

Removal of the Cationic Textile Dye by Recycled Newspaper Pulp and Its Cellulose Microfibers Extracted: Characterization, Release, and Adsorption Studies

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ABSTRACT: *In this work, the Cellulose MicroFibers (CMF) were produced by using Recycled Newspaper Paper (RNP). The two adsorbents (CMF) and (RNP) were used in the adsorption of the basic textile dye BY28. RNP was characterized by X-ray diffraction and FT-IR spectroscopy while the morphology was visualized by SEM analysis. The chemical composition was determined by EnergyDispersive Spectroscopy (EDS); the characteristics showed a good extraction of cellulose from a newspaper pulp compared to cellulose alone. The release of RNP was followed by measuring both the absorbance and Chemical Oxygen Demand (COD) in water after washing and deinking. Alkaline pH medium has a positive effect on the release of various compounds of the pulp. The adsorption of BY28 on RNP and CMF according to various parameters such as biosorbent dose (1 - 8 g) and initial BY28 concentration (25 - 200 mg/L) was investigated. The adsorption isotherms were studied by using the Langmuir and Freundlich. Maximal RNP and CMF adsorption capacities of 91.21 and 52.67 mg/g respectively were obtained at 20 °C.*

KEYWORDS *Adsorption; Cellulose; Lignin; Paper pulp; BY28; Isotherm study.*

INTRODUCTION

Dyes are extensively employed in different industry fields like the textile, paper, food, leather, cosmetics and pharmaceutical industries [1]. About 0.7 million tons of organic dyes are produced annually all over the world,

and more than 10000 types of dyes and pigments are used in the industry [1-2]. The water polluted by the textile wastes is a serious environmental threat; its treatment uses a set of complex techniques and the purification plants

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have difficulties to control the costs. Therefore, research processes were focused on inexpensive treatments using natural materials like clays, sawdusts, agricultural wastes, biomasses, algae and activated carbons [2]. Recent works have investigated several adsorbents to adsorb dyes as rice husk [3], wheat shells [4], *Luffacylindrica* [5] and *Cerastodermalamarcki* shell [6].

Currently, the recoveries of paper wastes are mainly composed of cellulose, lignin and hemicelluloses as a low cost biomaterial for adsorption [2]. They contain alcoholic, carboxylic, sulfonic and phenolic functional groups which can absorb cationic compounds by electrostatic forces. Cellulose has become one of the potential materials in the energy production and storage, sensors, gas recovery and thermal insulation. Thermal stability and crystallinity are among the factors for reinforcing material in the development of polymeric composites and are improved by the removal of lignin and hemicelluloses from cellulose sources [7]. The demand of cotton and wood pulp as raw materials will not be affordable in the future for the formation of modified cellulose products. Thus, the research is oriented toward low cost, non-competitive and sustainable raw materials. Recently some new and underutilized raw materials like water hyacinth [8], cotton linters [9], textile waste [10], recycled news paper [11-12-7].

RNP was used to adsorb Cu (II) with a maximum capacity of 30 mg/g [13] while *Dehghani et al.* [14] have tested RNP to remove chromium (VI). Paper fibers were used for the biosorption of cationic dyes like methylene blue, crystal violet, phenylazo-4 nitrate N and N-trimethylanilinium [15], and the adsorption proportional to the amount of anionic groups on the fibers surface which have a marked affinity for these dyes for low lignin content fibers. The regenerated cellulose has been studied [16] for the removal of Ni (II) with an adsorption yield of 98%. Cellulose based cationic adsorbent elaborated by radiation grafting process tested for the treatment of AB25 and AB74 showed adsorption capacities of ~ 540 and 340 mg/g respectively [17].

The present study is devoted to microfiber cellulose CMF extraction from recycled newspaper paper RNP, and used for the elimination of BY28 by adsorption. A method of extraction of cellulose was applied, and the characterisation results are compared with pure cellulose. The washing, deinking and bleaching of RNP have been proposed to reduce the release of different organic

materials in the paper pulp. The contact time, pH, adsorbent dose and BY28 concentration were studied.

EXPERIMENTAL SECTION

Preparation of the RNP adsorbent

The recovered newspapers were cut into 2 cm × 2 cm form and washed with water at 60 °C to get RNP with a good homogeneity. Hypochlorite NaClO (5%) and biodegradable soap were used to bleach and disperse the ink and to remove dirt particles attached to RNP, the end product was dried at 80°C for 48 h.

