Adsorption Studies on the Removal of Textile Effluent over Two Natural Eco-Friendly Adsorbents

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1.Introduction

Water is the source of life on Earth. However, various human activities, such as industrial, domestic usage, or agricultural, cause its pollution. For instance, there are still significant quantities of dyes in wastewater coming from textile, tannery industry, sugarcane industry, and dyeing industries [1, 2]. The release of these effluents causes an abnormal coloration of the surface waters. Some researchers determined that textile wastewaters are considered to be the most polluted water because of the presence of a high amount of dyes which have a toxic effect on the environment [3], due to dyes, pH changing, biological oxygen demand (BOD), chemical oxygen demand (COD), the presence of a large quantity of suspended solids, total dissolved solids (TDS), and salts and organic compounds [4–7]. Thus, legislations controlling the use of these substances have been established in many countries. However, in various developed countries, due to the lack of water, there is a great necessity for recycling and reusing wastewater for industrial and agricultural purposes. Consequently, the textile and dyeing industry has begun seeking efficient methods for the removal of dyes. Meanwhile, there are various treatment processes that have been investigated and employed extensively for dyes elimination from wastewater [8, 9], including coagulation and flocculation [10], biological treatment using anaerobic granular [11], catalytic wet oxidation photochemical treatment [12–14], Fenton’s process, and adsorption technology. However, many of these technologies are expensive and complex when used to treat these dyes. Consequently, the adsorption technique seems to have the best potential for wastewater treatment in industry [15], thanks to its great capacity to purify contaminated water and economic aspects [2].

The aim of this study is to investigate the possibility of using two abundant natural mineral materials (collected from two Moroccan regions) as adsorbents to treat real textile wastewater (Fez city, Essabbaghine). The investigation
will focus on using the materials without pretreatment or activation to reveal their cost effectiveness compared to other treated adsorbents from the literature and to prove their eco-friendly processes. In order to accomplish this examination, physicochemical characteristics of the textile wastewater (COD, BOD, etc.) and characterization of the two natural adsorbents were highlighted. Also process key parameters optimization (adsorbent dose, contact time, dilution factor, and temperature), isotherms, kinetics, toxicity, and desorption studies are important for both adsorbents to prove their effectiveness.

2. Materials and Methods

2.1. Adsorbents Preparation. The natural mineral materials M1 and M2 used in this study were collected from the regions of Berrechid and Tiflet, respectively (Morocco) (Figure 1). The natural materials were primarily washed several times by distilled water and dried for 24 h at 100°C then milled and sieved, acquiring the average diameter of 80 μm. During the adsorption experiment, HCl (0.5 M) and NaOH (0.5 M) from Sigma-Aldrich were used to adjust the pH solution without further purification.

2.2. Adsorption Experiments. The adsorption over M1 and M2 test was carried out by using a mass of adsorbent mixed with a solution of textile wastewater at room temperature under continuous stirring. In order to investigate the kinetic of the adsorption, a 5 ml sample was collected each 10 min to measure its concentration. The adsorption efficiencies were calculated using formula (1) [16, 17], where \( C_0 \) and \( C_t \) are, respectively, the initial and the final COD concentrations (mg O₂ L⁻¹). COD was determined from the closed reflux tube method (5220D). Two equations were used to investigate the kinetic studies, number (2) for pseudo-first-order (PFO) and (3) for pseudo-second-order (PSO) [18, 19]. The Langmuir isotherm admits that adsorptions happen at specific homogeneous active sites on the adsorbent surface; (4) is its corresponding equation, where \( K_L \) is the Langmuir constant (L mg⁻¹) [20]. The Freundlich model describes the nonuniform and multilayer adsorption on heterogeneous surfaces; its model is represented by equation (5), where \( K_F \) is the adsorption capacity and \( 1/n \) represents the intensity of adsorption. The germination rate was calculated using (6) as reported in [21, 22].

\[
\text{adsorption (\%) } = \left( \frac{C_0 - C_t}{C_0} \right) \times 100 \tag{1}
\]

\[
\ln(q_e - q_t) = \ln q_e - k_1 t \tag{2}
\]

\[
\frac{t}{q_t} = \frac{1}{k_2 q_e} + \frac{t}{q_e} \tag{3}
\]

\[
\frac{C_e}{q_e} = \frac{C_x}{q_m} + \frac{1}{K_L \cdot q_m} \tag{4}
\]

\[
\log q_e = \log K_F + \frac{1}{n} \log C_e \tag{5}
\]

\[
\text{germination rate }% = \frac{\% \text{ of germination blank } - \% \text{ of germination test}}{\% \text{ of germination test}} \tag{6}
\]

