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Removal of brilliant scarlet by MCM-48 materials

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Abstract. Wastewater resulted from textile dyeing is an enormous pollutant around the world. Therefore, the existence of dyes, such as brilliant scarlet (BS), in the water environment, has to be removed. Mesoporous silica (MCM-48) was used to remove BS through the adsorption process. MCM-48 was hydrothermally prepared using co-surfactant of CTAB and Triton X-100 as a template. After being synthesized, the material was washed once and twice with HCl-ethanol to remove surfactants used. Fourier Transform Infra-Red (FT-IR) and X-ray Diffraction (XRD) were methods utilized to characterize MCM-48 materials. We studied the ability of washed MCM-48 materials (washed once, MCM-48-W1 and washed twice, MCM-48-W2) to remove BS from aqueous media with a variation of contact time, pH, and concentration. A UV-Vis Spectrophotometer was an instrument used for measuring the concentration of BS after adsorption at a wavelength of 505 nm. The adsorption isotherms of BS by washed MCM-48 materials were studied using Langmuir and Freundlich models. The result showed that the optimum time of BS by MCM-48-W1 and MCM-48-W2 was 60 min. The optimum pH adsorption of the dye by MCM-48-W1 was 6, whereas those by MCM-48-W2 was 5. The adsorption of BS by MCM-48-W1 and MCM-48-W2 obeyed the Langmuir and Freundlich models. The adsorption capacity of the former was higher than that of the later. This result showed that mesoporous materials that still had some surfactants are promising to be used as adsorbent of dye.

1. Introduction

The first contaminant easily recognizable in a watery environment is the color caused by dyes. Nowadays, dyes with different kinds are estimated at over 10,000 tonnes per year, but the precise amount was not known. Around 4% of them are considered reactive dyes classified as azo dyes [1]. One of the widely used azo dyes is a brilliant scarlet (BS). The dye is considered a carcinogenic dye and can cause hyperactivity in children. It is very soluble in water and produces a dark red color [2]. The existence of less than 1 ppm for some dyes in water is extremely noticeable and impacts the esthetic value, water transparency, and gas-solubility in ponds, waterways, and other water bodies [3]. Therefore, the removal of them is necessary before discharging into the water environment or on land. The removal of color from wastewaters is often more important than that of the soluble colorless organic substances.

There are several ways to remove dyes from wastewater. Among them, adsorption is a method widely used due to its simplicity and effectiveness [4]. Adsorbents used are various; natural, and synthetic adsorbents. The synthetic adsorbent has some advantages. It has various pore sizes, i.e., micro, meso, and macropore, depending on the template used to create the pore. One synthetic material that has mesopore is mesoporous silica with a three-dimension pore structure, i.e., MCM-48 [5].

Mesoporous silica (MCM-48) consists of two independent and intricately interwoven networks of mesoporous channels [6]. Therefore, it is a promising material to be used as adsorbent of a big molecule, such as dyes, because different host molecules are easier to access the pore [7]. This finding is because of the faster diffusion of adsorbates and resistance of pore-blocking [Shim, 2006]. However, after removing the template, the remaining groups in the washed MCM-48 are silanol and siloxane. We have studied the ability of the material in adsorbing azo dye (reactive red 1) and found that the interaction of the dye with the functional groups was small [8]. One explanation for this is that silanol groups in MCM-48 can interact with water by hydrogen bonding. The hydrogen bonding between silanol groups and water can inhibit the interaction between MCM-48 and the dye. Therefore, modification of silica is essential to increase the ability of the material in adsorbing dyes. Some researchers reported the use of modified silica as adsorbent of dyes. The modifying agents used were aminopropyltriethoxy silane and phenyltriethoxy silane[9]), amino silane [10]), aminopropyl silane [11], 3-Aminopropyltriethoxy silane [12], 3-aminopropyltrimethoxy silane [13], and 3-aminopropyltriethoxysilane [14]. Bevziuks *et al.* [15] used a surfactant, such as cetylpyridinium chloride as a modifying agent for silica to be used as dye adsorbent to increase the adsorption capacity.

Cationic and neutral surfactants are the template used in the synthesis of MCM-48 [7, 16]. The pores formed through the washing process with HCl-ethanol. However, the existence of the surfactant in the materials is useful in the adsorption of dyes because the surfactant has hydrophobic and hydrophilic parts. The number of washing processes can affect the amount of surfactants left in the silica materials. Therefore, in this study, we used MCM-48 washed once (MCM-48-W1) and MCM-48 washed twice (MCM-48-W2) to study the adsorption of BS from aqueous solutions at the various contact time, pH and initial concentration of dye. Mesoporous silica with more surfactants had a higher ability to adsorb the dye. The MCM-48 materials are very promising adsorbents for the dye used.

