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Removal of Cr (VI) from aqueous solution using olive tree bark as natural adsorbent

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Abstract:

The study examined the adsorption behavior of olive bark as a low-cost natural adsorbent to removeCr^{+6} ionsfrom aqueous solution by packed bed column technique (PBC). The effect of particle size on the adsorption of Cr^{+6}ionsusing two batches, raw and modified olive bark was investigated based on Box – Wilson design. Using different pH and contact time ranging from5-8 and from 0.25-5 hr. respectively. The best performance was achieved with small-particle-treated olive bark, which showed a removal efficiencyof 84.2% for Cr^{+6} ions at pH 5.4 and contact time 4 hr. Statistical study showed significant differences in removal efficiency at the level of probability ($p \leq 0.05$). The adsorbent was characterized using Fourier transform infrared spectroscopy (FTIR) and Field Emission Scanning Electron Microscope (FE-SEM). In the analysis of the infrared device (FTIR), the results show that the sites for adsorption are related to hydroxyl, carbonyl, aromatic, and ether groups. FE-SEM analysis showed the morphological changes on the olive bark surface before and after treatment of chromium aqueous solution.

Keywords: Agricultural Waste, Heavy Metals, standard aqueous solution, Water Pollution

Introduction

Global freshwater demand has been growing quickly as human populations and economies rise [1]. As industrial waste, a large number of dangerous heavy metals have been released into the environment, causing serious solid and water pollution [2]. Every day, wastewater has been released from a variety of sources, including homes, industrial and agricultural, with a wide range of compositions and enormous hydraulic loads[3]. The water-soluble forms of heavy metals are regarded as developing water contaminants. When these heavy metal levels exceed permissible limits, they can have detrimental health effects and risks to both people and animals[4]. In addition to having a detrimental effect on soil quality, heavy metal poses a health risk to humans due to bioaccumulation [5]. Bioaccumulation is the intake of heavy metals and their cumulative concentration in the organism [6]. There are two sources of heavy metal pollution in soil, natural due to geology, and others related to human activities. Activities such as fossil fuel burning, pesticides and fertilizer in agriculture, and sewage waste are some of these main sources of heavy metal pollution[7]. Because of their qualities of non-biodegradability, bioaccumulation, environmental stability, persistence, and biotoxicity, heavy metals (HMs) constitute a serious environmental hazard to living things and their ecosystems [8]. Cr (VI) pollution can have severe consequences for water and the soil environment, many human pathophysiological abnormalities, such as allergic responses, burns, sores, especially in the stomach and small intestine, anemia and affect different biological systems [9]. Numerous treatment techniques, including flocculation, membrane technology, ion exchange, chemical precipitation, coagulation/flocculation, and electrochemical techniques, such as biological techniques and phytoremediation, have been utilized for water and wastewater remediation. They are associated with drawbacks such as expensive disposal processes, increased chemical usage,high energy consumption,significant sludge/secondary pollutant generation, and sensitivity circumstances of operation. In conquering these disadvantages, adsorption techniques have shown to be more appropriate, useful, and effective for the removal of hazardous metals from wastewater because of their quick kinetics, high uptake capacities, selectivity, simplicity, and efficiency [10]. To simplify the wastewater treatment process in addition to being efficient and quick, it's critical to find adsorbents like bark that are reasonably priced and impose a high efficiency of adsorption. Thus, the demand for inexpensive adsorbents emerged. Adsorbents of low cost consist of those non-conventional materials. They arereadily accessibleand mostly made of industrial and agricultural waste [11]. Agricultural waste (peels, nutshells, rice husk, wheat straw, maize bran, etc.), forest waste (bark, leaves, and seeds), animal waste (chicken feathers, eggshells, and crab shells, etc.), and industrial waste (fly ash, red mud, and furnace slag, etc.) are the categories into which these low-cost adsorbent materials can be divided [12].The use of bark-derived chemical compounds could help to solve environmental and economic problems at the same time [14]. Olive tree bark was used in this research to meet the above-mentioned specifications. Furthermore, it contains many active compounds, such as phenols, tannins, and lignans, in addition to its abundance. [13]The aim of this study was to discover the adsorption potential of olive bark to removeCr (VI) from aqueous solution.