2.3. Characterization Techniques. The X-ray diffraction (XPERT PRO) equipped with a detector operating at 40 KV and 30 mA with Cu Kα radiation (λ = 1.540598 Å). Infrared spectroscopy (VERTEX 70) and scanning electron microscopy (QUANTA 200) were used to identify the composition and the morphology of adsorbents materials. The X-ray fluorescence (XRF) was used to explore the chemical composition of M1 and M2.

3. Results and Discussion

3.1. Effluent Characteristics. The solution of the studied wastewater has a violet color, a sign of the presence of a significant load of dyes and suspended solids (MES). Table 1 shows that the effluent has a pH of about 6.2 and therefore does not require any neutralization. A conductivity of around 252 μs cm⁻¹ at 22°C, explained by the low presence of ions. Likewise, the effluent has a very low value of biological oxygen demand (BOD₅) which is equal to 20 mg L⁻¹ and a chemical oxygen demand (COD) of 440 mg L⁻¹. The values of these two parameters indicate that the effluent is not too loaded with organic and mineral matter [23].

The precipitation test was carried out during 48 hours at room temperature. The result (Figure 2) shows that turbidity decreases from 125 to 13 NTU.

3.2. Adsorbents Characterization. The XRD diffractograms (Figure 3) of the two sieved adsorbents indicate a distinguished mineralogical composition. We note the presence of
Figure 1: Natural raw material adsorbents M1 (a, b) and M2 (c, d).

Table 1: Physicochemical characteristics of the textile wastewater.

| Parameters                | Textile wastewater | Moroccan standards |
|---------------------------|--------------------|--------------------|
| Temperature (°C)          | 22.7 ± 1           | 30                 |
| pH                        | 6.2 ± 0.5          | 5.5–9.5            |
| Color                     | Purple             | —                  |
| MES (mg·L⁻¹)              | 3016               | 100                |
| Conductivity (µs·cm⁻¹)    | 252                | 2700               |
| Turbidity (NTU)           | 125                | —                  |
| COD (mg O₂·L⁻¹)           | 440                | 500                |
| BOD₅ (mg O₂·L⁻¹)          | 20                 | 100                |
| Ca (mg·L⁻¹)               | 0.04               | 0.25               |
| Cr (mg·L⁻¹)               | 0.24               | 2                  |
| Cu (mg·L⁻¹)               | 0.14               | 2                  |
| Fe (mg·L⁻¹)               | 1                  | 5                  |
| Zn (mg·L⁻¹)               | 1.29               | 5                  |
| As (mg·L⁻¹)               | ‘0.01              | 0.1                |
| Pb (mg·L⁻¹)               | ‘0.01              | 1                  |
| Ni (mg·L⁻¹)               | ‘0.01              | 5                  |

Figure 2: Turbidity of textile dyes from Fez city.
3.3. Adsorption of Textile Dyes. In order to study the adsorption of textile wastewater over M1 and M2, the effects of various parameters on the adsorption efficiency were investigated in this section.

The effect of the contact time on the adsorption rate was studied by putting 1 g of adsorbents in one liter of the textile wastewater solution diluted 10 times (pH 2) at room temperature. The obtained results (Figure 6) show that the adsorption of the effluent on the two adsorbents M1 and M2 goes through two stages; the first one is a rapid stage as soon as there is a strong adsorption until 61% for M1 and 46% for M2 in one hour; the second one is slow stage beyond 60 min the percentages can vary widely up to 240 min for M1 and M2. This is due to the availability of the high number of vacant adsorption sites on the surface of the two adsorbents at the initial stage of adsorption. However, the remaining unoccupied exterior sites are difficult to occupy due to the formation of repulsive forces between the effluent on the surface of the solid and those of the aqueous phase [32, 33]. These lead to a decrease in the adsorption rate, and a plateau is observed which corresponds to the state of equilibrium after 160 minutes.