2. Methods

2.1 Materials

All materials in this study were in analytical grades, and double-distilled water is the solvent used for preparing solutions. The materials used are Ludox HS-40 (Dupont), cetyltrimethylammonium bromide, and (Triton X-100 (Sigma-Aldrich), CH_3COOH, HNO_3, and NaOH (Merck). They are used directly without any purification.

2.2 Equipment

Equipment used was an analytical balance (Ohauss), magnetic stirrer (cole-palmer), Vacuum pump (ABM type 4EK F6 3CX-4), Buchner, UV-Vis spectrophotometer (Spectronic 20 D), FT-IR Spectrometer Prestige-21 (Shimadzu), X-ray diffractometer (Rygaku).

2.3 Procedures

2.3.1. Preparing MCM-48. MCM-48 was prepared by a hydrothermal process following the previous procedure [7, 16]. We removed some surfactants by washing them once and twice with HCl and ethanol. The procedure of surfactant removal was described in the previous study [16]. After conducting the process, the white product was dried in the oven.

2.3.2. Characterizing MCM-48 materials. The X-ray powder diffraction (XRD) was conducted to characterize MCM-48 before the surfactant removal at room temperature with the use of an X-ray diffractometer with a Cu K-alpha (λ = 1,541Å) as the X-ray source. The FTIR spectra of MCM-48, MCM-48-W1, and MCM-48-W2 were taken using a Shimadzu: IR Prestige-21 FTIR Spectrometer scanned at wavenumbers ranging from 340-4000 cm^{-1} with a scan number of 300, and a resolution of 4.

2.3.3. Determining the optimum contact time. Washed materials, MCM-48-W1, and MCM-48-W2 (100 mg) were put separately into a different beaker containing 50 mL of BS solution with a concentration of 50 mg/L. The mixture was stirred with a magnetic stirrer for 15 minutes then filtered. The absorbance of the filtrate was measured by using a UV-Vis spectrophotometer at a wavelength of 505 nm. The experiment was repeated with contact time variations, 30, 45, 60, 75, 105, and 120 min, successively. Each experiment was performed twice

2.3.4. Determining the optimum pH. Washed materials, MCM-48-W1, and MCM-48-W2 (100 mg) were put separately into 16 different beakers containing 50 mL of BS solution with a concentration of 50 mg/L. The mixture was stirred at an optimum contact time and various pH (2, 3, 4, 5, 6, 7, 8, and 9), and filtered. The absorbance of the filtrate was measured by using a UV-Vis spectrophotometer at a wavelength of 505 nm. Each experiment was repeated.

2.3.5. The effect of concentration on adsorption. Washed materials, MCM-48-W1, and MCM-48-W2 (100 mg) were put separately into ten different beakers containing 50 mL of BS solution with various concentrations of 100, 200, 300, 400, and 500 mg/L. The mixture was stirred at the optimum pH and contact time. The absorbance of the filtrate was measured by using a UV-Vis spectrophotometer at a wavelength of 505 nm. We repeated each experiment.

The adsorption capacity was studied using Langmuir and Freundlich isotherms. The linear models of them are given in equation (1) and (2), respectively.

$$\frac{C_e}{q_e} = \frac{1}{Q_o b} + \frac{C_e}{Q_o} \qquad (1)$$

$$\log (x / m) = \log k + 1 / n (\log C_e) \qquad (2)$$

where x is the amount of BS adsorbed on MCM-48 materials (g), m is adsorbent mass (g), C_e is the equilibrium concentration (mg L^{-1}). Q_o and b successively indicate the Langmuir constants corresponding to the adsorption capacity (mg g^{-1}) and the adsorption coefficient (L mg^{-1}), k, and n are the Freundlich constants related to the adsorption capacity (mg g^{-1}) and adsorption intensity (g L^{-1}), respectively. Plotting C_e/qe to C_e for the Langmuir equation or log (x/m) to log C_e for the Freundlich equation will give the adsorption capacities.

