

# Valorization of Spent Ground Coffee as Renewable Source to Methylene Blue Removal

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**Abstract:** - This study aims to examine the possibility of valorizing natural coffee grounds, which are rich in cellulose and hemicellulose, as renewable sources of carbonaceous material for wastewater treatment in the textile dyeing industry. On average, in Algeria, coffee consumption is 15 grams per person and day, or around 4 kilos for a year. The waste generated from coffee used in this study shows approximately a high moisture content of between 50 and 85%. The calcined sorbent was synthesized, and the sorption parameters pH and dye concentration have been optimized for methylene blue (MB) dye. The adsorption isotherm and kinetic were studied to describe the equilibrium of methylene blue (MB) adsorption. The obtained sorption data for calcined coffee grounds show an adsorption capacity for Methylene Blue (MB) removal, about 5.34 mg g<sup>-1</sup>. To confirm the adsorbent performance, the results were compared with a commercial activated carbon (CAC). The Methylene Blue (MB) sorption is favored by the pseudo-second-order kinetic model. Fourier transform infrared spectroscopy (FT-IR) characterization was carried out before and after Methylene Blue dye (MB) sorption.

**Key-Words:** - Spent coffee, Methylene blue, Carbonization, Sorption, Kinetics, Equilibrium.

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## 1 Introduction

Rapid urbanization, coupled with continuous population growth, has resulted in an increase in waste generation. According to a new World Bank report published on 20 September 2018, the amount of waste produced worldwide each year is expected to rise to 3.4 billion tonnes over the next three decades, compared with 2.01 billion tonnes in 2016, and global waste production will increase by 70% by 2050 if there is no rapid change, [1]. In order to reduce the impact of food waste accumulation generated from domestic, commercial, industrial, municipal, and agricultural activities, it is imperative to transform this waste into valuable materials to support the circular economy. A number of solutions have been suggested for removing hazardous contaminants from wastewater, using adsorbent based on carbon, [2], [3]. Recent scientific publications have demonstrated the most alternative materials derived from renewable sources for their efficiency in the removal of dyes in

different environments, [4], [5]. Coffee grounds are generally considered household waste, but they can be reused in many different ways, [6]. However, the high consumption of coffee generates a large quantity (8 to 10 million tonnes) of used coffee grounds around the world annually, [7], [8]. Several studies have examined spent coffee ground (SGC) as an adsorbent for the removal of various types of water contamination, such as lead and fluoride [9], [10], antibiotics [11], [12] and dyes [13]. The carbon content in spent ground coffee is a key factor in its potential applications as an adsorbent in wastewater treatment. According to [14], the carbon composition of spent grounds coffee is significant; it is 49.7 %, indicating that carbon is the predominant element in this by-product of coffee preparation. Methylene blue (MB) is a cationic dye with many uses in the medical, pharmaceutical, and chemical fields. It is widely used for dyeing paper, silk material, bamboo, hemp, and wood in dyeing and printing factories, [15]. This study examines the

production of calcined spent ground coffee (CSC) and its application to methylene blue dye adsorption in equilibrium and in batch kinetic tests. The adsorbents were characterized by Fourier transform infrared (FTIR) spectroscopy and their properties related to their (MB) adsorption performance.

## 2 Materials And Methods

### 2.1 Reagents

All chemicals and reagents were of analytical grade. Methylene blue (C<sub>16</sub>H<sub>18</sub>ClN<sub>3</sub>S), from CLchem LA, with a molecular weight of 319.85 g/mol.

### 2.2 Biomass Preparation

Coffee waste was collected from household coffee grounds in Algiers-Algeria. The biomass used in this study was cleaned, washed several times with tap water to remove impurities, dried in sunlight for around 24 h, and then dried in an oven at 105 °C for 24 h. To obtain (CSC), the biomass was carbonized the muffle furnace at 600 °C for four h and stored in bottles at room temperature until use. For a comparative reference, a commercial activated carbon (CAC) was purchased from Sigma Aldrich, [16].

