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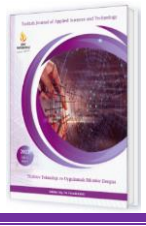

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Synthesis and Characterization of Activated Carbon and Carbon Molecular Sieves from Lentil Stalks for Methylene Blue Adsorption

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Abstract
In this study, activated carbon was synthesized using hydrochar obtained from lentil stalks, which is generally evaluated as animal feed, by hydrothermal method. Thus, a low value-added biomass source was transformed into a higher value-added product. Activated carbon was synthesized using $ZnCl_2$ activator by chemical activation method and then carbon molecular sieve was obtained by chemical vapor deposition method. Characterization of synthesized activated carbon and carbon molecular sieves was carried out using FTIR, BET and SEM analysis techniques. SEM analysis results showed that synthesized activated carbon had a more porous structure compared to hydrochar and raw lentil stalk. Synthesized activated carbon and carbon molecular sieve were used in methylene blue adsorption for the removal of organic pollutants. In this process, parameters such as pH and initial concentration affecting adsorption performance were investigated and adsorption kinetics were determined. Methylene blue adsorption equilibrium data were applied to Langmuir and Freundlich isotherms and it was determined that the system showed the best fit to Langmuir isotherm. The maximum adsorption capacity (qmax) was determined as 98 mg/g for activated carbon and 87 mg/g for carbon molecular sieve. In addition, it was concluded that the methylene blue adsorption kinetics for both adsorbents fit the pseudo-second-order kinetic model. This study reveals that agricultural wastes such as lentil stalks can be effectively used in reducing environmental pollution by converting them into high-performance adsorbent materials.

Keywords: Hydrochar, activated carbon, carbon molecular sieve, methylene blue.

1. Introduction

Dyes are organic compounds widely used in textile, paper, food, printing, plastic, beverage, leather and pharmacology industries. In general, dyes can be classified as anionic (acidic dyes) and cationic (basic dyes). Among these, azo dyes (anionic) are the class of dyes that contain nitrogen-nitrogen double bonds and are considered the largest group of organic dyes, but are also highly toxic.

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While some of them have been reported to have carcinogenic potential, the same effect has been observed in the metabolites formed from them after degradation. Therefore, there is a great need to recover them from industrial wastes before they enter water bodies. Despite their harmful effects, dyes continue to be an important and significant group of chemicals used in coloring objects. According to reports, there are over 100,000 commercially available dyes with an annual production of approximately 30,000 metric tons (1). Due to their complex aromatic structures and non-biodegradable nature, dyes in wastewater pose a significant threat to living organisms, making their detection and removal difficult. Dyes enter water reservoirs in excessive amounts from a number of industrial sources. These sources include textiles, paper, beauty products, food, and polymers (2). The continuous entry of dyes into the aquatic environment creates serious health problems for humans and also damages the ecosystem. Most of these dyes are highly toxic, mutagenic, and carcinogenic. Methylene blue (MM) is an aromatic and widely used synthetic cationic dye with high adsorption capacity, widely used in the dyeing of silk, wool, and cotton in industrial processes. MM is also used to treat diseases such as hepatitis B, psoriasis, and West Nile virüs (3). Despite its medical application, some health complications are associated with its use, such as permanent eye damage, vomiting, gastritis, breathing difficulties, nausea, mental confusion, painful urination, tissue necrosis, cyanosis, and methemoglobinemia-like syndromes. Methylene blue is also highly carcinogenic in living organisms. Methylene blue is not considered highly toxic, but ingestion produces a burning sensation. Therefore, it is extremely important to remove it from industrial wastewater before it is discharged into the environment. Different physicochemical methods such as solvent extraction, membrane filtration, coagulation and chemical oxidation have been used to remove various pollutants from wastewater. Unfortunately, these techniques are not cost-effective and also have a number of disadvantages, including a high consumption of reagents and energy, as well as the production of toxic clay or other by-products that must be disposed of after the completion of the processes. In addition, some colors used in the textile industry are extremely difficult to eliminate using traditional waste disposal methods, as they are resistant to light and oxidizing agents and do not change with aerobic digestion. As a result, new procedures for removing dyes are widely used in industries. Activated carbon is used as the most effective adsorbent (4-6).