3. Result and discussion

3.1. Characterization of MCM-48

Figure 1 shows the diffraction pattern of MCM-48 before washing with HCl-ethanol. The pattern shows that there is a strong peak at 2-theta of 2.11° and a weak one at 2.46°. The peaks are 2 of 8 peaks usually appear in MCM-48 indexing as 211 and 220. Therefore, the material prepared in this work was MCM-48. The other peaks do not appear in the pattern because the experiment was conducted in the instrument where there were no accessories for measuring at a low angle. The same sample with different batch showed the eight peaks, as reported in previous works [7, 17].

FTIR spectra of MCM-48, MCM-48-W1, and MCM-48-W2 are given in Figure 2. A broad peak appearing at a wavenumber of 3444 cm^{-1} is a stretching –OH vibration supported by the bending vibration of -OH at 1647 cm^{-1}. These peaks are contributions from the hydroxyl group and the physically adsorbed water by MCM-48. The stretching C-H model consists of -CH_2 symmetry at 2852

cm^{-1} and $-CH_2$ antisymmetry at 2922 cm^{-1} are from surfactants. A terminal $-CH_3$ asymmetric vibration is at ca. 2960 cm^{-1}. Peaks observed at 1471 and 1483 cm^{-1} belong to C-H bending modes. After washing with HCl-ethanol, the intensity of the C-H stretching and bending modes decrease, indicating that some of the surfactants leach. The intense peaks at 1229 and 1067 cm^{-1} and weaker peak at 957 and 795 cm^{-1} belong to the Si-O stretching vibrations of silicate lattice. After the washing process, the peak at 1067 cm^{-1} shifts to 1088 and 1092 cm^{-1} for MCM-48-W1 and MCM-48-2, indicating the existence of lattice contraction during template removal as has been reported in previous studies.[16, 17]

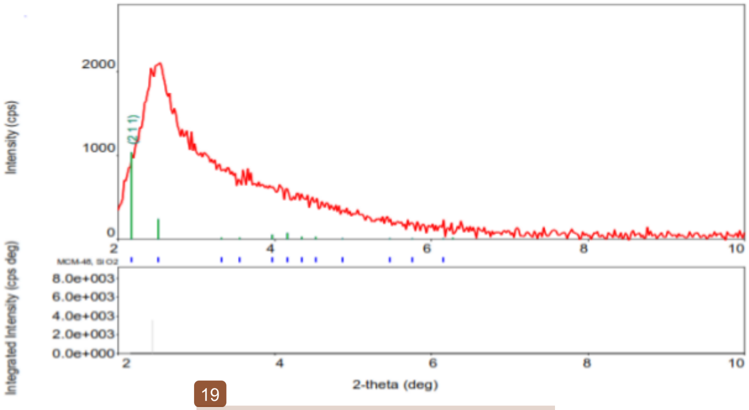

Figure 1. XRD pattern of MCM-48.

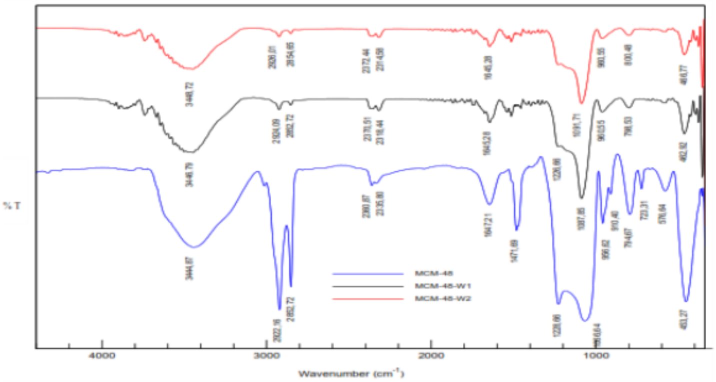

Figure 2. FTIR spectra of MCM-48 materials.

3.2. The optimum of contact time

The time needed by adsorbent to achieve the maximum amount of brilliant scarlet adsorbed is the optimum contact time. Figure 3 shows the effect of contact time on the amount of dye adsorbed by MCM-48-W1 and MCM-48-W2. It can be noted that the amount of dye removed from the solution increases as increasing the time from 15 to 60 min. After 60 min of adsorption, the amount tends to decrease (desorption process occurs), showing that the adsorption-desorption process is reversible (Putri). At 60 min, the equilibrium has achieved. Therefore, the optimum contact time for adsorption on both materials is 60 min. The optimum time was used for further experiments.

