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THE ADSORPTION OF ERIOCHROME BLACK T ONTO THE ACTIVATED CARBON PRODUCED FROM PEPPER STALKS

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Abstract

The aim of this paper is to present the evaluation of the adsorption of Eriochrome Black T (EBT) on the activated carbon (trH-BS) produced from pepper stalk by activating with 50% phosphoric acid solution (H_3PO_4) and then by carbonizing at 650 ^oC for 30 min. in atmosphere of nitrogen (N_2) was presented. The isotherm of trH-BS was Type IV, representing micro, meso and macroporous structures. The micropore volume and micropore surface area values of trH-BS were 0.39 cc/g and 1107.294 m^2/g, respectively. Also, its meso and macropores had a volume of about 15-17 cc/g and an average surface area of 400 m^2/g. The adsorption energy was 18.156 kJ/mol and the BET surface area was 756.257 m^2/g. Depending on Langmuir parameters, monolayer capacity was determined as 55,56 mg/g. the enthalpy change was calculated as 30.78 kJ/mol. The structure of trH-BS was clarified structurally and morphologically and the formations like nano rod were seen at Scanning Electron Microscopy (SEM) images. By using trH-BS, the adsorption of EBT from the aqueous medium was investigated using 5 different parameters. As a result, it was found that the adsorbents obtained from pepper stalks are suitable for use in EBT adsorption with a ratio of about 50% under suitable conditions.

Keywords: Pepper Stalk, phosphoric acid, activated carbon, eriochrome black T, adsorption

1. Introduction

Adsorption; since it is a method of removing ions and molecules in the liquid and gas phase from the environment by means of an active surface, it is a cheap, easy to apply and practical method used for cleaning and purifying air and water that has been polluted in one way or another with various chemicals [1, 2]. Another reason why adsorption is preferred is that waste materials can be used [3]. In this way, it is both an inexpensive method and prevents the accumulation of solid waste, which is another part of environmental pollution. Considering this, the method is a particularly important method as it helps both to obtain adsorbent using solid wastes and to clean water or air with this adsorbent. In the method, the surface area and the micropore structure of the adsorbent are especially important for the success of adsorption [4].

Activated carbon (AC) is a material having porous structure and this structure can be developed with various methods [5]. Recently, especially the dyestuff wastes of industries such as pharmaceuticals, plastics and textiles, which have experienced technological developments, have threatened water, which is essential and vital for the continuation of life [6, 7]. Especially, in the textile industry, azo-based dyestuffs used in dyeing textile products such as wool, silk and nylon, when mixed with water, cause serious and irreversible damages. Eriochrome Black T (EBT) is one of these substances. Various wastes such as waste sludge was used as an adsorbent for the removal of the EBT from the aqueous medium and it achieved high efficiency (97.08%) at pH.6 [8]. In another study, EBT was separated from the aqueous medium with magnetic graphene oxide [9]. According to the literature, using many other materials like this, EBT was tried to be removed from the aqueous medium by adsorption [10-15].

Successfully adsorption largely depends on the porosity and the surface area of the adsorbent used. In previous studies, agricultural waste is one of the preferred raw materials, especially in adsorbent use, due to its abundance, cheapness, contribution to environmental waste management and cellulosic structure. Activated carbon obtained from agricultural wastes are highly valuable adsorbents with high surface area and hierarchical porosity. In literature, it is known that many researchers have been carried out various methods to obtain ACs with high surface area and micropore volume. Also agricultural wastes such as pepper stems [16], rice-straw [17], cotton stalk [18], coconut shell [19] and pistachio shell [4, 20] have been used in production of AC to remove the organic/inorganic types.

The adsorption capacity and performance of ACs were determined by using isotherms obtained from equilibrium concentration data at constant temperature. In isotherm studies Langmuir and freundlich isotherms were frequently used in isotherm studies [21].

The Langmuir isotherm was an isotherm that assumed that dirt and dye molecules were adsorbed in a single layer on the adsorbent surface. Its equation was expressed by Eq.1 [22].

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

Here, C_e was the equilibrium concentration in solution in mg/L and q_e represented adsorbed quantity in the equilibrium. In addition, q_m gave the monolayer adsorption capacity of the adsorbent in mg/g, while K_L represented the adsorption constant in L/mg and was obtained by plotting C_e/q_e against C_e.