Dyes are organic compounds that are widely used in many industries, especially in the paint, textile, paper, food, printing, plastic, beverage, leather and pharmacology sectors. Dyes are generally classified as anionic (acidic dyes) and cationic (basic dyes) depending on their chemical structure and ionic character. Azo dyes, which are among these groups, are compounds characterized by nitrogen-containing -N=N- double bonds and constitute the largest class of organic dyes. However, it is known that azo dyes have high toxicity levels and some types have carcinogenic properties (7). In addition, environmental degradation products of these compounds can have similar toxic and mutagenic effects. Therefore, it is a critical requirement to effectively remove dyes found in industrial wastewater before they reach water bodies. Although dyes have important functions in coloring objects, they cause serious environmental problems. According to research, annual commercial dye production reaches millions of

metric tons. It is known that dyes generally persist in wastewater for a long time due to their complex aromatic structures and their biodegradable nature, and they pose a significant risk to living organisms. As a result of industrial activities, high amounts of dyes enter water resources from various sectors such as textile, paper, cosmetics, food and polymer production (8, 9). This leads to permanent pollution in aquatic ecosystems and has serious negative effects on human health. In particular, a large part of the dyes exhibit toxic, mutagenic and carcinogenic properties. Methylene blue (MM) is a synthetic cationic dye with an aromatic structure, widely used in the industry, especially in the dyeing of natural fibers such as silk, wool and cotton. It has a widespread use in the textile industry due to its high adsorption capacity. Although it is not considered extremely toxic, it can cause chemical burn-like effects on mucosal tissues when taken orally. Therefore, it is of great importance to effectively remove it from industrial wastewater before it is discharged into the environment. Various physicochemical methods such as solvent extraction, membrane filtration, coagulation, and chemical oxidation are used to remove dyes and other organic pollutants from wastewater. However, the applicability of these methods is limited by factors such as high costs, reagent and energy consumption. In addition, the disposal of by-products (toxic sludge or other toxic compounds) formed as a result of these techniques creates additional difficulties. Some dyes used in the textile industry are resistant to light and oxidants and do not undergo aerobic degradation, making it difficult to effectively remove them using conventional waste disposal methods (10-12).

For this reason, innovative adsorption techniques are being developed to remove dyes from industrial wastewater, and activated carbon stands out as one of the most effective adsorbents for this purpose. In this study, in order to convert agricultural wastes into high value-added materials, hydrocarbon synthesized from lentil stalks using the hydrothermal method was converted into activated carbon using the chemical activation method, and then carbon molecular sieve was synthesized using the chemical vapor deposition method. The obtained activated carbon and carbon molecular sieve were evaluated in the adsorption of methylene blue, which is commonly found in industrial wastewater. As a result of the studies, it was determined that the synthesized activated carbon and carbon molecular sieve materials have high adsorption capacity in terms of methylene blue removal. In this direction, it is aimed to investigate the potential use of the developed materials as sustainable and environmentally friendly adsorbents in industrial wastewater treatment.

2. Experimental Part

2.1. Hydrothermal Carbonization

Hydrochar production from lentil stalks was carried out by hydrothermal carbonization (HTC) method. In this process, lentil stalks were ground and sieved and 3 g were placed in a 100 ml capacity stainless steel autoclave with 60 ml of pure water. The HTC process was completed by heating the autoclave to 200°C with a heating rate of 5°C/min and holding at this temperature for 5 hours. Then, the system was cooled to room temperature, the solid product was separated by filtration and washed with

pure water several times. The obtained lentil stalk hydrochar was dried at 105°C for 24 hours and then preserved for use in activated carbon synthesis.

2.2. Synthesis of Activated Carbon

Activated carbons were produced from the hydrochar obtained from lentil stalks by chemical activation method using ZnCI2 activator with HTC.

3 g of lentil stalk hydrochar was mixed with 3 g of ZnCl2 activator dissolved in 10 ml of pure water and kept at room temperature for 24 hours. Activated carbon was synthesized by subjecting the chemically impregnated lentil stalk hydrochar samples to a 45 min activation time at 500 oC activation temperature in the presence of N2. The synthesized activated carbon was washed with 0.5 M HCI and then washed with hot pure water until the pH value became 6-7. The obtained activated carbon was used in the production of molecular sieves and the removal of methylene blue.

The characterization of the obtained activated carbons was carried out by SEM, BET surface area and FTIR analyses.

2.3. Molecular Sieve Synthesis

Hydrochar obtained from lentil stalk by HTC method was synthesized by chemical activation method using $ZnCl_2$ activator. In the synthesis of activated carbon, 3 g of lentil stalk hydrochar was mixed with 3 g of $ZnCl_2$ dissolved in 10 ml of pure water and kept at room temperature for 24 hours. Then, the chemically impregnated hydrochar samples were subjected to activation process at 500°C in N_2 atmosphere for 45 minutes. The synthesized activated carbon was washed with 0.5 M HCl and then washed with hot pure water until the pH value reached 6-7.

The obtained activated carbon was used in the production of molecular sieves and the removal of methylene blue. Characterization studies were carried out by SEM, BET surface area and FTIR analyses.

2.4. Methylene Blue Adsorption

The adsorption of methylene blue was investigated in the batch system using the synthesized activated carbon and carbon molecular sieve. In this process, 100 ml of solution containing 50 ppm methylene blue was subjected to adsorption process using 0.1 g adsorbent at a certain pH value, temperature of 30°C. The concentration of methylene blue remaining in the solution medium was determined by UV spectroscopy at 664 nm wavelength.