To study the effect of adsorbents particles diameters, a series of experiments were performed with a constant adsorbent mass 1.5 g L\(^{-1}\), dilution factor of 10, pH 10, and different mesh sizes of adsorbents particles at room temperature. Figure 7 illustrates that decreasing particles diameters ameliorate the adsorption percentage, and the 40 μm particle diameter has the highest adsorption rate 90% for M1 and 79% with M2. This could be explained by the relation between the active surface area of adsorbents materials and adsorption efficiency [34].

The pH is an important factor in any adsorption phenomenon, since it can influence both the adsorbent and adsorbate structures as well as the adsorption mechanism. The effect of the textile wastewater pH solution on the adsorption experiment using 3 g L\(^{-1}\) as dose of M1 or M2 and concentration dilution factor of 10 at room temperature was studied. The pH range of the solution was adjusted from 2 to 12 using HCl (0.5 M) and NaOH (0.5 M). It has been observed that adsorption percentage increases by raising the dye pH solution, where it reaches its maximum 93% and 86% for M1 and M2, respectively, at pH 10 (Figure 8). This behavior could be explained by the following: (i) at acidic pH, H\(^+\) ions will compete with dyes molecules, which reduce the adsorption efficiency. (ii) At basic pH, this competition decreases and the active sites become more negatively charged on M1 and M2 surface, which enhances the adsorption by electrostatic attraction forces [35]. In addition, we can conclude that the adsorbents’ surfaces are negatively charged up to a pH 6.5 and 7.3 for M1 and M2, respectively, and positively charged below these values (Figure 9).
The effect of textile wastewater concentration was studied over the adsorbents M1 and M2. A concentration dilution was varied in a range of 0–10 as a dilution factor using 1 g of adsorbents, pH 6 particle size 80 μm at room temperature. Figure 10 illustrates the adsorption evolution of textile wastewater over both adsorbents at different concentrations. The results revealed that adsorption efficiency decreases from 77 to 23% and from 64 to 14% with a

![Figure 4: Infrared spectrum of both adsorbents.](a) ![Figure 4: Infrared spectrum of both adsorbents.](b)

![Figure 5: SEM images of M1 and M2 adsorbents.](a) ![Figure 5: SEM images of M1 and M2 adsorbents.](b)

![Figure 5: SEM images of M1 and M2 adsorbents.](c) ![Figure 5: SEM images of M1 and M2 adsorbents.](d)

Table 2: Chemical composition of sieved clays (<80 μm) using XRF (%).

| Samples | SiO₂ | Al₂O₃ | Fe₂O₃ | K₂O | MgO | Na₂O | CaO | TiO₂ | P₂O₅ | LOI* |
|---------|------|-------|-------|-----|-----|------|-----|------|------|------|
| M1      | 53.6 | 26.5  | 5.61  | 2.1 | 2.05| 1.5  | 0.67| 0.84 | 0.18 | 6.58 |
| M2      | 42.6 | 11.0  | 2.32  | 1.3 | 3.29| 0.82 | 18.8| 0.44 | 0.24 | 18.7 |

*LOI: loss of ignition at 1000°C.
decrease of concentration factor dilution from 10 to 0 for M1 and M2 adsorbents, respectively. This rate evolution could be explained by the low dispersion of pollutants’ molecules into the adsorbents due to their high concentration. Furthermore, the high concentration of adsorbate saturates the adsorbents’ active sites, which decreases the adsorption percentage [36].

The effects of M1 and M2 dosage on the adsorption of textile wastewater (pH 10, 40 μm, and 10 as dilution factor) were shown in Figure 11. The results indicate that the efficiency increased from 46 to 94% and from 30 to 86% when the adsorbents mass increased from 0.25 to 1.5 g·L⁻¹ for M1 and M2, respectively. These observations are in accordance with the increase of the active site on the adsorbents surface [37]. However, the wastewater concentration remains constant, whereas the adsorbents dose up to 2 g·L⁻¹ which means the saturation of the adsorbents surface.

Despite the fact that the effect of temperature on adsorption (pH 6, 80 μm, 1 g of adsorbent, and 10 as dilution factor) has been carefully studied using a sand bath to stabilize the temperature, no universal law has been found. Indeed, these studies have shown that an increase in temperature can lead to an increase in the adsorbed molecules amount. As shown in Figure 12 at higher temperature, the adsorbents lead to higher adsorption efficiency. As a result, enough energy ameliorates the adsorption fixation of pollutants molecules on the M1 and M2 active sites, that could be explained by the fact that the temperature creates new active sites capable of adsorbing more dyes molecules [38].
The adsorption treatment of textile dyes was tested through applying the optimal conditions found after it had been studied: adsorbent mass 1.5 g·L⁻¹, 10 as dilution factor, 328 K in temperature, 40 μm as particles diameters, pH 10, and the contact time of 90 and 160 minutes for M1 and M2, respectively. Figure 13 illustrates the evolution of the adsorption efficiency using M1 and M2 adsorbents. The adsorption percentage achieved 100% during 90 minutes for M1 and 160 minutes for M2.