Another characteristic parameter used in the Langmuir isotherm is the R_L separation factor [23].

$$R_L = \frac{1}{1+K_L C_o} \qquad (2)$$

here, Co represented initial concentration. This factor gives information about whether the adsorption is appropriate or not or linear or irreversible. Accordingly, if the R_L value is between 0 and 1, it is understood that the adsorption process is favorable, if it is greater than 1 it is unfavorable and if it is equal to 1 it is linear process. it is understood that the adsorption process is irreversible if it is equal to 0.

Freundlich approach, on the other hand, represented adsorption where adsorption took place on a heterogeneous surface and was expressed by Eq. 3 [24].

$$lnq_e = \frac{1}{n} lnC_e + lnK_F \qquad (3)$$

Here, n denoted the degree of adsorption and K_F denoted the adsorption constant. It was obtained from the slope and intercept of the ln C_e versus ln q_e graph.

In our previous study, methylene blue adsorption was carried out successfully with adsorbents prepared with pepper stalks under different conditions. The adsorption capacity was determined as 178,4121 mg/g [25].

Based on the information obtained, the examination and evaluation of the adsorption of EBT was aimed in the concept of this study, as it was wondered what kind of performance would be obtained in adsorption of EBT with activated carbon to be obtained by using pepper stalks. For this reason, ACs were produced from pepper stalks that were treated with two stages, which were activated in activating solutions and carbonized 650 ^oC 30 min. in atmosphere of nitrogen (N_2).

2. Material and method

2.1. Solution preparation and AC production

All chemicals used in the study were purchased from Merck. All solutions were prepared with distilled water. The process of obtaining AC consisted of two stage: activation and carbonization. Primarily, collected pepper stalks were washed with distilled water and dried in stove at 80 ^oC and for 24 h. Then, the dried stalks were kept in small pieces in 50% H_3PO_4 solution for 24 h. The filtered stalks pieces were boiled several times and washed with distilled water. The dried stalks were kept in an atmosphere of N_2+H_2O(g) for 30 min. in a high temperature furnace at 650 ^oC. The ACs obtained at the end of carbonization were named trH-BS.

2.2. Used apparatuses

SEM and EDX data of AC were taken by using ZEISS brand EVO 50 Model device and structural characterization was obtained with 4 cm-1 resolution with the spectrum taken with SHIMADZU IRTracer-100 FTIR device. Active surface analysis was determined with the NOVA 4000e-Quamtachrome surface and pore size analyzer. Spectrophotometric data were taken by using UV-Vis spectrophotometer (HITACHI U-0080D).

2.3. Analysis procedures

In adsorption experiments, 1000 ppm stock solution of EBT was used to prepare working solutions at the desired concentration. The working solutions were 100 mL. Adsorption was carried out in suitable flasks using a shaker with temperature and shaking speed control. At certain times, the measurements of the samples taken to determine the EBT concentration were made in the presence of pH.10 buffer in UV-Visible at a wavelength of 612-616 nm.

3. Experimental results

Adsorption isotherm obtained under cryogenic conditions were used to determine the surface area and pore structure of activated carbons obtained from pepper stalk. The BET isotherm and BJH pore size-pore surface area distribution graph versus radius width were given in Figure 1. The information about the micropore volume and surface area of trH-BS was determined from DR method, the meso and macro pore volume and surface area information was determined by using the BJH method. the adsorption energy was also determined by the DR method.