Material and Methods

Preparation of Chromium standard solution

An adsorbate standard solution of 50 mg/L (ppm) of Chromium was prepared by dissolving 0.14 g of K2Cr2O7 in 1000 ml of D.W. and served as the standard solution for experiments. The concentration of this solution was measured by atomic absorption spectroscopy before and after treatment. [28]

Preparation of Adsorbent

Olive bark was obtained from the University of Baghdad, Al-Jadriyah. (Fig. 1) for this study, two batches of olive bark pieces were to be prepared. The first batch consists of untreated, raw bark pieces. An agricultural scissor was used to cut the bark strips from an olive tree into two sizes: big pieces measuring 8–10 mm and small pieces measuring 4-6 mm. (Fig. 2) After washing the sample with distilled water, they were dried for two hours at 100 °C in an oven. For the second batch, the strips of bark were also cut into small and big pieces and washed with distilled water. But then they were treated with 0.1 M HCL for 1 hour, rinsed with distilled water, and treated with 0.1 M NaOH, after treatment with chemicals, they were dried in the oven at the same temperature and time duration. [18]

Fig. 1: Collected Raw olive bark.

Fig. 2: Different particle sizes of olive bark.

Adsorption experimental procedure (Adsorption experimental studies)

Prior to each adsorption experiment, distilled water was used to clean all three glass columns. The two batches of olive barkare packed into 3/4 of the length of the column for the treatment of chromium aqueous solution. (Fig. 3)each column has a certain pH of chromium aqueous solution and contact time, then collected the chromium solution in test tubes to measure its concentration after treatment by using atomic absorption spectroscopy according to figure 4. [29] The adsorption of Cr (VI) was also characterized by Fourier Transform Infrared Spectroscopy (FTIR) and Field Emission Scanning Electron Microscope (FE-SEM).

Fig. 3: Laboratorial Column Design

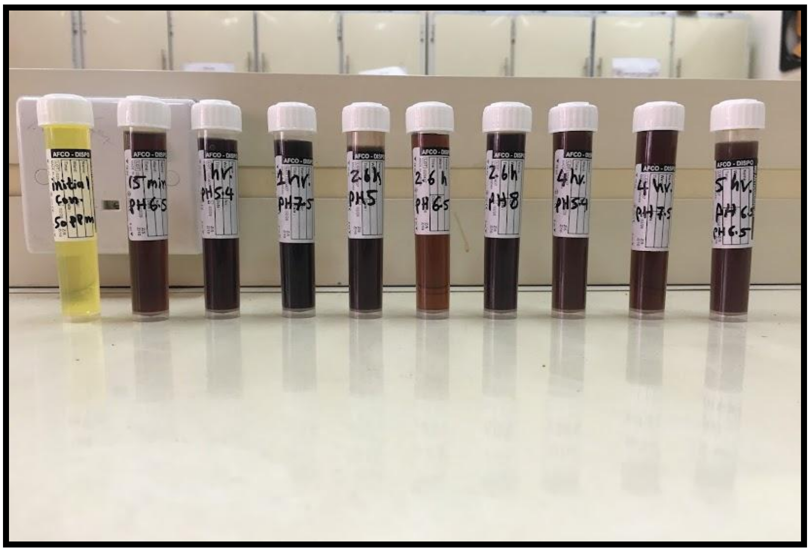

Fig 4**:**Initial and collected chromium standard solution in test tubesafter treatment with different pH and times

Parameters affecting adsorption of heavy metals by plant waste adsorbent

pH value effect

The experiment was conducted using the Box-Wilson design with a pH range of 5-8 for the chromium aqueous solution. Both the surface charge of the adsorbent and the protonation/deprotonation of the adsorbate are impacted by pH [15].

Contact time

According to the Box-Wilson design, the impact of contact time for the removal of heavy metals by adsorbent was calculated at various times between 0.25 and 5 hours. There are three phases to the Cr (VI) reaction process on adsorbents: rapid, diffusion, and equilibrium. Because the material has a porous structure, a large number of surface functional groups, and active sites, which promote the quick adsorption of Cr (VI) by electrostatic force attraction [16].

Particle size of plant waste

Two particle sizes of adsorbents were used for the removal of heavy metals, 4-6 mm and 8-10 mm, at pH=5-8 and contact time 0.25 – 5hrs. *Eze et al 2013*who observed greater adsorption capabilities were associated with smaller particle sizes. This could be explained by the higher available external surface that has smaller particles with a constant mass overall[17].

Adsorbent modification

The modification caused by 0.1 M HCl and 0.1 M NaOH is also another crucial factor that plays a role in the effectiveness of olive bark. Agricultural wastes that are raw or unaltered typically have a low adsorption capacity for pollutants because of their underlying chemical composition and structure. The limited specific surface area of industrial wastes and the mixed chemical structures that make up agricultural wastes restrict their chemical and physical interactions with various contaminants [18].

