential to maximize efficiency in real-world water treatment applications. These findings indicate that the carboxymethylation of pearl millet fibres, through both physicochemical changes and increased functional group availability, is a simple yet effective approach to substantially enhance their adsorption capacity for MB dye, making CPMF a promising material for cationic dye remediation in water treatment applications.

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