Extraction of cellulose particles

The extraction of cellulose particle (CMF) was reported elsewhere [7, 18], wastepaper used in this work was sourced from old newspaper, cut into small pieces and boiled for more than 12 h, during which water was added periodically. It was then ground to form slurry, filtered and rinsed several times. The slurry was re-boiled and treated with 5% (w/v) NaOH followed with 2% (v/v) of NaClO. The slurry was filtered and washed with water until neutral pH was achieved. The resulting material was analyzed by ATR / FT-IR spectroscopy to confirm the presence of cellulose particles from the source material. Cellulose powder was used as reference material.

The X-Ray Diffraction (XRD) was performed in a mini diffractometer MD-10 while the morphology of the papers was studied with a Scanning Electron Microscope (SEM).

Preparation of aqueous solution of the dye BY28

Concentrations in the range (25 - 200 mg/L) were prepared by dilution in water from a stock solution of BY28 (1000 mg/L). The initial pH solution was adjusted with HCl and NaOH (0.1 M). The Chemical Oxygen Demand (COD) equipment (Brand AQUALITI) was used for the measurement in the range (0 - 1500 mg O₂/L). The concentration of BY28 was determined by UV-Visible spectrophotometry (Optizen 2120 UV) at 460 nm (λ_{max}). The adsorption capacities of BY28 adsorbed per gram of RNP adsorbent at equilibrium (q_e) and time t (q_t) (mg/g) and the percentage removal are given by the relations [19-20]:

$$q_e = (C_o - C_e) \times \frac{V}{m} \quad (1)$$

$$q_t = (C_o - C_t) \times \frac{V}{m} \quad (2)$$

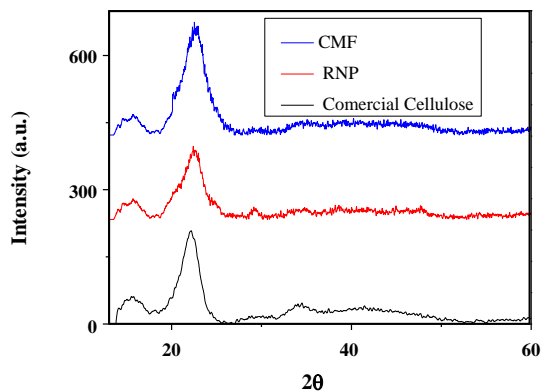


Fig. 1: XRD patterns of RNP, its extracted cellulose (CMF) and commercial cellulose.

$$\% \text{ Removal} = \frac{(C_0 - C_e)}{C_0} \times 100 \quad (3)$$

C_0 and C_e (mg/L) are the initial and equilibrium liquid phase concentrations, m the PNP adsorbent mass (g) and V (L) the solution volume. Distilled water was used in all preparations.

RESULTS AND DISCUSSION

Characterization of adsorbents

Fig. 1 shows the XRD comparative patterns of RNP, its extracted cellulose and commercial cellulose.

RNP and CMF exhibit two XRD peaks at $2\theta = 15.75$ and 22.13° similar to those of commercial cellulose; the pattern displays a typical crystal lattice of cellulose type I [7], in agreement with others [21, 22]. RNP presents also additional peaks at 29.5 and 48° attributed probably to the presence of calcite CaCO_3 [2]. Consequently, the absence in our case of commercial and extracted celluloses, show an efficiency of our delignification and extraction method.

Fig. 2 (a) and (c) shows the SEM images of RNP which reveals the presence of irregular fibres with variable dimensions ($50 - 100 \mu\text{m}$), the images also indicate that both the diameter and size of the purified cellulose fibril, have been reduced to a large extent due to the removal of lignin with other extractive substances (Fig. 1d). Fig. 2 (b) illustrates the EDS spectrum of RNP with the composition. It reveals the existence of the main elements as C, O, Al, Si and Cl which come from the industrial manufacture of paper pulp and the anchor used in the printing process; such results agree with those of Pitsari *et al.* [23] that gave

the same mineral and organic chemical composition. In Fig. 2 (d) The EDS spectrum of CMF with the composition analysis reveals the existence of the component elements as C, O, and small amounts of Al and Si compared to RNP.