3. Results and Discussion

3.1. Activated Carbon and Carbon Molecular Sieve Characterization

FTIR analysis was performed to determine the structural properties of activated carbon (Zn-AC) and carbon molecular sieve (Zn-AC-ME) samples synthesized with lentil stalk, hydrochar, $ZnCl_2$ activator. The analyses were performed in the wavenumber range of 4000-500 cm^{-1} and the obtained results are presented in Figure 1. With this analysis, it was aimed to determine the functional groups in different samples and to examine the chemical changes that occurred during the synthesis process.

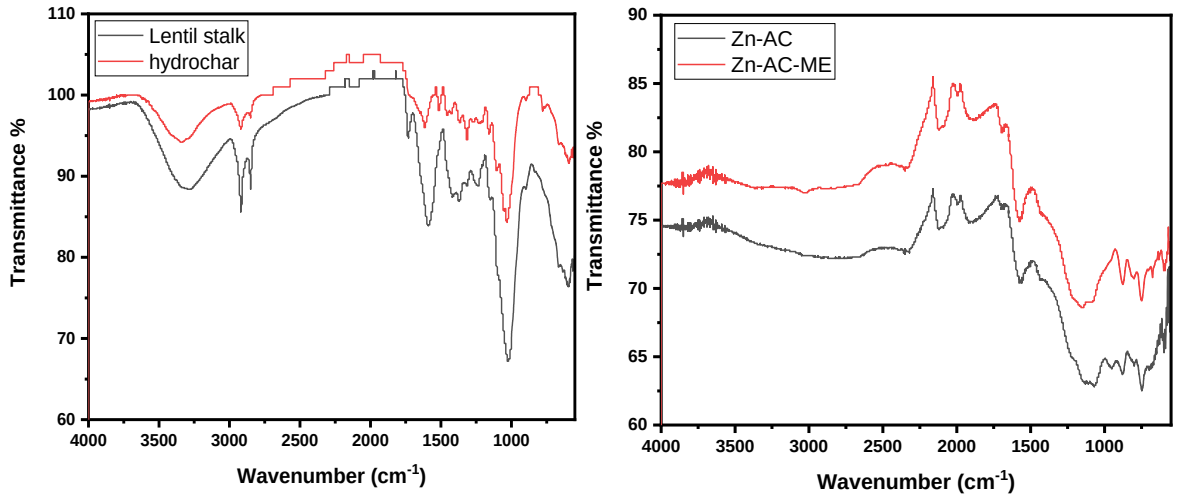
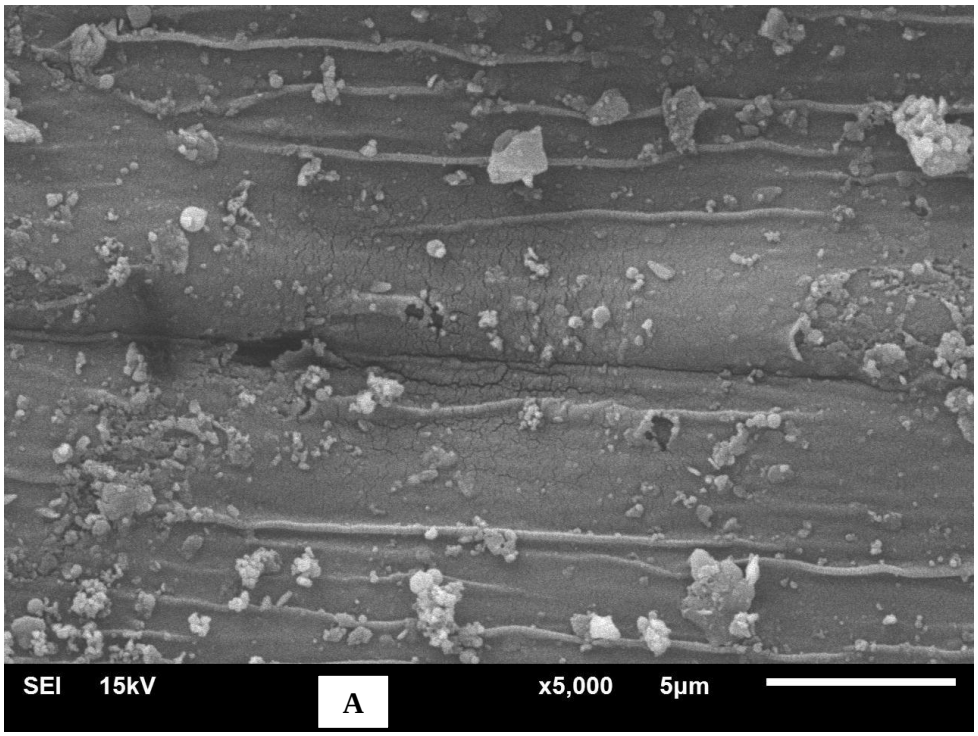