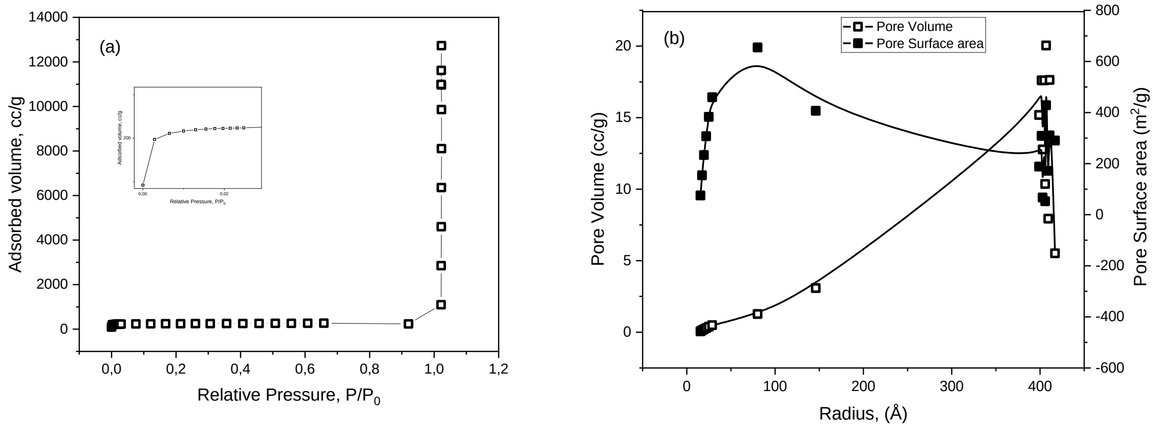

Figure 1. a) The adsorption isotherm and b) The change of Pore volume and Pore surface area versus Radius of trH-BS

The pore size distribution and the isotherm were given by using the BJH and BET methods, respectively. The isotherm was type IV according to IUPAC classification. Such isotherms were observed in the pores at high relative pressure values after monolayer formation. This formation was clearly observed in Fig. 1a. Fig. 1b showed that the meso and macropores were more in volume and surface area than the micropores. As result, the micropore volume and micropore surface area values of trH-BS were 0.39 cc/g and 1107.294 m^2/g, respectively. On the other hand, its meso and macropores had a volume of about 15-17 cc/g and an average surface area of 400 m^2/g. The energy of adsorption was determined as 18.156 kJ/mol and the surface area of BET was determined as 756.257 m^2/g. The low surface area of BET can be explained by the smaller volume of micropores compared to meso and macropores.

In this study, the adsorption of EBT on the obtained activated carbons (trH-BS) was investigated by considering parameters such as temperature, the solution pH, the amount of adsorbent (dosage), the particle size and the concentration of EBT. The results of these studies were presented in the next section.

3.1. Temperature effect

In order to examine this effect, the temperature of working solution was changed for each experiment. Other parameters were kept constant while the temperature was changed. So, the amount of adsorbent was determined as 1 g, the concentration as 100 ppm, pH as 7, and particle size as 400-600 micron. Chosen temperatures were 30 °C, 40 °C, 50 °C and 60 °C. In figure 2, the changing in the percentage of adsorption of EBT with time for different temperatures was compared.

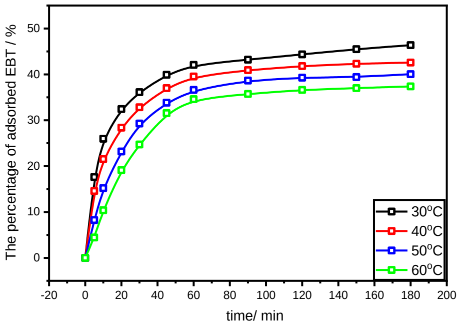

Figure 2. The changing of EBT adsorption with temperature

According to the obtained data, it was observed that the adsorption decreased as the temperature increased. At the end of 180 minutes, the adsorption amount was determined as 42.72 % for 40 °C, 39.58% for 50 °C, and 37.22 for 60 °C; this value was 46.26 for 30 °C. It was observed from the graph that the adsorption reached equilibrium in 40 minutes for all temperature conditions. When equilibrium was reached, the adsorption reached 39.97% for 30 °C. This result was expected since such studies in the literature exhibit physical adsorption.

3.2. pH effect

In the effect of pH, which is another parameter, the pH of the solution was changed by keeping all parameters constant. Since pH is dominant parameter, 6 different value (2, 4, 6, 7, 8 and 10) were used as acidic, basic and neutral. The data obtained were plotted against time in Figure 3.