Fig. 3 shows the FT-IR spectrum of RNP between 4000 and 450 cm^{-1} , shows several bands assigned to functional groups of RNP adsorbent. The bands centred at 3333 and 2900 cm^{-1} are attributed to the stretching $-\text{OH}$ groups and $\text{C}-\text{H}$ bonds of cellulose respectively.

The band at 1602 cm^{-1} is associated to the deformation vibration of OH groups of H_2O . The smallest peaks at 1505 cm^{-1} of RNP is characteristic of lignin that refers to the aromatic $\text{C}=\text{C}$ in a symmetrical plane stretching of the aromatic ring [24-25-7]; this peak is reduced after cellulose extraction. The characteristic peak at 1425 cm^{-1} is related to the presence of CaCO_3 [26-2]. The band centred at 1031 cm^{-1} is assigned to the vibration of $\text{C}-\text{O}$ elongation of cellulose and hemi celluloses while the band at 538 cm^{-1} is assigned to the vibration of the sulfonic acid group.

Evaluation of release of RNP in water

The release of RNP in water after washing and deinking was made by measuring both the absorbance and COD [2]. Mezet *et al.* [27] and Pokhrel *et al.* [28] have reported that a suspension of RNP in water can cause health problems [29]. Effectively, the release of organic such as lignin and mineral matter can modify the characteristics of the water, pH, ionic composition, turbidity, COD and colour.

Fig. 4 shows the spectra of the absorbance evolution and COD of treated and untreated RNP. The samples exhibit a peak about 280 nm specific to lignin [30]. The COD analysis shows that treated RNP exhibits an efficiency of 31 % after deinking, washing and bleaching.

Release evaluation of RNP according to pH of the solution

After elution of recycled newspaper pulp RNP with NaOH at different pHs ($2 - 12$); the dissolved lignin was absorbs at 280 nm [30]. The release of RNP in water according to pH and NaOH additions to the paper pulp was performed by measuring the absorbance. Mezet *et al.* [27] have reported that an aqueous suspension of RNP can cause problems. The release of lignin and minerals can alter the water characteristics, pH, ionic composition, turbidity, COD and color.