Calculations

The heavy metal removal rate was determined using the formula ER%=$\frac{Co-Ce}{Co}\times 100$ whereER% is the removal rate expressed as a percentage, C_0 is the initial concentration of the heavy metal, and $C_e$is the equilibrium concentration of the heavy metal after adsorption. Both C_0 and C_e are expressed in mg/L[20].

Results and discussion

The concentration of chromium was 50 ppm then after treatment, the concentration of Cr^{+6} ions showed different lower values (less than 50 ppm), but the lowest concentration (7.9 ppm) when treated small particles of olive bark was used at pH 5.4 for 4 hours contact time as shown in table 1. The selection of this range of pH values, which ranged from acidity to basicity, is to determine the optimal conditions and the best pH value for the adsorption and removal process of Cr (VI).

Table 1: Concentration of Cr^{+6} ions after treatment with olive bark at different pH and contact time.

Time and pH	Small Raw	Small Treated	Big Raw	Big Treated
0.25 hr. pH 6.5	24	18.85	25	19.75
2.6 hr. pH 6.5	16.95	15.35	17.85	16.25
2.6 hr. pH 5	20.1	13.6	21.2	14.5
2.6 hr. pH 8	19.95	16.4	20.85	17.3
4 hr. pH 7.5	15.1	11.95	16.75	12.7
4 hr. pH 5.4	11.7	7.9	16.25	9.75
5 hr. pH 6.5	15.4	12.3	16.25	13.5
0.95 hr. pH 5.4	22.4	21.95	22.95	21.4

0.95 hr. pH 7.5	18.95	17.9	19.4	18.45

The removal efficiency was better (84.2 %) when treated small particles olive bark was used at pH 5.4 with contact time 4 hours as shown in table 2.

Table 2: Percentage of removal to Cr^{+6} ions after treatment with olive bark at different pH and contact time.

Time and pH	Small Raw	Small Treated	Big Raw	Big Treated
0.25 hr.pH 6.5	52 %	62.3 %	50 %	60.5 %
2.6 hr. pH 6.5	66.1 %	69.3 %	64.3 %	67.5 %
2.6 hr. pH 5	59.3 %	72.8 %	57.6 %	71 %
2.6 hr. pH 8	60.1 %	67.2 %	58.5 %	65.4 %
4 hr. pH 7.5	69.8 %	76.1 %	66.5 %	74.6 %
4 hr. pH 5.4	76.6 %	84.2 %	67.5 %	80.5 %
5 hr. pH 6.5	69.2 %	75.4 %	67.5 %	73 %
0.95 hr. pH 5.4	55.2 %	56.1 %	54.1 %	57.2 %
0.95 hr. pH 7.5	62.1 %	64.2 %	61.2 %	63.1 %

The effect of pH and contact time on removal efficiency by olive bark is illustrated in Fig.5

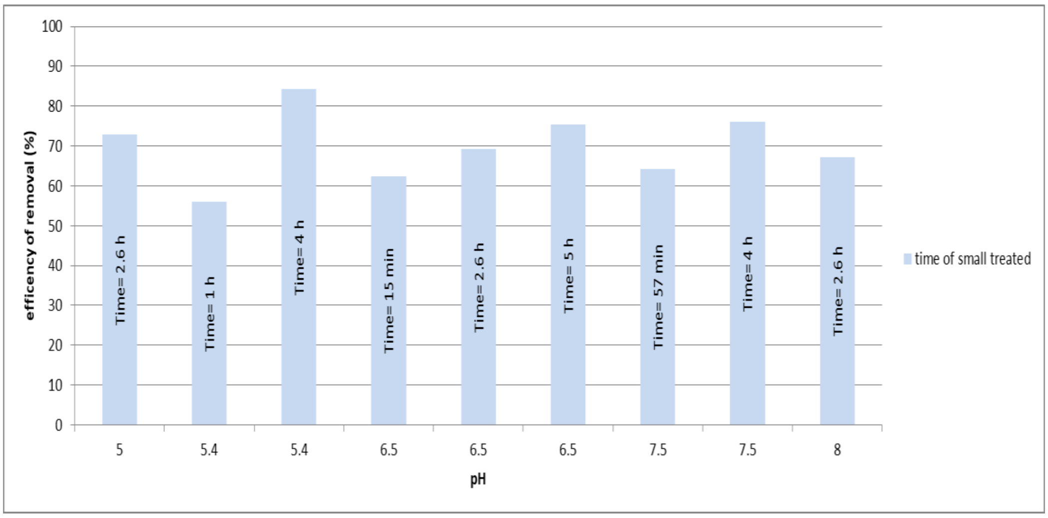

Fig 5**:** Effect of pH and contact time on removal efficiency by olive bark.