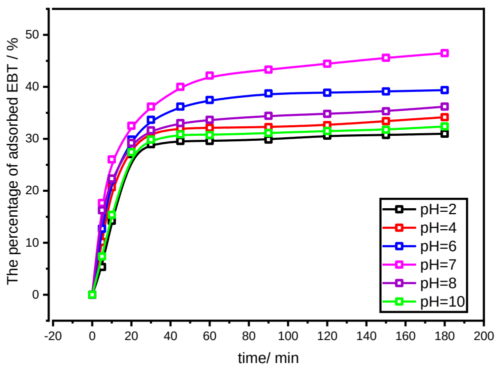

Figure 3. The changing of EBT adsorption with pH

When the figure was examined, it was observed that the adsorption in acidic and basic media were relatively low and the best adsorption was obtained in neutral media. This result was an expected result and the reason for the low adsorption in acidic and basic solutions may be that the interaction of EBT and trH-BS was negative due to their structures. Equilibrium adsorption value was reached in 20 minutes. It can be easily observed from the figure that after the equilibrium was reached in acidic and basic medium, the amount of adsorption remained lower that the adsorption in neutral medium. While this amount of changing was 2.54 for pH 2, it was 3.63 for pH 10.

3.3. Concentration effect

While examining the concentration effect, EBT solutions were prepared at 5 different (50 ppm, 100 ppm, 200 ppm, 300 ppm and 400 ppm) concentration values and all other parameters were kept constant. The data obtained are given in Figure 4.

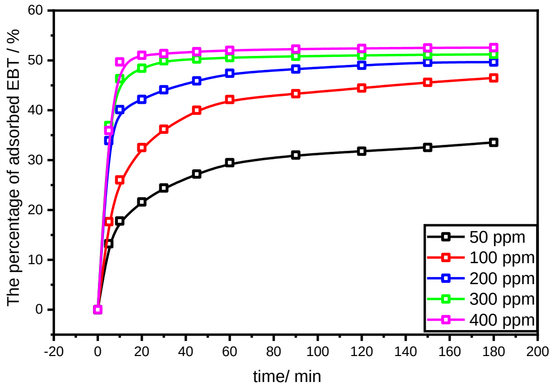

Figure 4. The changing of the EBT adsorption with concentration

It was expected that adsorption will increase with increasing concentration in adsorption. The expected result was also obtained in this study. At the end of 180 minutes, the obtained adsorption amounts were 33.31 %, 46.5 %, 49.46 %, 50.75 % and 51.9 % for 50 ppm, 100 ppm, 200 ppm, 300 ppm and 400 ppm, respectively. As can be seen, an increasing adsorption was obtained with increasing concentration. This effect was due to the large number of molecules present in the medium as the concentration increases because of that the excess surface area to be adsorbed was provided at this ratio.

3.4. Dosage effect

A positive effect on the adsorption was expected with an increase in the amount of adsorbent used. In order to see this effect, 5 different adsorbent amounts (0.05 g; 0.1g; 0.2g; 0.4g and 0.5g) were used in the experiments of this section and other parameters were kept constant. Figure 5 was showed the effect of the amount of adsorbent used in different amounts on EBT adsorption depending on time.

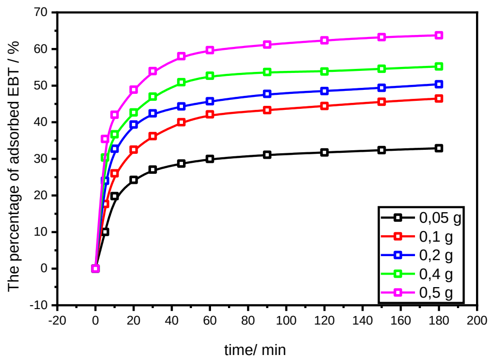

Figure 5. The changing of the EBT adsorption with dosage

According to the obtained data, the EBT adsorption increased with increasing adsorbent dosage. It took approximately 40 minutes for the adsorption to reach equilibrium. At the end of 180 minutes, the adsorption was reached 63.57% for 0.5 g, while it was 32.69 % for 0.05 g.

3.5. The particle size effect

The particle size of the adsorbent used was another factor that had an effect on the adsorption. Since it was thought that the surface area was increase, it was expected that the adsorption would increase as the particle size decreased. The adsorbents called trH-BS were divided into 5 different parts according to the sieve gap used and the desired size adsorbents were used in adsorption experiments. While ACs with particle size of 400-600 microns were used in the other factor experiments, in this section, 5 different particles were used: powder under the sieve (<400 microns), 400-600 microns, 600-1000 microns, 1-2 mm and large particles (2mm<). The obtained data are presented in Figure 6.