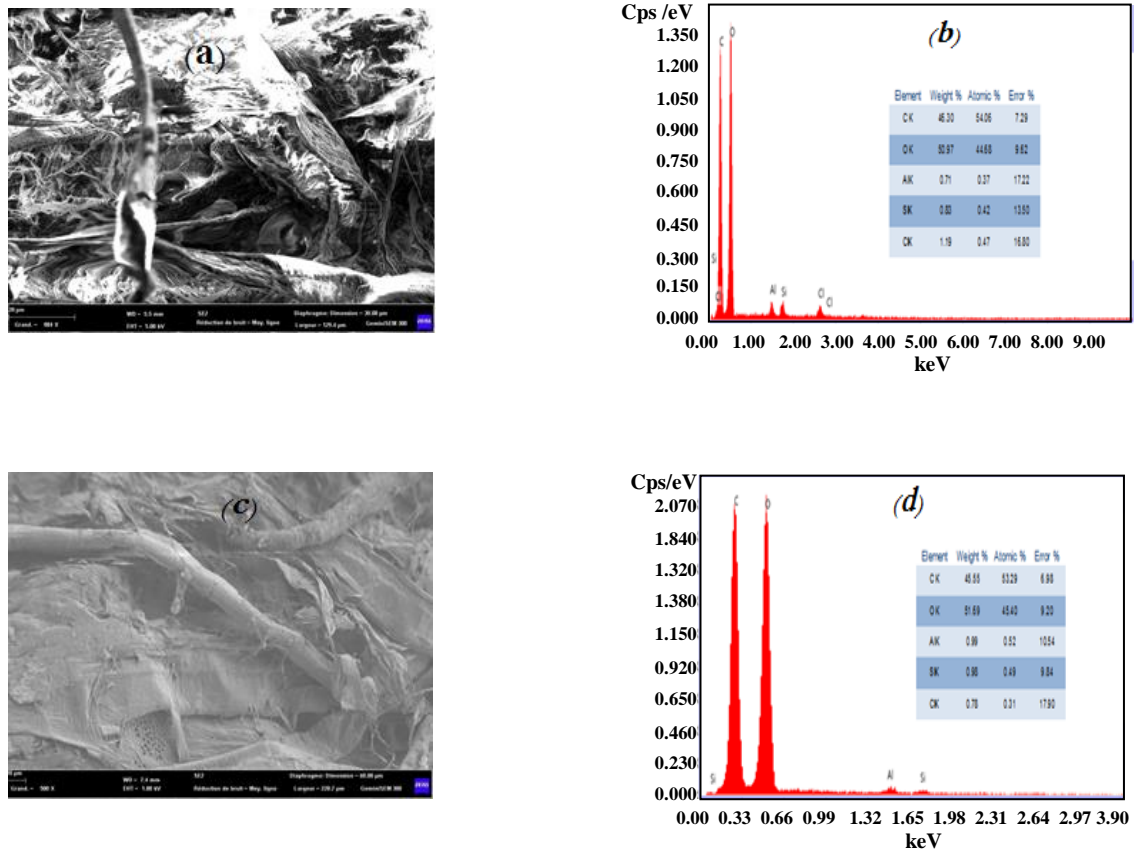


Fig. 2: (a) and (c) SEM micrograph of RNP and CMF, (b) and (d) EDS spectrum of RNP and CMF with elemental composition microanalysis.

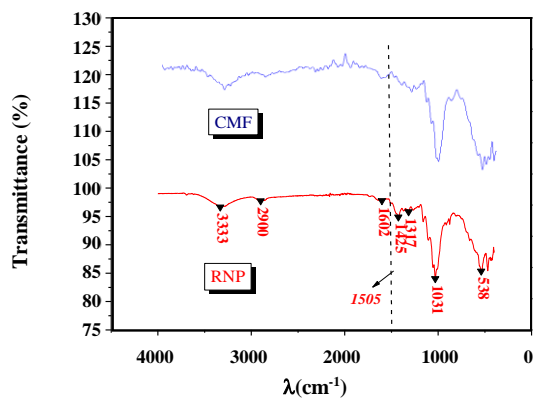


Fig. 3: FT-IR spectrum of RNP and CMF extracted.

The UV spectrum shows that release increases with raising pH and the alkaline medium promotes release of lignin which absorbs at 280 nm [30] (Fig. 5).

Effect of pH

Fig. 6 shows the chemical structure of cationic BY28 and spectra UV-Visible obtained at different pHs (2, 4 and 6). Three absorption bands are observed at 260, 335 and 460 nm and the equilibrium concentration was determined from the absorbance in the visible region (460 nm). At pH 8 and 10, one can observe the total disappearance of the three bands, indicating that the pH strongly influences the behaviour of BY28.

Fig. 7 shows the effect of the pH on the adsorption capacity of RNP; it has been observed that at basic pHs (8-10), the colour of the solution completely disappears (Fig. 6).

Therefore, we have studied the influence of pH only at non-basic values [2]. The adsorption capacity increases with increasing pH to a maximum value (pH = 4-7) and this can be explained by the competition between H_3O^+ ions and cationic molecules BY28. RNP as cellulose

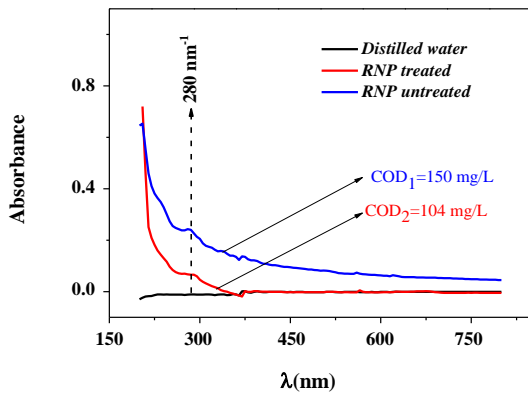


Fig. 4: Evolution of the absorbance and COD of treated and untreated RNP ($T = 20^{\circ}\text{C}$, contact time = 3h, pH 6 and RNP dose = 2 g/L).

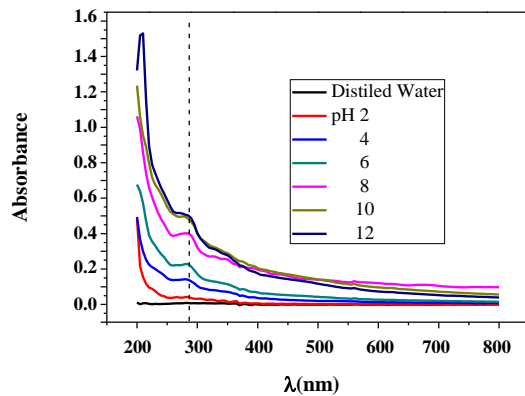


Fig. 5 : Release evaluation of RNP according to pH of the solution RNP ($T = 20^{\circ}\text{C}$, contact time = 3h, RNP dose = 2 g/L).