This finding is agreement with *Muthulakshmi, A., & Baskaran, R. (2017)*who studied the effect of pH on adsorption of Cr (VI) ions by P.dulce bark. and hence the effect of pH on different time intervals and adsorbent concentration was studied. It can be observed that the adsorption increased with increasing solution pH. Maximum adsorption was observed at pH 5. [21] Also, *Kareem, M.K. (2016)*used olive leaf powder for adsorption of Cr (VI) ions from aqueous solution and the best removal efficiency at pH=4[22].

*El-Shinnawy, A. (2013)*Studied the effect of contact time on Cr (VI) and Pb (II) adsorption on water hyacinth leaves powder was investigated to study the rate of Cr (VI) and Pb (II) ions removal. It is easily seen that the removal percentage of Cr

(VI) and Pb (II) metal ions increased with increasing the contact time[23]. Also,*Pant BD et al., (2022)* studied the effect of contact time on adsorption of Cr (VI) on chemically modified arecanut leaf sheaths (CALS). Adsorption of Cr (VI) ions onto CALS increases with an increase in time[24]. *Castañeda-Figueredo JS et al., (2022)*studied the effect of particle size and concluded that the bioadsorbent's particle size influences how well it adsorbs metallic ions; in most of the peels, the bioadsorbent with smaller particles removed more Cr (III) and Pb (II)[25]. *Onyi Mathias Lawrence (2019)*concluded that the percentage of adsorption was 91.346 % at the lowest particle size of 45 µm and 88.936 % at the largest particle size of 180 µm[26].

Statistical analysis

In the comparison between raw and treated small particles, the results of statistical analysis showed no significant differences depending on P-value that was 0.118 because $P \leq 0.05$ as shown in table 3. While in comparison between raw and treated big particles, the results of statistical analysis showed significant differences depending on P-value that was 0.038 because $P \leq 0.05$ as shown in table 6. And the comparison between effect of pH and Time on raw and treated small particles showed significant differences depending on P-value which was 0.022 because $P \leq 0.05$ as shown in table 5.

Table 3: Comparison between raw and treated small particles.

Group No.	Mean	S.D.	F-value	P-value	Note
1	18.283	3.868	2.725	0.118	Not Significant
2	15.133	4.219			

Table 4: Comparison between raw and treated big particles.

Group No.	Mean	S.D.	F-value	P-value	Note
1	19.611	3.125	5.129	0.038	Significant
2	15.955	3.698			

Table 5: Comparison between effect of pH and Time on raw and treated small particles

GroupNo.	Mean	S.D.	F-value	P-value	Note
1	21.425	3.641	4.262	0.022	Significant
2	16.15	1.131			
3	25.85	8.131			
4	18.175	2.510			
5	13.525	2.227			
6	9.8	2.687			
7	13.85	2.192			
8	22.175	0.318			
9	18.425	0.742			

Fourier transform infrared spectroscopy (FTIR) analysis

The FTIR is a crucial instrument for identifying the distinctive functional groups in the adsorbent that have the ability to adsorb metal ions. FTIR spectra were used within the 400–4000 cm^{-1} wave number range [30]. The functional groups found in olive bark were identified using FTIR. The FTIR spectra of Cr^{+6} ions before and after sorption are shown in (Fig 6 a) and (Fig 6 b) respectively. The olive bark before adsorption showed a number of peaks regardingdifferent functional groups. The broad band observed at 3436.91 cm^{-1}, 3429.20 cm^{-1} indicates (O-H) stretching vibrations of the hydroxyl group. In turn, the band at 2921.96 cm^{-1}: was considered as C-H stretching vibrations from aliphatic hydrocarbons with the methylene (-CH_2-) and methyl (-CH_3) groups. [31] The band observed at 1739.67 cm^{-1}: indicated C=O stretching vibration, suggesting carbonyl groups, possibly from esters or carboxylic acids. The peaks observed at 1645.17 cm^{-1} and 1510.16 cm^{-1} is the stretching vibration of C=C and (C=C) bonds of aromatic rings indicative of aromatic rings likely from lignin. [27] The bands at 1107.06 cm^{-1} and 1062.70 cm^{-1} showed stretching vibrations of C-O and C-O-C respectively, indicating cellulose or hemicellulose content. The peak at 883.34 cm^{-1} was corresponded to aromatic C-H bending. After adsorption, the FTIR spectra analysis of olive bark revealed several shifts in key functional groups, including O-H, C=O, C=C, C-O, and C-O-C. These shifts suggest that hydroxyl, carbonyl, aromatic, and ether groups in the olive bark are actively involved in the adsorption of Cr^{+6} ions.

