

# Synthesis and characterization activated carbon using a mix (asphalt- sulfur Waste) supported nickel Catalyst for hydrogenation process

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## Abstract

In this work, the preparation of activated carbon using low-cost materials ( asphalt : sulfur waste) through thermal fusion carbonization and the activation by microwave technique. Carbonization was carried out at temperatures ranging from  $240 \pm 5^\circ\text{C}$ , followed by hot steam and microwave activation to improve porosity and surface structure. The synthesized of activated carbon exhibit the presence of pores on the surface with various diameter ranges (from 2.2 to 2.3  $\mu\text{m}$ ). an active surface area of  $203.42\text{m}^2\text{g}^{-1}$ , pore volume 0.03-0.08  $\text{cm}^3/\text{g}$  and shows a high adsorption capacity for dye. Evaluations included the adsorption of methylene blue and iodine dyes as key indicators of surface activity and pore size. Sample C6 demonstrated excellent adsorption performance, surpassing, in some cases, commercial carbon.

**Keywords** :Activated carbon, asphalt, sulfur waste, thermal fusion carbonization.

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## 1-INTRODUCTION

Activated carbon is a highly porous material, and its porosity primarily arises from structural defects that occur during the production process. These imperfections lead to the formation of pores with limited or negligible energy content, which, in turn, enhances the material's adsorption capabilities [1]. The irregular structure of these pores contributes to the exceptionally high surface area of activated carbon, which typically ranges from 300 to 2000  $\text{m}^2/\text{g}$  and can reach up to 5000  $\text{m}^2/\text{g}$  in certain instances [2]. The efficiency of activated carbon can be categorized into three primary mechanisms: adsorption, mechanical filtration, and surface interactions. Regarding adsorption, two principal types are recognized: physical adsorption and chemical adsorption. Physical adsorption occurs due to van der Waals forces between the adsorbent and the adsorbate, and it is influenced by several factors, including the physical and chemical properties of both phases, adsorbate concentration, and solution conditions such as temperature and pH. Notably, it is dependent on the interaction distance between the adsorbate and the surface of the adsorbent [3]. Mechanical filtration, in contrast, involves the removal of suspended solids by passing the solution through an activated carbon column. This process is influenced by the particle size of the carbon, its apparent bulk density, and its mechanical strength (i.e., hardness) [4-8]. Surface interactions significantly influence adsorption efficiency, especially due to the presence of oxygen-containing surface groups and other oxidizable species on activated carbon [9]. A review of the literature reveals a diverse range of raw materials and synthesis methods for producing activated carbon. Commercially, it has been produced from sources such as sawdust, peat, and petrochemical coal [10]. Ramadan et al. reported the synthesis of activated carbon from bituminous waste derived from crude oil refineries, utilizing an air stream at  $350^\circ\text{C}$  for 3 hours, followed by oxidative treatment using with pentoxide ( $\text{V}_2\text{O}_5$ ) and chemical activation with using hydroxide [11]. Similarly, Hamdoun and his team prepared activated carbon from bituminous materials sourced from northern Iraqi refineries [12]. In another study, Hamdoun utilized Baiji asphalt as a precursor, applying air oxidation under direct current (DC) conditions and employing orthophosphoric acid as an activating agent at concentrations ranging from 2% to 5% by weight [13]. Yamaguchi developed a method to synthesize activated carbon from asphalt using sodium hydroxide at  $550^\circ\text{C}$ , yielding resulting in material with exceptionally high adsorptive properties [14].

## 2- MATERIALS AND METHODS

In this study, Qayyarah asphalt containing sulfurous residues (Table 1) was selected as the primary precursor for the synthesis of activated carbon. The initial carbonization process was conducted in a nickel-coated stainless steel reactor. The reactor was gradually heated to a temperature of  $240 \pm 5$  °C, and this temperature which 5 to 6 hours to allow for complete ensure the removal of sulfurous waste materials (foam). [15]

Following carbonization, the resulting char was mechanically ground and subjected to steam activation at 900 °C for 3 hours, promoting the development of porous structures [16]. The activated product was then purified through thermal sublimation, involving acid with 10 mL of 10% hydrochloric acid to eliminate residual metal contaminants [17]. Subsequently, the purified carbon was oven-dried at 110 °C, and °C treated using microwave irradiation at 720 watts to enhance surface and stability [18].