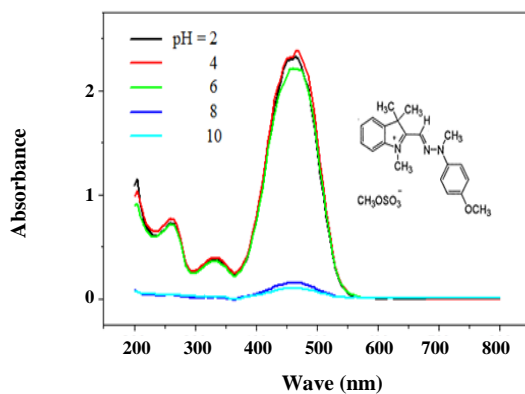


Fig. 6: Behaviour of BY28 at different pHs.

matrix contains three OH^- groups at low pH 2 [13]. The H_3O^+ ions concentration neutralizes a portion of anionic groups present on the RNP surface. When the pH increases (Fig. 7), BY28 is preferentially adsorbed onto the RNP surface.

Comparative study of BY28 dye adsorption on a pulp of RNP and its extracted cellulose

Effect of adsorbent dose

Fig. 8 shows that the removal percentage of BY28 increases with raising the RNP dose; a reverse effect was observed for the capacity adsorption. This is due to the agglomeration of RNP and CMF particles above 2 g/L, thus causing a decrease in the active sites, in agreement with the results reported elsewhere [31] where the authors have also reported that the equilibrium concentration of BY28 is lower in the presence of high adsorbent concentrations. Therefore, an optimum dose of 2 g/L is selected for the further experiments. We also noticed a significant BY 28 retention for RNP compared to CMF.

Adsorption isotherms

To optimize the BY28 adsorption for the removal, it is important to establish the most appropriate correlation and two isotherms were tested for this purpose.

The expressions of the Langmuir and Freundlich isotherms follow [32,33]:

$$q_e = \frac{q_m K_L C_e}{1 + K_L C_e} \tag{4}$$

$$q_e = K_F C_e^{1/n} \tag{5}$$

Where C_e is the equilibrium BY28 concentration (mg/L), q_e the adsorbed amount (mg/g), q_m the amount for a complete monolayer adsorption (mg/g) and K_L the Langmuir constant related to the free energy of adsorption (L/mg), K_L (L/g) the Freundlich constant and (1/n) the heterogeneity factor related to the capacity and the adsorption intensity.

The comparison of experimental data and predicted adsorption isotherms of BY28 onto NPP adsorbent according to the two models is illustrated in Fig. 9; one can see that both models fit adequately the isotherms data for RNP an correlation coefficient R^2 close to 1, compared to the adsorbent CMF with adsorption capacities of 91.51

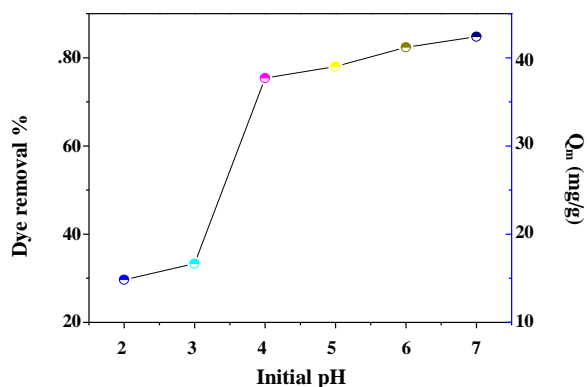


Fig. 7: The pH effect on the adsorption capacity of RNP: ($T = 20^{\circ}\text{C}$, contact time = 3 h and $[\text{BY28}]_0 = 100 \text{ mg/L}$).