### 2.3 Sorption Experiment

Molar solutions (HCl and NaOH) were used to control the pH of (MB) from 2 to 8, with an initial dye concentration C<sub>0</sub> of 10 mg (MB)L<sup>-1</sup>. Batch experiments were conducted at room temperature at (25 °C). For sorption isotherm, the experiment was carried out at constant pH in 50 mL with initial dye concentration C<sub>0</sub> varied between 5 to 100 mg(MB)/L<sup>-1</sup> at room temperature for 24 h. To investigate the effect of sorption time on the prepared material, kinetic sorption was conducted with a constant volume of 0.4 L of a dye solution with an initial concentration of C<sub>0</sub> (25 mg/L) and a constant dose of sorbent and stirred at 150 rpm. The effect of sorbent dosage on MB sorption was evaluated by varying the initial sorbent dosage at 1-5 g/L at constant dye concentration C<sub>0</sub>: 50 mg (MB)L<sup>-1</sup>. The sorption capacity (q, mg g<sup>-1</sup>) was calculated by a mass balance designed by the following equations:

$$q_e = (C_0 - C_e) \times V/m. \quad (1)$$

C<sub>0</sub>: Initial (MB) concentration in mg/L; C<sub>e</sub>: (MB) concentration at equilibrium. m: Mass of biomass in

g; V: Volume of dye solution in L; q<sub>e</sub>: sorption capacity at equilibrium (mg g<sup>-1</sup>).

## 3 Results And Discussions

### 3.1 Effect Of pH

The effect of pH on the adsorption of (MB) on (CSC) is depicted in Figure 1. The results revealed that the sorption of (MB) by (CSC) reaches its maximum value at pH 5, with the sorption yield of 98.16%. It was examined that the sorption efficiency increased as the pH increased after pH 5. At lower pH values, the surface charge may become positively charged, resulting in H<sup>+</sup> ions interacting with dye cations, which decreases the amount of dye adsorption. On the contrary, when the pH is higher, the (CSC) surfaces can become negatively charged, which supports the positively charged cations by the electrostatic force of attraction. Taking into account the work of [17], the PZC values of spent grounds coffee are in the range of 3.4 to 3.6, and the pH value should be maintained above 4 to ensure a predominant negatively charged surface. A similar performance on (MB) sorption has been reported in several studies, [15], [17].

### 3.2 Effect of Sorbent Dosage

From the curves, we can clearly see that increasing the dose of biosorbent has a negative impact on the Methylene blue sorption (Figure 2). An increase in the amount of biosorbent in the reaction medium has an opposing effect on sorption capacity. A decrease in the mass of the biosorbent improves the sorption capacity per unit mass of biosorbent equal to 1g L<sup>-1</sup>. These results are in good agreement with the measured data by [17].

### 3.3 Adsorption Equilibrium

To predict the types of interaction between (CSC) and methylene blue (MB), the data sorption was evaluated by fitting Langmuir [18] and Freundlich [19] mathematical models. For the Langmuir model, the linearized isotherm equations are expressed as the following equation:

$$\frac{1}{q_e} = \frac{1}{K_L q_m} + \frac{1}{C_e} + \frac{1}{q_m} \quad (2)$$

where q<sub>m</sub> is a constant reflecting the maximum sorption capacity (mg g<sup>-1</sup>), C<sub>e</sub> is the equilibrium liquid-phase concentration (mg L<sup>-1</sup>), K<sub>L</sub> is a Langmuir constant (Lmg<sup>-1</sup>) and q<sub>e</sub> is the amount sorbed at equilibrium (mg g<sup>-1</sup>).