Figure 1. FTIR graphs

It is seen from Figure 1 that lentil stalk hydrochar has almost the same functional groups. It is seen from Figure 1 that there are some peaks that are not in the hydrochar structure when activated carbon is prepared. It is seen from Figure 1 that the functional groups of carbon molecular sieves are the same as those of activated carbons. The peaks observed in the FTIR spectra of lentil stalk, hydrochar, activated carbon and carbon molecular sieves can be interpreted as follows; 3500-3300 cm^{-1} peak, The peak in this region corresponds to hydroxyl (-OH) groups and is commonly found in organic components such as cellulose, hemicellulose and lignin. This band indicates water molecules or hydroxyl groups on the surface in hydrochar and activated carbons. 2930-2900 cm^{-1} peak, Peaks in this wave number range are associated with C-H stretching vibrations and generally indicate aliphatic hydrocarbon chains. It is prominent in lentil stalk and may decrease after carbonization. 1625-1610 cm^{-1} peak, The peak observed in this region represents C=O (carbonyl) and C=C (aromatic) stretching vibrations. It shows the presence of conjugated aromatic systems in hydrochar and activated carbon structures, which proves that the carbonization process has occurred. 1260-1000 cm^{-1} peak, The peaks in this region are associated with C-O stretching vibrations and indicate alcohols, esters, phenolic compounds or carbohydrate structures. These peaks tend to decrease with the carbonization process. 700-400 cm^{-1} peak, The peaks in this low wavenumber region are generally associated with metal-oxygen or C-H bending vibrations. It may indicate the formation of structures interacting with Zn-O or Zn-Cl after activation with $ZnCl_2$.