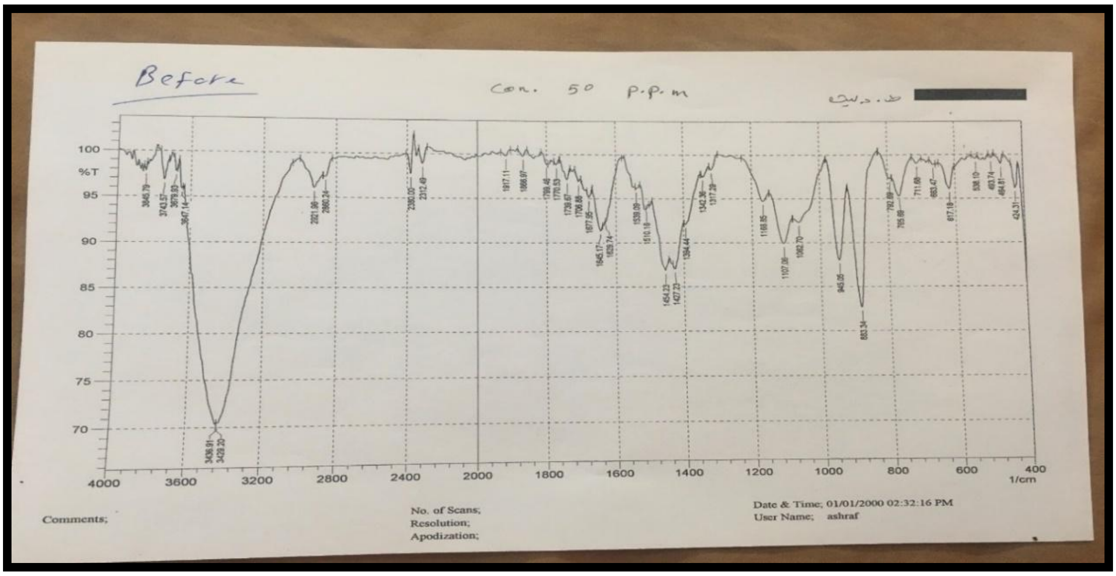

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***Fig 6 a*:** Olive bark FTIR before treatment.

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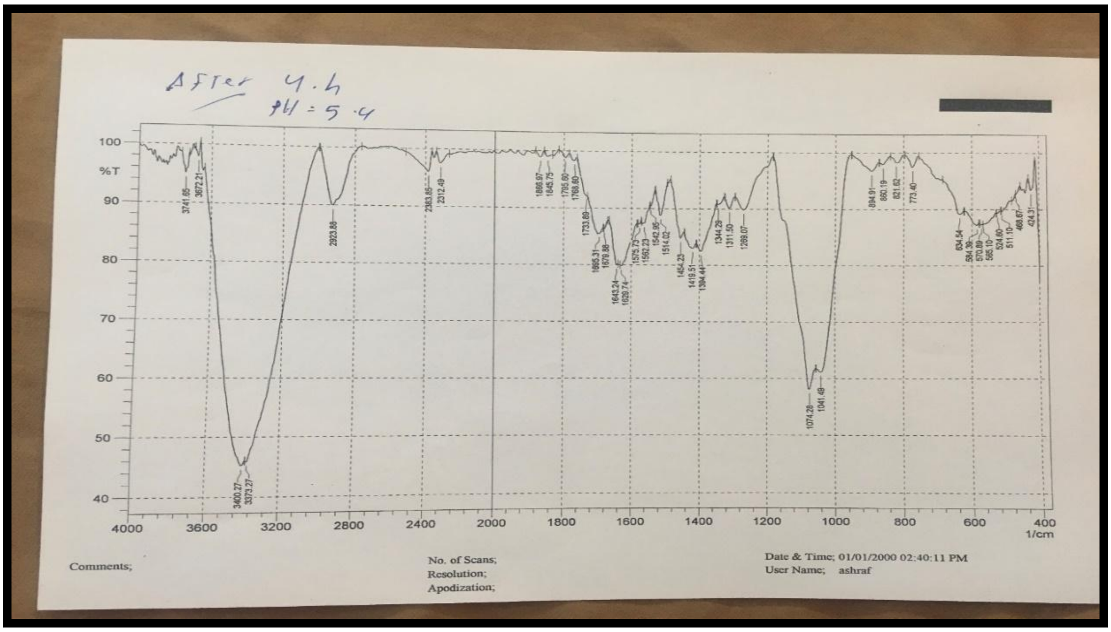

Fig 6 b:Olive bark FTIR after treatment.