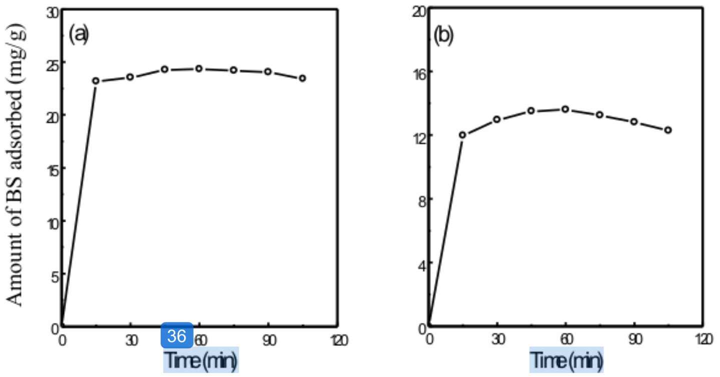

Figure 3. The amount of BS adsorbed on (a) MCM-48-W1 and (b) MCM-48-W2 as a function of the contact time.

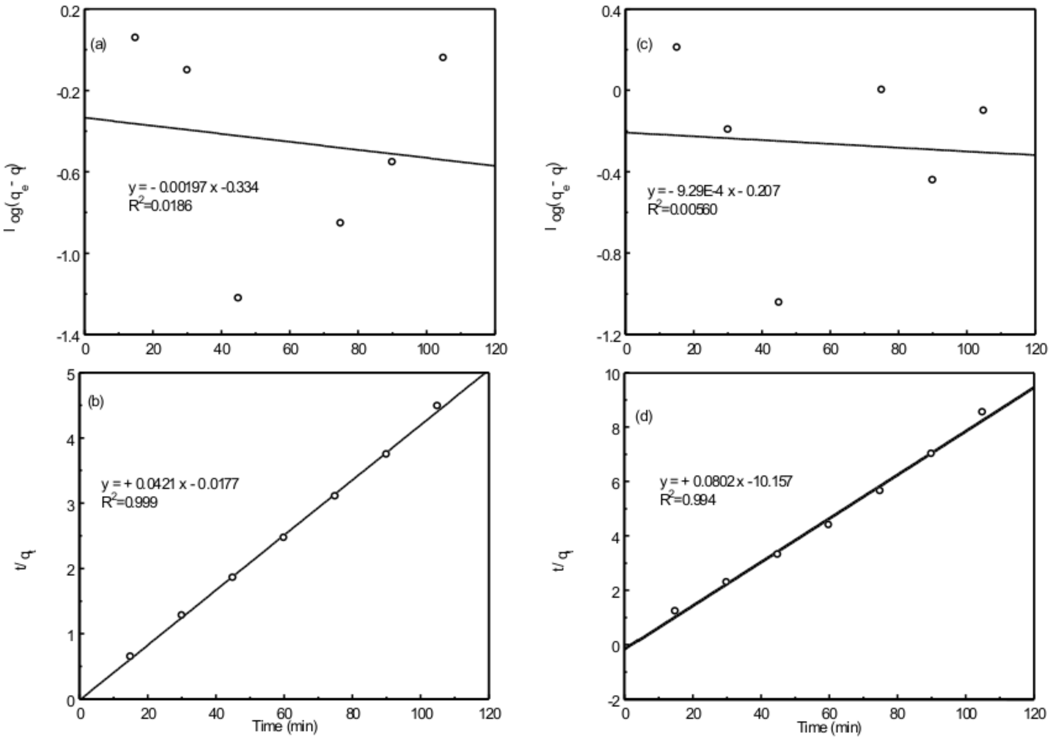

Figure 4. Kinetic study of Co(II) adsorbed using a) pseudo-first-order and b) pseudo-second-order equations.

The kinetic model in adsorption of BS was investigated by using models of pseudo-first-order, and pseudo-second-order. We used data on the amount of BS adsorbed as a function of the contact time. The adsorption pattern was obtained by the comparison in the least-squares of the equations Illustration of the adsorption kinetics model is given in Figure 4 based on pseudo-first-order and pseudo-second-order equations. Table 1 gives the values of R^2, k_1 (pseudo-first-order constant), k_2 (pseudo-second-order constant), along with the calculated and experimental q_e values.

Table 1. The value of R^2, k_2, k_1, q_e calculated from the experiments in adsorption of Co(II) ions on *E.spinosum*.