SEM analyses were performed to determine the surface morphology of lentil stalk, hydrochar, Zn-AC and Zn-AC-ME samples and the obtained images are presented in Figure 2.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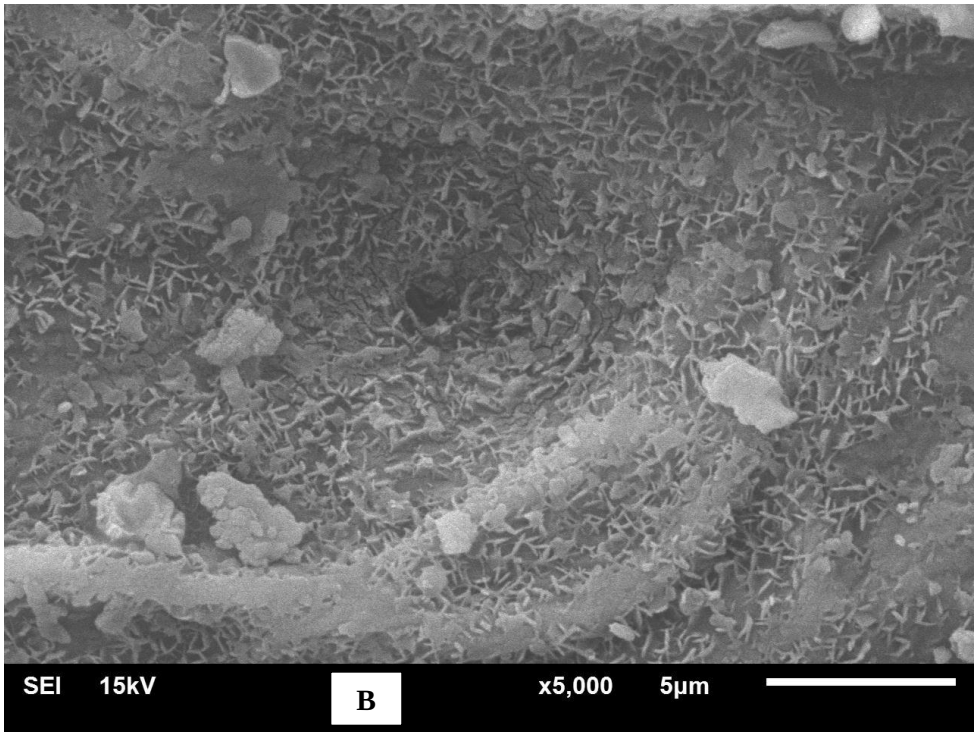
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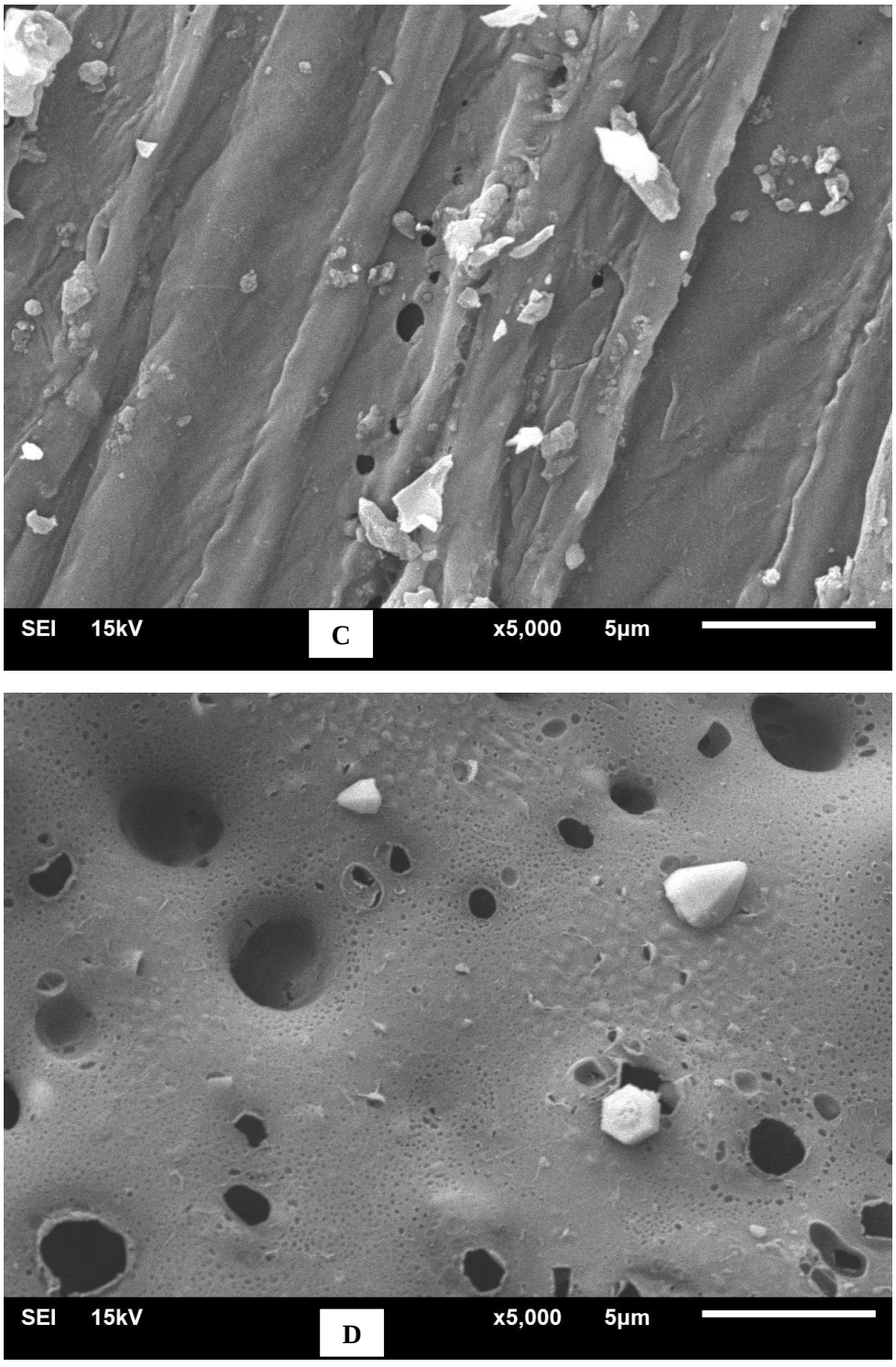

Figure 2. SEM images, A-) Lentil stalk, B-) Hydrochar, C-) Zn-AC, D-) Zn-AC-ME

When the surface morphology of the lentil stalk is examined in Figure 2A, it is seen that it does not have a distinct pore structure and its surface is relatively flat, only slightly rough. This shows that the natural structure of the lentil stalk has low porosity. When the surface of the hydrochar obtained after the hydrothermal carbonization (HTC) process is examined in Figure 2B, it is observed that it has a rougher structure and flaking on the surface compared to the lentil stalk. This morphological change

is related to the thermal decomposition and structural transformation that occur in the structure of the material during the HTC process. When the surface morphology of Zn-AC synthesized using the $ZnCl_2$ activator is examined in Figure 2C, it is determined that it exhibits a distinctly more porous structure compared to the raw material (lentil stalk) and hydrochar. It is also seen that the surface is quite rough and micro and mesopores are formed due to the effect of $ZnCl_2$ activation. When the carbon molecular sieve (Zn-AC-ME) synthesized from Zn-AC is examined in Figure 2D, it is determined that the surface has higher porosity compared to activated carbon and the pore structure is more homogeneous. In addition, it is seen that the Zn-AC-ME surface is more regular and smooth compared to the Zn-AC surface. When the pore structure of Zn-AC-ME is examined in detail in Figure 2E, it is determined that the pore sizes are quite close to each other. This is a desired feature for carbon molecular sieves, because the homogeneous pore distribution increases the selective adsorption capacity and provides more efficient performance in specific applications.