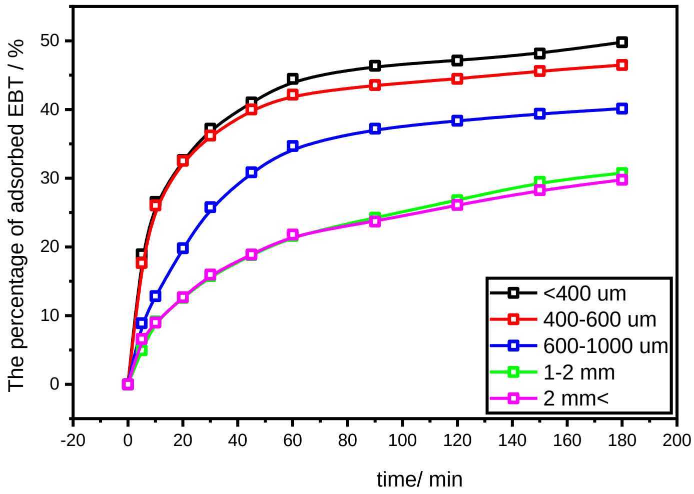

Figure 6. The changing of the EBT adsorption with particle size

As can be seen from the graph, adsorption with coarse particles reaches a value of 30% at the end of 180 minutes, while the adsorption value made with smaller adsorbents reached 50%. In addition, while the adsorption reached equilibrium in approximately 50-60 minutes in adsorbents with small particles, it was not observed that the adsorption reached equilibrium in this time period in adsorbents with large particles. This result was considered useful as a result that can be used in application in the next stages.

3.6. Isotherm and thermodynamic studies

The relation of adsorption phenomenon with the surface was tried to be explained by the Langmuir and Freundlich isotherms. The graphs drawn using the data obtained in the experiments with different concentrations are given in Fig. 8 and b.