The Field Emission Scanning Electron Microscope (FE-SEM)analysis

An approach for examining an adsorbent's surface structure both before and after adsorption is called FE-SEM. It is also used to look into the adsorbent's appropriate size distribution, porosity type, and particle shape. (Fig7 a) shows the FESEM scan of the adsorbent surface, revealing the presence of irregularly shaped particle agglomerates. After adsorption, the surface becomes shiny and smooth with filled pore structures (Fig 7 b) most likely as a result of physic-chemical interaction between the Cr^{+6} ions and the functional groups on the surface [27]. After adsorption, the surface's shape changed, showing that the Cr^{+6} ions had been adsorbed to the olive bark's surface.

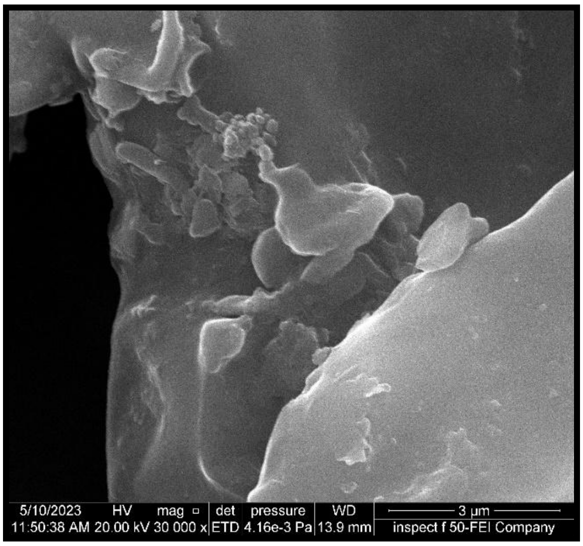

Fig 7 a: FE-SEM of olive bark before the adsorption process.

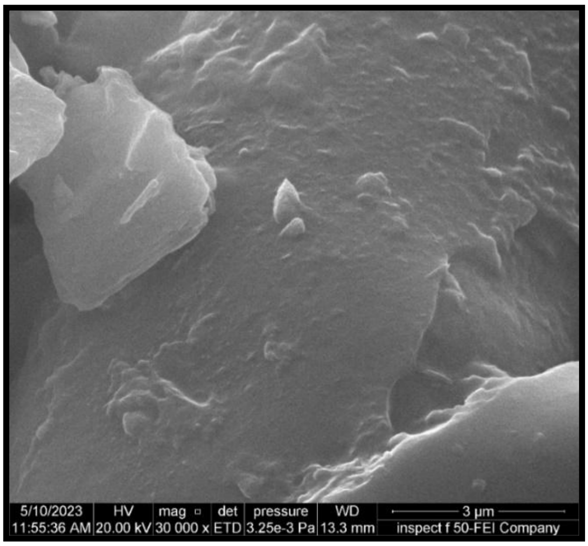

Fig 7 b: FE-SEM of olive bark after the adsorption process.

Conclusions

The study concluded that olive bark treated with 0.1 M HCl and having a small particle size of 4-6 mm showed the best ability for Cr^{+6}ionsremoval. The Box-Wilson design model was found to be suitable for explaining these adsorption experiments. Moreover, ANOVA tests revealed that various environmental parameters, such as pH and contact time, significantly influenced the removal effectiveness of olive bark. Furthermore, the FTIR study identified functional groups in olive bark, includingO-H, C=O, C=C, C-O, and C-O-C, as crucial to theadsorption process. And microscopic morphology examinations using FE-SEM analysis showed changes in the shape, texture, and structure of olive bark after the adsorption of Cr^{+6} ions. Finally, the study recommends investigating olive bark's potential to reduce pollution from substances such as oil, toxins, dyes, and pesticides.

Author Contributions Statement

All research stage: laboratory work, collection and analysis of data, research write were done by the authors

Declaration of competing interest

The authors declare that there were no competing interests.

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