The surface areas of the Zn-AC and Zn-AC-ME samples were determined and BET (Brunauer-Emmett-Teller) analysis was performed for their characterization. This analysis was performed to evaluate the adsorption capacity by examining the specific surface areas and pore structures of the materials. The obtained BET results are presented in Table 2, and these data provide an important reference to evaluate the effectiveness of Zn-AC and Zn-AC-ME in adsorption and separation processes.

Table 1. BET surface areas

Sample	BET surface ar, m^2/g
Zn-AC	454
Zn-AC-ME	412

According to the data presented in Table 2, the BET surface area of Zn-AC was measured as 454 m^2/g, which indicates that the activated carbon has a very high surface area. This indicates that Zn-AC can exhibit effective performance in adsorption application. On the other hand, the data in Table 2 reveal that the BET surface areas of molecular sieves are lower than those of activated carbon. The probable reason for this difference is that the pore structures of molecular sieves are partially or completely blocked by benzene molecules. Benzene molecules can reduce the surface area by adsorbing into the pores, which may result in lower BET surface areas of molecular sieves.

3.2. Methylene Blue Adsorption

Zn-AC and Zn-AC-ME adsorbents were used to investigate the potential of MB, which is found in industrial wastes and is harmful to the environment, to be removed from water by adsorption. The effect of pH on the adsorption process of methylene blue was investigated and the results obtained are given in Figure 3. In experiments conducted at different pH values (acidic, neutral and basic conditions), how the adsorption capacity changed was measured and these data were presented graphically.

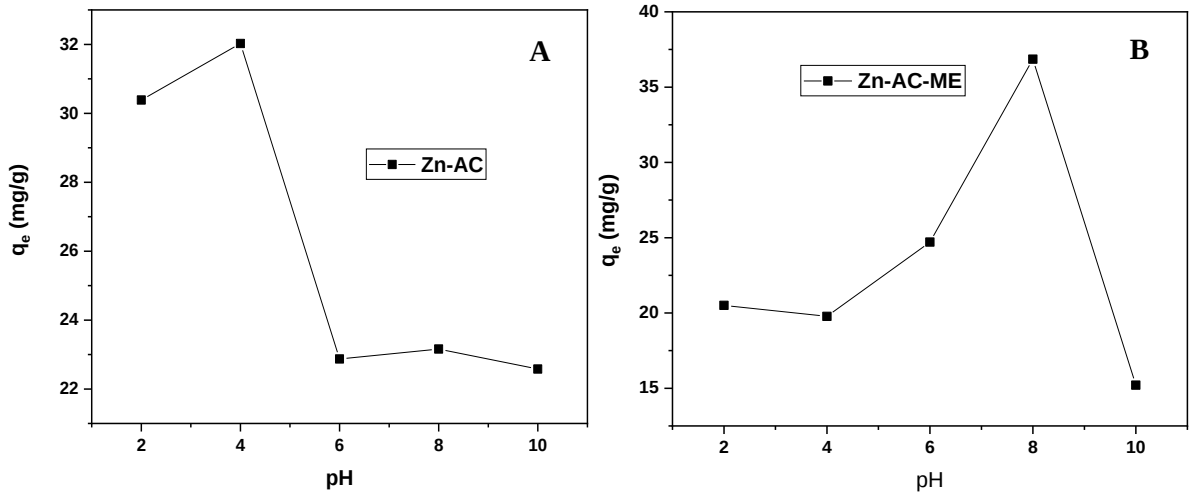

Figure 3. pH effect on methylene blue adsorption, A) Zn-AC, B) Zn-AC-ME

In Figure 3A, when the MB adsorption behavior is examined using the Zn-AC adsorbent, it is seen that the adsorption capacity increases as the pH increases from 2 to 4, but the capacity starts to decrease as the pH increases further. In Figure 3B, a similar trend is observed for the Zn-AC-ME adsorbent; the adsorption capacity increases as the pH increases from 2 to 8, but this capacity decreases at higher pH values. At low pH values (acidic conditions), the high H^+ ion concentration in the solution prevents the interaction between the methylene blue molecules and the adsorbent surface. This is due to the fact that H^+ ions bind more easily to the adsorbent surface, limiting the adsorption of methylene blue. At high pH values (basic conditions), the OH^- ions in the solution affect the structure of methylene blue, making it difficult for it to bind to the adsorbent surface. In addition, at high pHs, the charge distribution on the adsorbent surface may change, which may negatively affect the adhesion of methylene blue to the adsorbent surface. These results show that pH has a significant effect on both the adsorbent surface chemistry and the molecular form of methylene blue, and directly affects the adsorption efficiency.