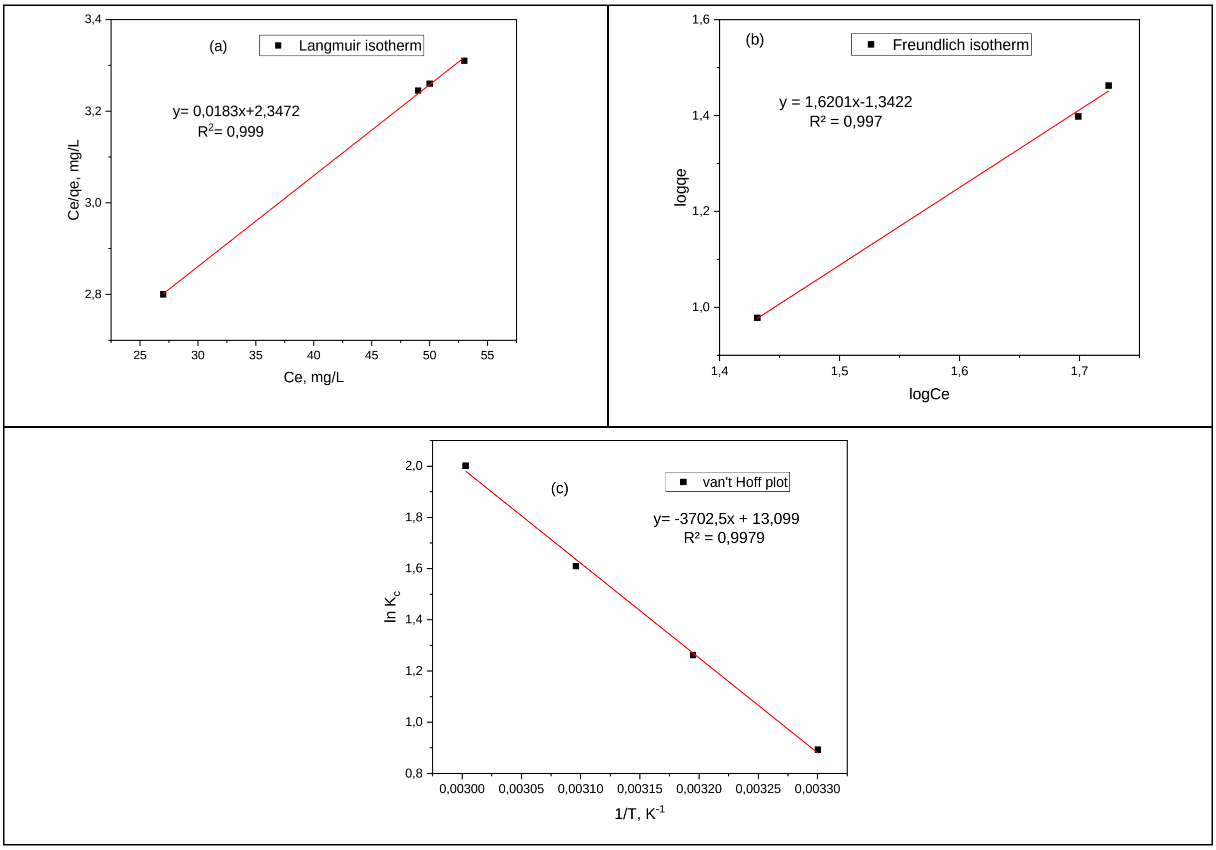

Figure 7. The graphs of a) Langmuir isotherm, b) Freundlich isotherm, and c) van't Hoff

According to the graph lines drawn from the obtained data, the Langmuir isotherm graph with the highest R^2 value of 0,999 was observed, so the EBT adsorption using trH-BS adsorbent complied with the Langmuir isotherm. This suitability led to the conclusion that the homogeneity of the surface and the adsorption event took place in a single layer. Monolayer capacity was determined as 55,56 mg/g. In addition, for this study, the R_L separation factor, which is one of the characteristics of the Langmuir isotherm and expressed by the Eq. 2, was determined as 0,89. In the literature, if the R_L value is between 0 and 1, based on the suitability of the adsorption event, the EBT adsorption event was thought to be appropriate under these conditions.

Since the R^2 value of the straight line obtained in the Freundlich isotherm was found to be 0,998, it was thought that this adsorption event mostly suited Langmuir, so the adsorbent surface had a homogeneous structure. On the other hand, adsorption degree was determined as 0,61 and adsorption constant K_F as 21,98, depending on the Freundlich isotherm equation.

The thermodynamic data of the adsorption event were obtained by using the data obtained from different temperature experiments. These data were plotted in accordance with the van't Hoff equation and shown in Figure 8c. the enthalpy change (ΔH), entropy change (ΔS) and Gibbs free energy change (ΔG) were determined from the line equation with R^2 value of 0,9979. The obtained all isotherm and thermodynamic data were shown in Table 1 in comparison with the data obtained in other studies.

Table 1. The obtained ısotherm and thermodynamic data

		Ref. 3	Ref.9	Ref. 26	Ref. 27	This study
Langmuir	$q_{L, (mg/g)}$	14,05	210,53	14.025	85.55419	55, 56
	$K_{L, (L/mg)}$	0,04	0,538	0.0399	0.700694	0,0019
	R_L	-	0,28	0.2636–0.7143		0,89
	R^2	0,9997	0,998	0.9904		0,999
Freundlich	n	6,21	3,60	1.4892	4.045079	0,61
	K_F	92,36	76,66	0.8052	35.16842	21,98
	R^2	0,9833	0,9339	0.9996		0,997
Thermodynamic data						
	ΔH, kJ/mol	-	-28,47	16.8467	-	-30,783
	ΔS, J/molK	-	-88,46	11.0808	-	108,90509
	ΔG, kJ/mol	-	-2,11	13.5999	-	-63,236

As can be seen from the table, the results obtained were close to the results obtained in literature. It was determined by the enthalpy value that the adsorption was exothermic. The fact that the Gibbs energy value was negative "-" also showed that the adsorption event was voluntary and spontaneously.