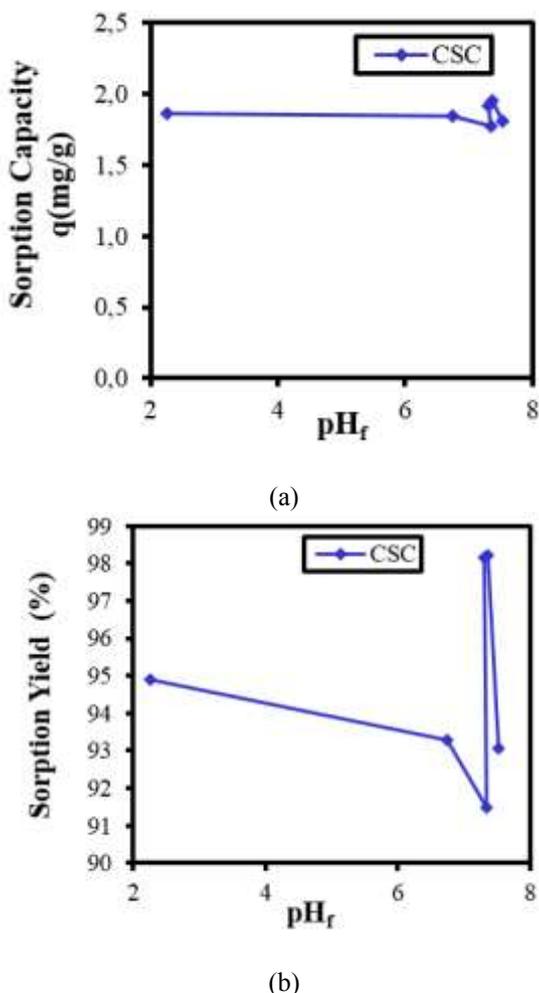


Fig. 1: Influence of pH on Methylene blue (MB) sorption using Calcined Coffee spent-grounds (CSC) adsorbent: sorption capacity (a) and sorption rate (b) ( $V$ : 50 mL;  $C_0$ : 10 mg  $MBL^{-1}$ ; Sorbent dosage, SD: 1g  $L^{-1}$ )

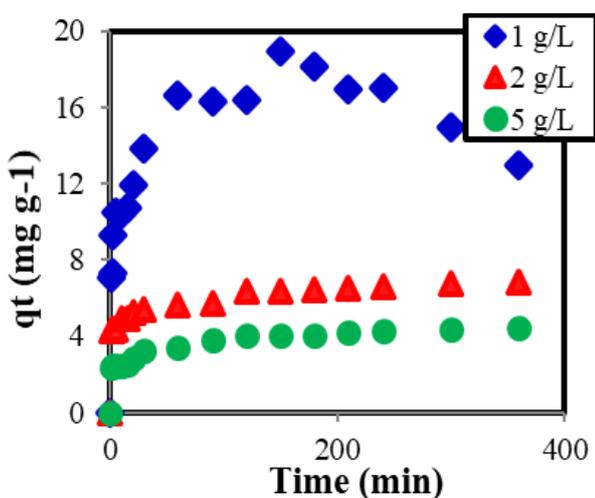


Fig. 2: Influence of Sorbent Dosage on Methylene Blue (MB) sorption using Calcined Coffee spent-grounds (CSC) adsorbent ( $V$ : 400 mL;  $C_0$ : 50 mg  $(MB)L^{-1}$ ;  $pH$ : 5;  $T$ : 25°C)

The linear form of Freundlich isotherm is given by the following equation:

$$\log q_e = \log K_F + \frac{1}{n} \log C_e \quad (3)$$

where  $K_F$  ( $(mg/g)(L/mg)^{1/n}$ ) and  $n$  are empirical constants related to the adsorption intensity which depends on the heterogeneity of material. The adsorption isotherms for (CSC) and (CAC) are shown in Figure 3 (Appendix). As can be seen, the Langmuir model fits well with the adsorption of (MB) on (CSC) with the regression coefficients  $R^2$ : 0.870 more than Freundlich  $R^2$ : 0.756. This result indicates that the Langmuir model considers adsorption to be a homogenous process and applicable for monolayer adsorption and suggests that the material has limited adsorption sites. For a more comprehensive comparison, the adsorption properties of (CSC) were compared with commercial activated carbon (CAC). The values of data fitting obtained from the adsorption of (CAC) at the equilibrium show that the Langmuir isotherm model adequately describes the sorption, with a maximum monolayer capacity of 108.696  $mgg^{-1}$  and the regression coefficients  $R^2$  of 0.957 (Table 1, Appendix).

Table 2 (Appendix) highlights a comparative study on methylene blue (MB) sorption on effective adsorbent

### 3.4 Adsorption Kinetics

Two kinetic models, Pseudo-First order (PFO) [31] and Pseudo-second order (PSO) [32], were employed respectively to predict the removal of methylene blue (MB) on (CSC) and (CAC).

The following equations describe the adsorption according to the (PFO) (4) and (PSO) (5):

$$\ln(q_e - q_t) = \ln(q_e) - k_1 t \quad (4)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (5)$$

where  $q_e$  and  $q_t$  are the equilibrium adsorption capacity ( $mgg^{-1}$ ) and the adsorption capacity ( $mgg^{-1}$ ) at the time  $t$  respectively, respectively.  $K_1$  ( $min^{-1}$ ) and  $K_2$  ( $gmin mg^{-1}$ ) are the kinetic constant respectively. The corresponding experimental kinetics parameters for (CSC) and (CAC) are given in Table 3.