### 3. Evaluation of Activated Carbon Performance

#### 3.1 Determination of Internal Surface Area via Iodine Adsorption

The internal surface area of activated carbon can be accurately quantified using the iodine adsorption method, a widely recognised standard technique in adsorption studies. This method estimates the adsorption capacity by measuring the amount of iodine adsorbed from an aqueous solution by a specific mass of activated carbon. The iodine number (I.N.) indicates the milligrams of iodine adsorbed per gram of activated carbon and is calculated using the following formula:

$$\text{I.N.} = \frac{X}{M} \times D$$

Where X represents the mass of iodine adsorbed (mg), M denotes the mass of the activated carbon sample (g), and D indicates the iodine solution concentration factor. This metric offers a reliable indication of the microporous structure and internal surface area of the adsorbent material [19-20].

#### 3.2 Determination of External Surface Area via Methylene Blue Adsorption

The external surface area of activated carbon is typically assessed by measuring the adsorption of methylene blue dye from an aqueous solution. In this procedure, a precise quantity (e.g., 0.1 g) of activated carbon is added to a known volume (100 mL) of methylene blue solution at a concentration of 20 ppm. The mixture is then agitated under controlled conditions—usually for 24 hours at ambient temperature—to reach equilibrium. After decolourization, an aliquot (50 mL) is separated, and its absorbance is measured at 665 nm using UV-Vis spectrophotometry. The concentration of methylene blue adsorbed is determined by comparing the absorbance to a pre-established calibration curve with known dye concentrations (5-10-15-20-25 ppm). This technique provides an estimate of the mesoporous and external surface characteristics of the activated carbon [21].

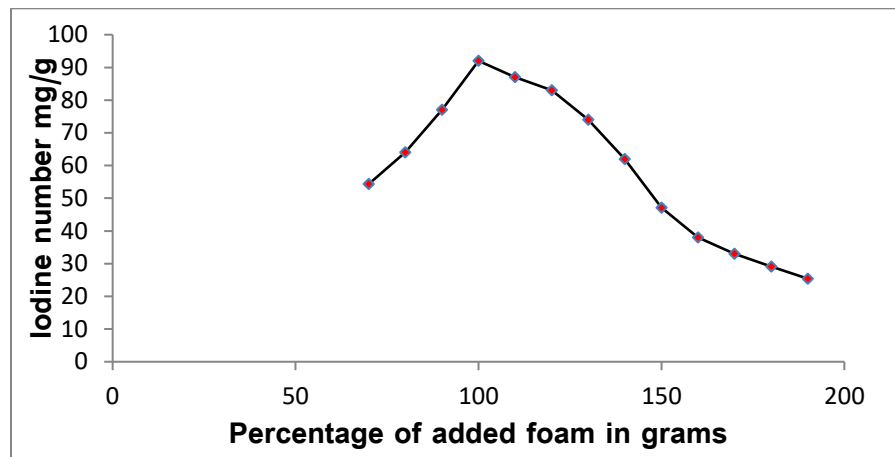
Complementary to these adsorption measurements, the activated carbon samples are further characterised using analytical methods, including optical microscopy for morphological analysis, elemental analysis for compositional data, and X-ray diffraction (XRD) to elucidate the crystalline structure and degree of graphitisation In addition to Scanning electron microscope(SEM).

## 4 RESULTS AND DISCUSSION

### 1- Iodine number

Iodine number: The iodine number is a crucial test that determines the adsorption efficiency of prepared carbon for small molecules. It can be considered a function of the pore volume present in the carbon structure. (Table 1) illustrates that the iodine number gradually increases with the quantity of foam up to a certain point, indicating an enhancement in microporosity within the structure of the prepared activated carbon. The iodine number peaked at models C6 = 965 and C5 = 843.64, signifying a strong reaction that leads to saturation and stability in the chemical composition. Subsequently, the iodine number began to decline with an increase in foam percentage, ultimately reaching 572 mg/g in the final model. This decline suggests that larger quantities of foam result in the formation of larger or irregular pores, along with an increase in the inorganic materials and ash originally present in the foam, which diminishes the carbon's effectiveness in adsorption. The iodine number is closely related to the material's porosity and reached its

highest levels when the foam quantity was within the optimal range. Therefore, controlling the amount of foam is a key factor in enhancing the quality of activated carbon prepared for adsorption applications.