Adsorbent	Parameter	Pseudo-first-order	Pseudo-second-order
MCM-48-W1	R^2	0.903	1.000
	q_e calculated (mg g^{-1})	0.010	0.947
	q_e obtained from experiment (mg g^{-1})	0.949	
	Rate constant	0.070 min^{-1}	49.02 g mg^{-1} min^{-1}
MCM-48-W1	R^2	0.903	1.000
	q_e calculated (mg g^{-1})	0.010	0.947
	q_e obtained from the experiment (mg g^{-1})	0.949	
	Rate constant	0.070 min^{-1}	49.02 g mg^{-1} min^{-1}

The adsorption of BS on MCM-48-W1 and MCM-48-W2 follows the pseudo-second-order equation based on R^2 supported by the q_e values calculated using the pseudo-second-order equation for both adsorbents which is similar to the ones obtained from the experiments (Table 1). The pseudo-second-rate constant (k_2) calculated from the regression equation for MCM-48-W2 is higher than that for MCM-48-W1. This result may be due to the pore size of the former adsorbent compared to that for the later.

3.3. The effect of pH on adsorption

In the adsorption process, the solution pH is very important. Figure 5 gives the adsorption of BS as a function of pH.

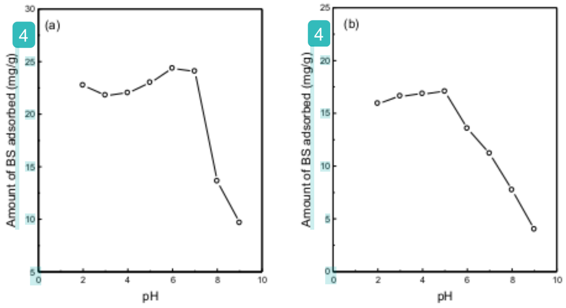

Figure 5. The effect of pH on the adsorption of BS: (a) MCM-48-W1, and (b) MCM-48-W2.

The amount of BS adsorbed on MCM-48-W1 and MCM-48-W2 at pH 2 is 22.73 and 15.92 mg $g^{-1,}$ respectively. They increase and achieve a maximum at pHs of 6 for the former and 5 for the later. The pHs were used for the experiment at various concentrations.

3.4. The effect of the initial concentration of BS on the adsorption

Figure 6 gives the amount of BS adsorbed on MCM-48-W1 and MCM-48-W2 as a function of the equilibrium concentration of the BS solution. The higher the initial concentration of BS, the higher the equilibrium concentration after adsorption, and the greater the amount of BS ions absorbed by the adsorbents. The amount adsorbed in the concentration range used in this study still increases (has not reached). As a consequence, the capacity of adsorption was studied by Langmuir and Freundlich isotherms, as shown in Figure 7.

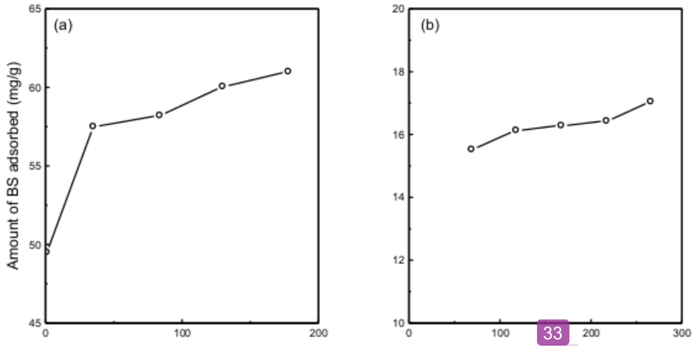

Figure 6. The effect of concentration on the adsorption; (a) MCM-48-W1, and (b) MCM-48-W2.