Table 3. Kinetic parameters of Methylene Blue (MB) onto calcined spent ground coffee (CSC) and commercial activated carbon (CAC)

Kinetic model	Parameters	Adsorbent	
		CSC	CAC
Pseudo 1 <sup>st</sup> order (PFO)	$q_e(\text{mgg}^{-1})$	4.826	2.720
	$K_1(\text{min}^{-1})$	0.013	0.031
	$R^2$	0.911	0.771
Pseudo 2 <sup>nd</sup> order (PSO)	$q_e(\text{mgg}^{-1})$	4.650	4.830
	$K_2(\text{gmin mg}^{-1})$	0.016	0.032
	$R^2$	0.999	0.994

The determination of contact time is an important parameter for the sorption process. Figure 4 depicts the results of the kinetic experiment and illustrates that the adsorption of methylene blue (MB) on (CSC) and (CAC) achieved equilibrium in 30 min at an initial concentration of 25 mg/L. The sorption capacity obtained by (CSC) from 4.650  $\text{mgg}^{-1}$  to 4.830  $\text{mgg}^{-1}$  for (CAC) represents an increase of 1%. The kinetic results for (CSC) and (CAC) show that the correlation coefficient of (PSO) is more significant than (PFO) (Figure 4).

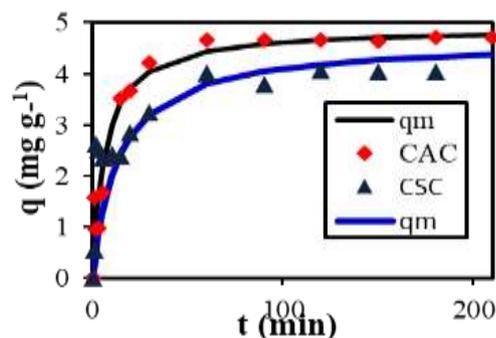


Fig. 4: Adsorption kinetics of Methylene Blue (MB) on (CSC) and (CAC) fitted by (PSO) model ( $C_0$ : 25 mg/L; pH: 5; SD: 1g/L; V: 0.400 L; T: 25°C)

#### 4 FT-IR Characterization

Figure 5 presents the FTIR spectra for (CSC) before and after (MB) sorption. The corresponding peaks before dye sorption were obtained at 1570  $\text{cm}^{-1}$ , which can be attributed to the elongation vibration of the C=C bonds. The peak at 1430  $\text{cm}^{-1}$  represents the elongation vibrations of the C-H bonds, while one at 871  $\text{cm}^{-1}$  represents the deformation of the C=C-H bond. The spectra after (MB) sorption reveal some more significant absorption located at 1500  $\text{cm}^{-1}$  associated to C=C elongation vibrations. After (MB) sorption, the peaks at 871  $\text{cm}^{-1}$  and 1430  $\text{cm}^{-1}$  disappeared totally, suggesting that these are the most involved groups in (MB) sorption. The findings of this study are similar to the one obtained

by [33] on methyl orange sorption by coffee grounds.

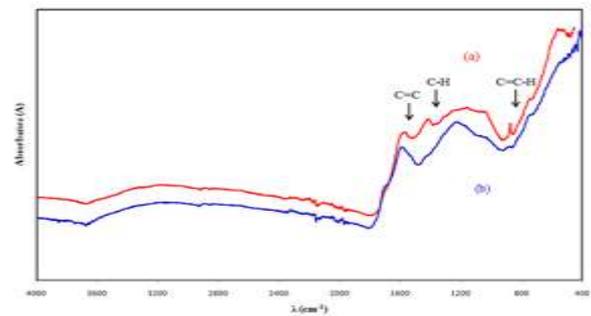


Fig. 5: FT-IR Spectra of the (CSC) before (a) and after (b) Methylene Blue (MB) sorption

#### 5 Conclusions

In this study, we synthesized calcined spent grounds coffee (CSC) by activation at 600°C for 4 h to evaluate the feasibility of (MB) sorption. Sorption experiments for (MB) removal using (CSC) and (CAC) were carried out. The maximum (MB) capacity for (CSC) and (CAC) were 5.34 and 108.69  $\text{mgMBg}^{-1}$ , respectively. It was found that the Langmuir model controlled the sorption process better than Freundlich, and the Pseudo 2-nd order model (PSO) best fitted the kinetics data. The results of this study indicate that (CSC) have potential opportunities and it would be interesting to activate it as material to improve the quality of wastewater treatment.