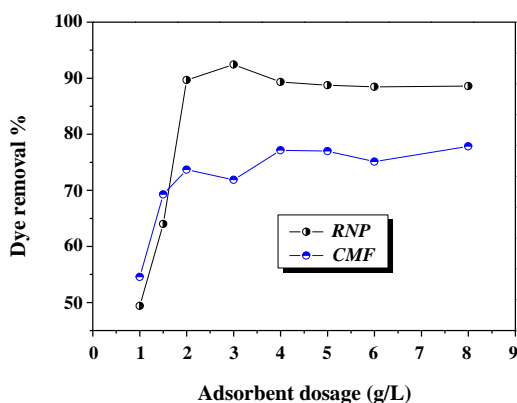


Fig. 8: Effect of adsorbents RNP and CMF doses on the percentages BY28 removal ($T = 20^{\circ}\text{C}$, contact time 3h, pH 6 and $[\text{BY28}]_0 = 100 \text{ mg/L}$).

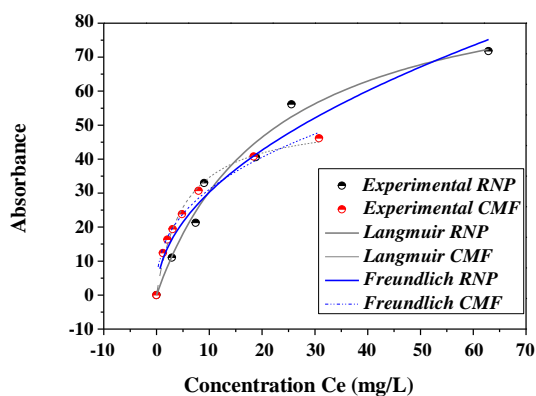


Fig. 9: Comparison of experimental and predicted adsorption isotherms BY28 dye onto RNP and CMF according to Langmuir, Freundlich models.

and 52.67 mg/g respectively (Table 1). Note also that the important BY28 retention by RNP may be due to the chemical composition of the paper pulp such as Lignin and the different chemical compound used in industrial manufacture; these elements eventually favor the retention of cationic dyes in RNP contrary to CMF which contains only one major element namely the cellulose [2].

Comparative adsorption capacities onto other material adsorbents

The maximum adsorption capacities of BY28 by various adsorbents according to the Langmuir model are regrouped in Table 2 [2].

It is instructive to outline that the adsorbents RNP and CMF have an acceptable adsorption capacity compared to other adsorbents given in the literature (Table 2). The performance of RNP as recycled material in the adsorption of BY28 is close to certain clay minerals.

CONCLUSIONS

In this work, a method for extracting cellulose from a newspaper paper was investigated. The physical characterisations showed an efficiency of delignification and extraction cellulose compared to pure cellulose.

The release of organic matter from the pulp in water like the lignin and minerals can alter the water characteristics, pH, ionic composition, turbidity, COD and colour.

The UV absorbance showed that the release increases with pH and that the release of lignin is favoured in alkaline medium

The adsorbent dose has a significant BY28 dye retention for RNP compared to CMF.

The results obtained in the present work have shown that RNP can be used as a recycled and effective adsorbent to remove the textile dye BY28, compared to other adsorbents available in the literature. The release of RNP was followed by absorbance measurements and the chemical oxygen demand (COD) in water after washing and deinking NPP, thus reducing the releases of different organic matters in the pulp suspension.

The Langmuir and Freundlich isotherms allowed us to obtain the best fit models for CMF with higher correlation coefficient. The maximum adsorption capacity of BY28 on RNP was found to be 91.51 mg g⁻¹ greater than that of CMF (52.67 mg/g), possibly due to the chemical composition of the newspaper pulp, such as lignin,

Table 1: Langmuir, Freundlich, of RNP and CMF adsorbent.

Langmuir			Freundlich		
q_{\max} (mg/g)	K_L (L/mg)	R^2	K_F (L/g)	n	R^2
RNP	91.51	0.050	10.76	2.227	0.892
CMF	52.67	0.191	12.88	2.63	0.983

Table 2: Comparison of maximum adsorption capacities of BY28 by various adsorbents.

Sorbent	Q_m (mg/g)	Ref.
Granulated clay	100–780	[34]
Bentonite	256.4	[35]
RNP	91.2	This work,[2]
CMF	52.67	This work
Boron industry waste	75	[36]
Clinoptilolite	59.6	[37]
Green macroalga	35.5	[38]
Green alga	27	[39]
Persian kaolin	16.2	[40]
Amberlite XAD-4	14.9	[37]

and the different chemical compounds used in industrial manufacturing. These chemical elements promote the adsorption of cationic dyes on RNP unlike CMF which contains a single major cell that is cellulose.

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