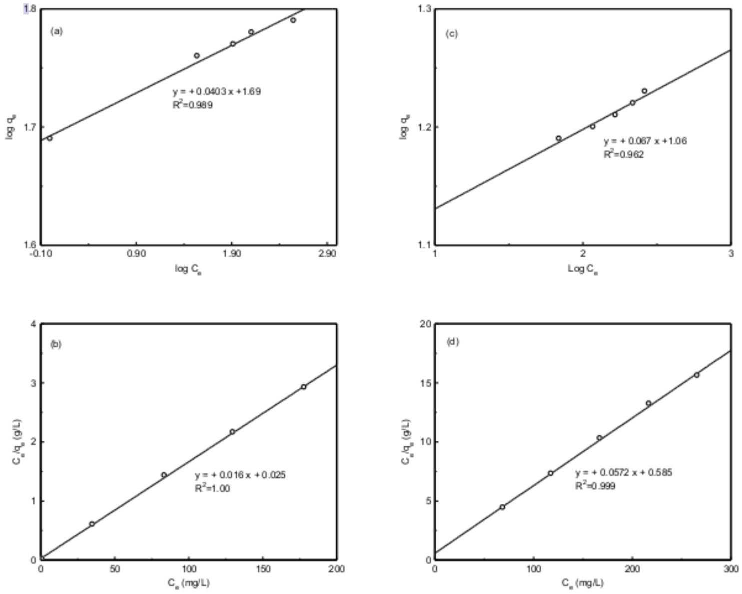

Figure 7. Adsorption isotherms for adsorption of BS; (a) Freundlich model on MCM-48-W1, (b) Langmuir model on MCM-48-W1, (c) Freundlich model on MCM-48-W2, and (d) Langmuir model on MCM-48-W1.

Figure 7 shows that the adsorption of BS on MCM-48-W1 and MCM-48-W2 fits the Langmuir and Freundlich isotherms. The Freundlich and Langmuir constants are given in Table 2. The adsorption capacity of BS using MCM-48-W1 is 3.6 times higher than that using MCM-48-W2. This finding showed that the adsorption occurred through interaction between the surfactant in the pore and the dye. The presence of surfactant can make a higher hydrophobicity to the adsorbent, increasing the organophilic attraction and the electrostatic attraction between its surface and dye molecules [9]. The amount of surfactant in MCM-48-W1 is higher than that in MCM-48-W2 supported by Figure 2, where the intensity of C-H stretching and bending modes in the later are lower than that in the former. Therefore, the amount adsorbed in the former is higher than the later.

Table 2. Langmuir and Freundlich constants in adsorption of BS on MCM-48-W1 and MCM-48-W2.

Adsorbent	Langmuir constants		Freundlich constants	
	Q_o (mg/g)	b (L/mg)	k (mg/g)	n (g/L)
MCM-48-W1	62.5	0.64	48.98	23.25
MCM-48-W1	17.54	0.09	11.56	14.92

3.5. *FT-IR spectra of MCM-48 materials after the adsorption*

Figure 8 illustrates the FTIR spectra of MCM-48 materials before and after the adsorption, along with the one that belongs to BS. The peaks before and after the adsorption are similar. There is no much shift in the wavenumber of peaks indicating that the interaction between the surfactant in the materials is physical interaction caused by the van der walls interaction and hydrophobic interaction between the surfactant and BS. This finding supported the result in the higher adsorption capacity of MCM-48-W1 compared to MCM48-W2.

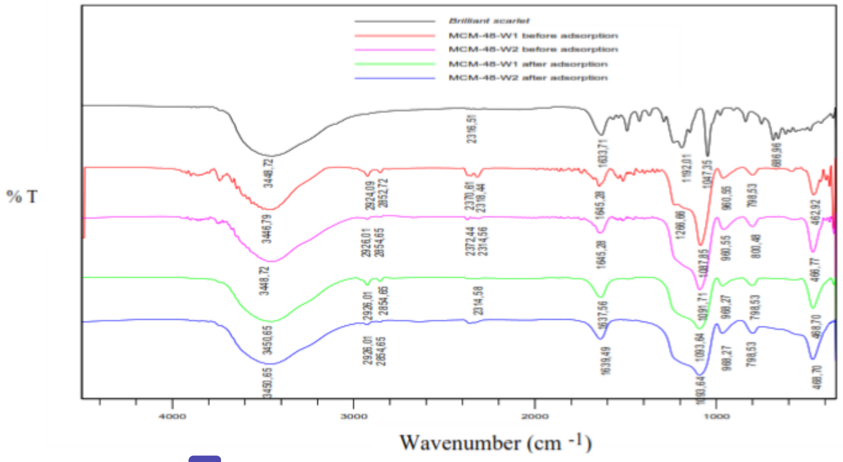

Figure 8. FT-IR spectra of MCM-48 materials before and after adsorption of BS.

4. Conclusion

The conclusions obtained from this study are as follows: the adsorption of Co(II) achieved the equilibrium after the contact time of 20 min at the optimum pH of 3. The adsorption in line with the Freundlich isotherm with an adsorption capacity of 3.46 mg g^{-1}. The kinetic adsorption follows the pseudo-second-order equation with the rate constant of 49.02 g mg^{-1} min^{-1}. The functional groups involved in the adsorption of Co(II) ions on *E. spinosum* seaweed was -OH groups.

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