#### Declaration of Generative AI and AI-assisted Technologies in the Writing Process

The authors wrote, reviewed and edited the content as needed and they have not utilised artificial intelligence (AI) tools. The authors take full responsibility for the content of the publication.

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### APPENDIX

Table 1. The constants of Langmuir and Freundlich parameters on (MB) sorption by (CSC) and (CAC)

Material	Langmuir			Freundlich		
	$q_m$ ( $mg.g^{-1}$ )	$K_L$ ( $Lm.g^{-1}$ )	$R^2$	$K_F$ ( $mg.g^{-1}$ )	$n$	$R^2$
(CSC)	5.34	4.08	0.870	2.418	3.731	0.756
(CAC)	108.696	1.02	0.957	37.864	1.425	0.918

Table 2. Maximum sorption capacity of effective adsorbent on Methylene blue dye.

Adsorbent	$q_e$ ( $mg.g^{-1}$ )	Reference
Coconut shell	916.26	[20]
Tea waste	402.26	[21]
Apricot stones	3.68	[22]
Commercial activated carbon	199.60	[22]
Walnut shells	178.9	[23]
Activated chitosan	121.45	[23]
Garlic peel	82.64	[24]
Kaolin	52.76	[25]
<i>Carica papaya</i> wood	32.25	[26]
<i>Posidonia oceanica</i>	357	[27]
Rice husk	4.58	[28]
Banana peel	109.89	[29]
Orange peel	1.393	[30]
Carbonized spent coffee	5.34	In this study
Commercial activated carbon	108.69	In this study

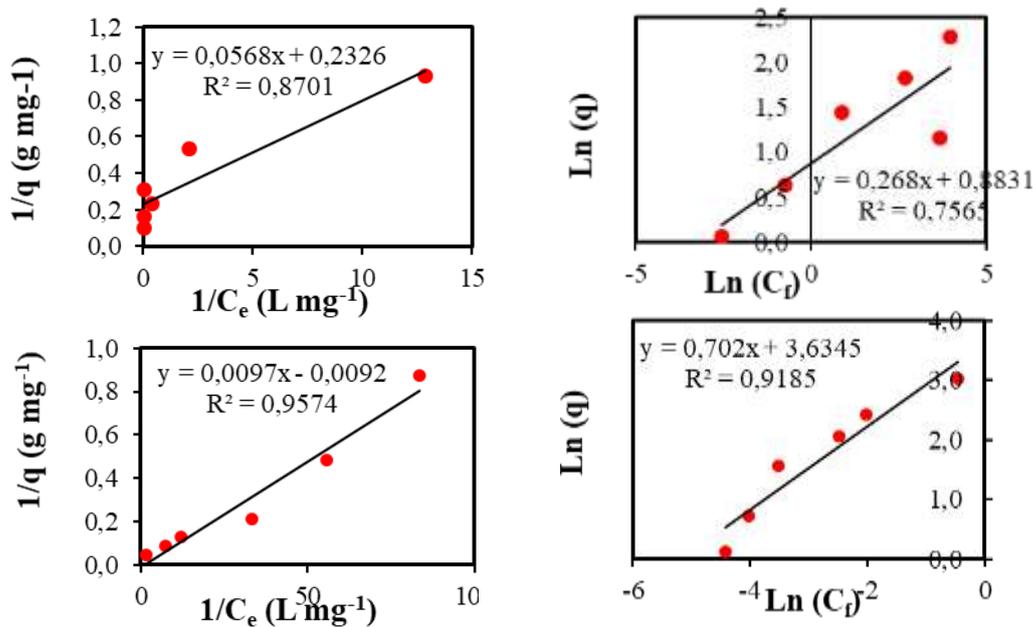


Fig. 3: Plots of the linear regression of Langmuir and Freundlich isotherm models for the sorption of Methylene Blue (MB) onto Calcined Spent Ground Coffee (CSC) and Commercial Activated Carbon (CAC)

**Contribution of Individual Authors to the Creation of a Scientific Article (Ghostwriting Policy)**

- F-N Allouche: Conceptualization-Methodology– Investigation-Writing-Original draft- Visualization.
- H. Doudou : Investigation.
- S. Hamideche : Investigation.
- A. Guezloun: Visualization.

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**Conflict of Interest**

The authors have no conflicts of interest to declare.

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