It has been shown that textile wastewater was adequately treated at pH 10, which may be due to the formation of surface hydrogen bonds between the hydroxyl groups on both adsorbents’ surface and the functional groups of textile dyes as suggested in Figure 14. The large number of different functional groups on the adsorbents surface (e.g., carboxylic and hydroxyl groups) implied the existence of many types of adsorbent–adsorbate interactions. Moreover, the desorption studies show that adsorbed textile dyes onto M1 and M2 form a stable chemical bond between the adsorbents’ surface and the pollutants’ molecules, which prevented the dye molecules from being eluted from the adsorbents’ surface using just water or heated water. However, small amounts of dyes molecules were eluted (~20%) using NaCl and NaOH, and a high desorption was achieved with acidic agents. Figure 14 illustrates the electrostatic attraction between the dyes compound and adsorbents materials. This
phenomenon makes both materials suitable to adsorb more quantity of dye. At acidic condition, however, the presence of H⁺ will make dyes competing for ionic interact with the adsorbent [39].

3.4. Kinetic Study. The pseudo-first-order and pseudo-second-order for adsorption kinetics over M1 and M2 were evaluated. Table 3 and Figure 15 show that the values of the pseudo-first-order (PFO) $R^2$ were 0.872 and 0.803, and the
values of the pseudo-second-order (PSO) $R^2$ were 0.999 and 0.997 for M1 and M2, respectively; thus, $R^2$ of the PSO was bigger than that of the (PFO), implying that the pseudo-second-order kinetic was the main kinetic process as long as it gives the best prediction for the kinetic data, and the adsorption capacity variable ($q_e$) calculated is very close to the experimental capacity [16, 40].

Isotherm models (Langmuir and Freundlich) were studied to explain the adsorption system at equilibrium. All isotherms are executed with a variation of textile wastewater facture dilution 10. The results of adsorption isotherms show that the adsorption process of textile dyes onto M1 and M2 adsorbate fitted more strictly the isotherm Langmuir equation (4) than the Freundlich equation (5). This result is based on the higher value of $R^2$, which is considered as a measure of the excellent fit of experimental data on the isotherms models. The best isotherm that fits with both materials adsorptions is Langmuir. Thus, this process is a nonideal sorption on heterogeneous surfaces and multilayer sorption [16, 41]. The corresponding data of the two models are presented in Table 4.

### 3.5. Desorption Reaction and Adsorbents Regeneration

Figure 16 reveals the effects of various chemical agents as eluents on the desorption efficiency. The desorption using 0.1 N HCl was optimum (89% for M1 and 78% for M2) followed by $H_2SO_4$ (0.1 N), $H_3PO_4$ (0.1 N), NaOH (0.1 N), and NaCl (0.1 N) [42]. The elution efficiency was further investigated at various HCl concentrations (Figure 16). This might be due to the deterioration of adsorption site on M1 and M2 surface in the presence of HCl agent.

The regeneration of both adsorbents is an important step in order to check the economic feasibility of the adsorption process. The regeneration studies were carried out using 0.1 N HCl solution as it gives optimum desorption elution (89.1%).

The regeneration studies were carried out in batch for five successive cycles using 200 mL of textile wastewater (Figure 17). Results showed that a decrease in the adsorption might be caused due to the decomposition by acidic solution to certain adsorption sites or functional groups present on M1 and M2 surface [33, 43].

### 3.6. Characteristics of the Treated Wastewater

After the adsorption on the two natural materials, the treated textile wastewater was characterized to determine the performance of the treatment. Consequently, we paid particular attention to the dosages of heavy metals Cd, Cr, Cu, and Zn and of chemical oxygen demand (COD). The values obtained (Table 5) indicate an elimination of 88 and 79% of the COD for M1 and M2, respectively. In addition, the concentration of heavy metals decreases in the case of both materials. This achievement could be explained by the capacity of both materials to adsorb organic matter and metal ions present in the wastewater studied. The results summarized in Table 5 confirm that M1 is more efficient than M2.