The initial concentration of the solution for methylene blue adsorption was investigated, and the results obtained for Zn-AC and Zn-AC-ME adsorbents are given in Figure 4.

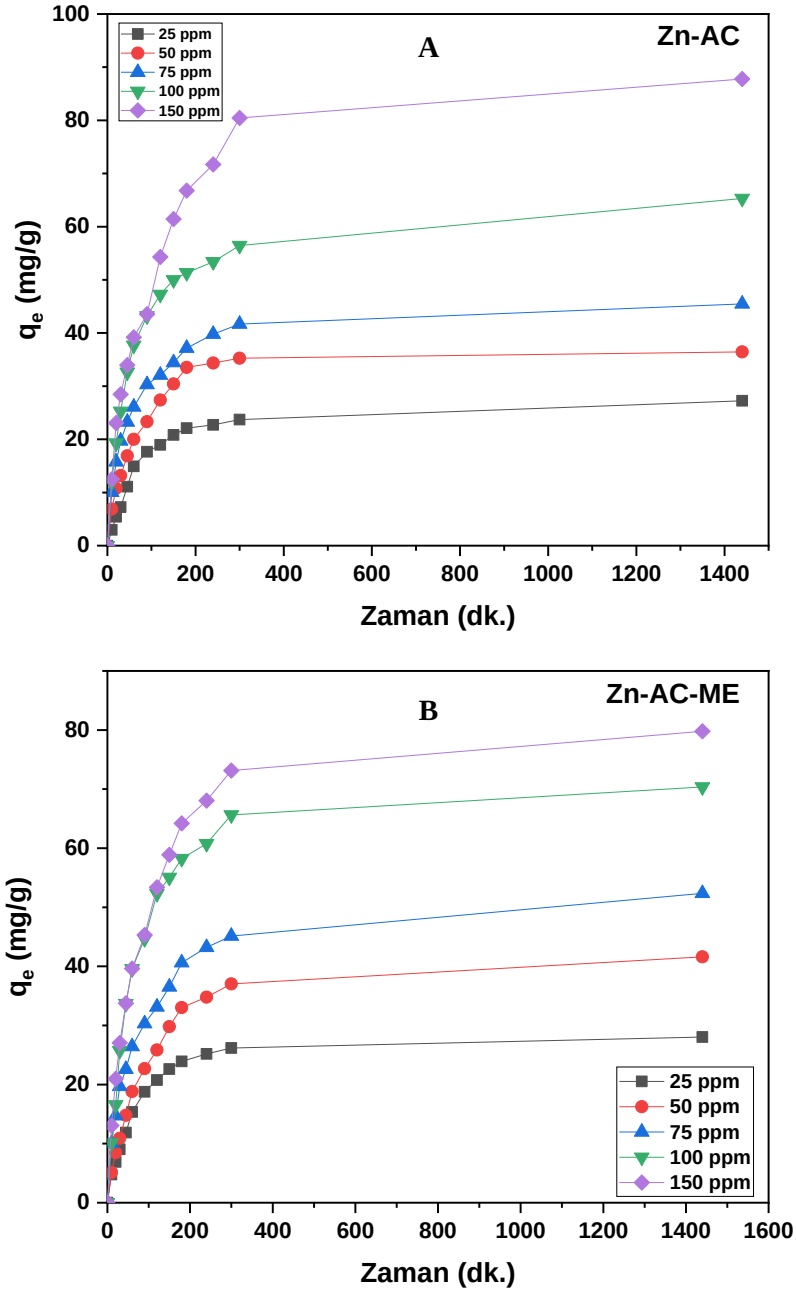

Figure 4. Effect of initial solution concentration on methylene blue adsorption, A-) Zn-AC, B-) Zn-AC-ME

In Figure 4, it is observed that the adsorption capacity increases as the initial MB concentration increases for all adsorbents (Zn-AC and Zn-AC-ME). The main reason for this increase is that the interactions between the adsorbent surface and methylene blue molecules increase with the increase in methylene blue concentration and a stronger driving force is formed for mass transfer. At high concentrations, the presence of more methylene blue molecules in the solution provides a greater possibility of contact with the active sites on the adsorbent surface, which increases the adsorption capacity. It is also observed from Figure 4 that the adsorption capacity increases faster at the beginning, but this increase slows down after a certain concentration. The initial rapid increase can be explained by the fact that the active sites on the adsorbent surface are not yet saturated with methylene blue and these

sites can be easily filled. However, with further increase in concentration, the active sites on the adsorbent surface are largely filled and methylene blue molecules compete more to reach the adsorbent surface, causing the adsorption rate to slow down. These results show that the adsorption process initially occurs with high efficiency, but the capacity becomes limited as the adsorbent surface approaches saturation point. This situation reveals that the initial concentration should be taken into consideration to optimize the performance of adsorbents.