3.7. Structural and morphological characterization

Since most agricultural wastes are of organic origin, they contain functional groups consisting of O, H and C such as esters, ethers, alcohol and carbonyl. The IR plot of trH-BS was given in Figure 8. The peaks inn this graph showed the functional groups in the structure of trH-BS. Since it is processed with acid, it is an expected result that it contains the peak of OH bonds in its structure. In addition, peaks belonging to various organic structures such as alkenes and alkynes were observed. These peaks were listed below both on the graph and in the groups they belonged to:

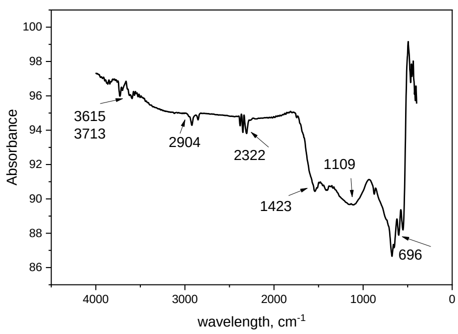

Figure 8. The FT-IR graph of trH-BS and related assignments

3615 $cm^{-1}$and 3713 cm^{-1}: The hydrogen bond vibration in alcohols and phenols or acid residue in adsorbent structure, 2904 cm^{-1} and 2322 cm^{-1}: The C-H and C=C stretching vibration in alkane, alkene or alkyne organic groups, 1423 cm^{-1}: The C=C vibration in the ring of aromatic structure. The board peak at 1109 cm^{-1}: The C-O bond stretching vibrations in functional groups such as ether and ester.

The sharp and multi vibrations at 696-400 cm^{-1} was thought to belong to inorganic formations.

Figure 9 showed the morphological pore structure of trH-BS in three different sizes: 30 micron, 10 micron and 1 micron distances.

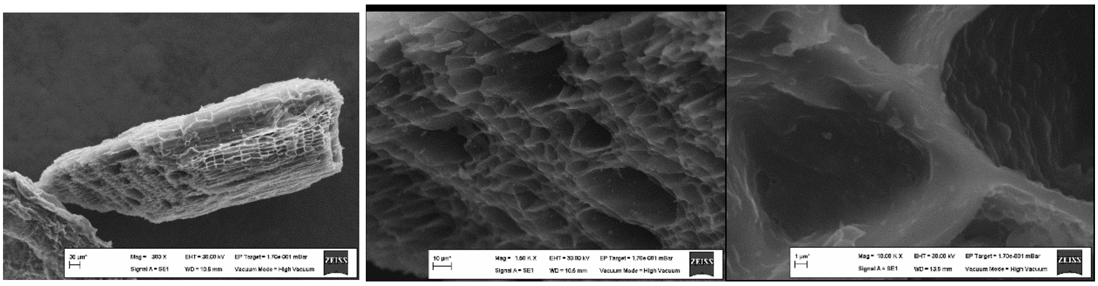

Figure 9. SEM images

Here, it was clearly observed that there were pore formations such as nano rods formed after carbonization in the structure of trH-BS. It was thought that the surface mounds in the pores were also formations that helped to form the active surface for adsorption.

4. Conclusions

Within the scope of this study, trH-BS adsorbent was produced from peppers stalks activated using H_3PO_4 solution and carbonized in a N_2 atmosphere at 650 ºC for 30 minutes. The isotherm of trH-BS was Type IV, representing micro, meso and macroporous structures. The micropore volume and micropore surface area values of trH-BS were 0.39 cc/g and 1107.294 m^2/g, respectively. Also, its meso and macropores had a volume of about 15-17 cc/g and an average surface area of 400 m^2/g. The adsorption energy is 18.156 kJ/mol and the BET surface area was 756.257 m^2/g. By using trH-BS, the adsorption of EBT from the aqueous medium was investigated using 5 different parameters. At the end of the study, it was found that the adsorbents obtained from pepper stalks are suitable for use in EBT adsorption with a ratio of about 50% under suitable conditions. Depending on Langmuir parameters, monolayer capacity was determined as 55,56 mg/g. the EBT adsorption on trH-BS adsorbent was found that it was favorable under these conditions. 30.78 kJ/mol entalphy change was calculated during the adsorption. The structure of trH-BS was clarified structurally and morphologically. So, the functional groups and pore formations in its structure were determined. The pores like nano rod were seen at the SEM images. As result, ıt was concluded that the pores in the structure of trH-BS could be developed with different methods and thus the sustainability of these studies could be realized.

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