### 3.7. Germination Test

In this test, corn kernels were germinated with wastewater before and after adsorption in order to evaluate their toxicity and possible reuse. The results described in Table 6 make it possible to observe the effect of all samples (after and before adsorption treatment) on the germination rate of corn kernels compared to blank (potable water). 15 grains were used per experiment; they were arranged identically in Petri dishes and incubated in a culture chamber for 24 hours. 5 ml of each solution (water, S1, and S2) was added which corresponds to $T_0$ of imbibition then 5 ml of each solution was added every 24 hours of incubation at 13°C for 6 days. The percentage of germination is noted every 24 hours. Figure 18 shows that in 6 days the germination of corn kernels using distilled water was successfully done. Moreover, the germination rate using treated effluent (S1) with both M1 and M2 materials achieved 97.8% in 6 days, which confirm a good adsorption yield. Unlike untreated solutions, the germination rate does not exceed 20%. As the toxicity depends on the concentration of pollutants in the solutions, the germination increases at low pollutants concentration [21, 44]. These results confirm the possibility of reusing the treated textile wastewater to irrigate plants in urban gardens.

| Material | Langmuir isotherm parameters | Freundlich isotherm parameters |
|----------|-------------------------------|--------------------------------|
|          | $q_m$ (mg·g$^{-1}$) | $K_L$ (L·g$^{-1}$) | $R^2$ | $n$ | $K_F$ (mg·g$^{-1}$) | $R^2$ |
| M1       | 321.43                        | 0.173                     | 0.994 | 3.303 | 27.04          | 0.606 |
| M2       | 357.6                         | 0.321                     | 0.992 | 2.125 | 18.74          | 0.571 |
4. Performance Comparison with Literature

The efficiency of the adsorption towards different dyes ef-
fluents according to literature studies is presented in Table 7 [8, 18, 45–48] in which we have involved the results of the present work as well as the necessary conditions to establish comparisons. Table 7 confirms that the present work shows an important adsorption of dyes during two hours. Consequently, Moroccan materials could be a promising adsorbent for the elimination of dyes. However, it was evident from the studies of textile wastewater adsorption, that it would be interesting and it is currently in progress in our lab to activate those materials which can be a promising material for wastewater treatment.
5. Conclusion

In this study, the ability of using both natural materials M1 from Tiflet and M2 from Berrechid areas as adsorbents has been explored. Without pretreatment or activation, both materials have been used and proved effectiveness to remove dyes from real textile wastewater samples of Fez city. In addition to the advantage of their availability in large quantities, they present an eco-friendly alternative to traditional processes of wastewater treatment, even though the test of adsorption–desorption cycles demonstrate that both of them cannot be used several times, they are still cost-effective adsorbents, taking into account the high adsorption yield reached. Another advantage of using these two materials is the possibility of removing different types of organic compounds and heavy metals having an attractive potential on the surface of the adsorbents. It would be interesting to continue testing on real wastewater not only batch processes but also column processes in pilot scale.

Data Availability

The authors confirm that all data underlying the findings of this study are fully available without restriction.

Conflicts of Interest

The authors declare that they have no conflicts of interest regarding the publication of this paper.

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| Adsorbent | Real rejection | Adsorption (%) | Reference |
|----------------|----------------|----------------|-----------|
| Clay from the Fouchana region (Tunisia) | Dyeing industry | (i) 97.0% BOD₅ (ii) 95.0% COD | [8] |
| Powder (α-NLP) | Textile effluent | (i) 98.9% BOD₅ (ii) 100% COD | [45] |
| Activated charcoal | Textile effluent | (i) 81.0% BOD₅ (ii) 87.6% COD | [46] |
| (i) Alum | Textile effluent | 92% COD | [47] |
| (ii) Activated carbon | Textile effluent | 85.0% turbidity 60.0% color in 30 min | [18] |
| (iii) Alum + activated carbon | Textile effluent | (i) 60.0% COD | [48] |
| Graphene oxide (GO) | Textile effluent | 85.0% turbidity 60.0% color in 30 min | [18] |
| Synthetic compounds based on iron/aluminum | Textile effluent | (i) 60.0% COD | [48] |
| Natural material (M1 and M2) | Textile effluent | (i) M1: 88% COD (ii) M2: 79% COD | Present work |

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