Kinetic models are used to determine the rate of the adsorption process. In this study, data on MB adsorption were obtained using Zn-AC and Zn-AC-ME adsorbents at 30°C operating temperature and 50 ppm initial concentration. In line with these data, the kinetic behavior of the adsorption process was investigated and the rate mechanism of the process was tried to be elucidated. The kinetic constants of the examined kinetic models are presented in Table 3.

Table 2. Kinetic constants

	Pseudo-first-order kinetic model			**Pseudo-second-order kinetic model**		
	k_1 ($dk.^{-1}$)	R^2	q_e (mg/g)	k_2 ($L.mg^{-1}.dk.^{-1}$)	R^2	q_m (mg/g)
Zn-AC	0.015	0.986	32.46	0.000355	0.9937	43.30
Zn-AC-ME	0.0072	0.9903	36.55	0.000205	0.9946	49.26

According to the R^2 values in Table 3, it is seen that the MB adsorption kinetics of Zn-AC and Zn-AC-ME adsorbents fit the pseudo-second-order kinetic model. This model indicates that the adsorption rate depends on the occupancy rate of the active sites on the adsorbent surface and that the process occurs by a chemical mechanism. The high fit of the pseudo-second-order model reveals that chemical interactions such as electron sharing or exchange between methylene blue and the adsorbent surface are dominant. Such interactions are generally called chemical adsorption (chemisorption) and mean that stronger bonds are formed compared to physical adsorption. In addition, these results support that the adsorption process occurs rapidly at the beginning, but the rate slows down with the filling of the active sites on the adsorbent surface over time. This situation is also consistent with the characteristic features of the pseudo-second-order model. Therefore, it can be concluded that Zn-AC and Zn-AC-ME adsorbents work effectively in removing methylene blue by chemical adsorption mechanism.

The adsorption equilibrium data of methylene blue (MB) were applied to Langmuir and Freundlich isotherm models and the constants of these models are presented in Table 4. These analyses were used to understand the mechanism of the adsorption process and the properties of the adsorbent surface.

Table 3. Isotherm constants

	Langmuir isotherm			**Freundlich isotherm**		
	q_{max} ($mg.g^{-1}$)	K_L	R^2	K_f $(mg.g^{-1})(mg.L^{-1})^{1/n}$	n	R^2
Zn-AC	98	0.049	0.8014	28.96	5.34	0.7299
Zn-AC-ME	86.96	0.0918	0.9378	25.95	4.18	0.9037

According to the data in Table 4, MB adsorption equilibrium data for all adsorbents (Zn-AC and Zn-AC-ME) comply with the Langmuir isotherm model when the regression coefficient (R^2) values are taken into account. This result shows that the adsorption process is well described by the Langmuir

isotherm and that this model is effective in explaining the adsorption mechanism. The Langmuir isotherm model assumes that the adsorbent surface has a homogeneous structure, all active sites have the same properties and the surface energy is distributed homogeneously. This model predicts that adsorption occurs in the form of a monolayer and that there is no interaction between the adsorbate molecules. According to the Langmuir model, each active site on the adsorbent surface can bind only one adsorbate molecule and once these sites are filled, no further adsorption occurs. These findings show that the surface properties of Zn-AC and Zn-AC-ME adsorbents are compatible with the assumptions of the Langmuir model. In addition, the high R^2 values of the Langmuir model support that methylene blue adsorption occurs on a homogeneous surface and in a monolayer. These results provide important information for the design of adsorbents and optimization of adsorption capacity.

4. Conclusion

Activated carbon was synthesized using hydrochar obtained from lentil stalks by hydrothermal method. Activated carbon was produced by $ZnCl_2$ chemical activation method. Then, carbon molecular sieve (CME) was synthesized from the synthesized activated carbons by chemical vapor deposition method. The structural properties of the synthesized activated carbon and carbon molecular sieves were characterized by FTIR, BET and SEM analyses. SEM analyses revealed that activated carbon had a more porous structure compared to hydrochar and lentil stalk. In addition, it was confirmed by SEM results that carbon molecular sieves contained more pores than activated carbons. BET analyses showed that the surface area of activated carbons was higher than carbon molecular sieves. The synthesized activated carbons and carbon molecular sieves were used in methylene blue (MB) adsorption studies. The effects of parameters such as pH and initial solution concentration on the adsorption process were investigated. For all adsorbents, it was observed that the adsorption capacity increased as the initial solution concentration increased. The adsorption kinetics of methylene blue were determined and the obtained equilibrium data were applied to the Langmuir and Freundlich isotherm models. The results revealed that the adsorption process showed better fit to the Langmuir isotherm model. These findings indicated that the synthesized activated carbons and carbon molecular sieves could be effectively used in the removal of organic pollutants such as methylene blue from water.

Acknowledgements

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