**Figure (1): Relationship between the percentage of foam added in grams and the iodine number (mg/gm)**

#### 2-Methylene blue dye

Methylene blue dye was utilized to assess the adsorption capacity of the prepared activated carbon samples. As this dye represents large organic molecules, it serves as an effective indicator of surface porosity and adsorbent efficacy. The results revealed significant variations in adsorption values among the samples, ranging from 21.97 mg/g to 97.65 mg/g, reflecting differences in their structural and surface properties. The samples exhibiting the highest adsorption capacities (C5, C6, C7, C8, C9, C10) demonstrate the success of the activation process, as illustrated in Figure 2. Table 1 presents the adsorption values of methylene blue dye for the prepared samples. The steam activation process enhanced the total surface area and increased the number of active binding sites. Conversely, samples with lower adsorption values may indicate inadequate steam activation, insufficient agitation within the tubular furnace during the activation process, or a lack of pores suitable for the pigment particle size. Additional factors may include the clogging of certain pores during preparation or the presence of residual ash that hinders adsorption. This aligns with the findings of reference [22], which showed that ineffective treatments diminish the efficiency of the active surface, resulting in reduced adsorption capacity. High adsorption values (exceeding 90 mg/g) signify the success of the activation process and the effectiveness of the micropores in capturing dye molecules. These results are consistent with reference [23], which indicated that activated carbon derived from carbon-rich sources can exhibit a high adsorption capacity for methylene blue dye, potentially exceeding 90 mg/g under optimal thermal activation conditions. In comparison, commercial activated carbon has demonstrated an adsorption capacity ranging from 34.8 to 90.0 mg/g in studies such as reference [24]. Notably, some of the locally prepared samples here rival the performance of commercial products, indicating the effectiveness of the preparation methods employed.

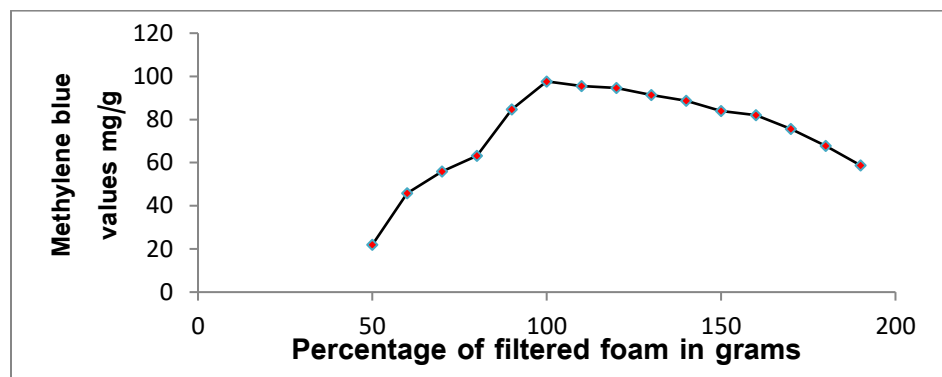


Figure 2: Relationship Between Methylene Blue Values and the Percentage of Foam Added per Gram

Table 1: Determining the Specifications and Effectiveness of Activated Carbon Prepared from Qayyarah Asphalt Treated with Foam through Steam Activation.

Models	Amount of asphalt in grams	Amount of foam added in grams	Iodine number mg/gm	methylene blue dye mg/gm
C1	100	50	230	21.97
C2	100	60	442	45.81
C3	100	70	534	55.93
C4	100	80	617	63.16
C5	100	90	843.64	84.62
C6	100	100	965	97.65
C7	100	110	942	95.56
C8	100	120	934	94.56
C9	100	130	912	91.41
C10	100	140	874	88.61
C11	100	150	841	83.95
C12	100	160	827	81.99
C13	100	170	742	75.56
C14	100	180	664	67.81
C15	100	190	572	58.71
Russ	----	----	61.5	34.8
B.D.H	////	///	908	90.0

Russ: Russian commercial granular activated carbon.

B.D.H:

British commercial granular activated carbon.

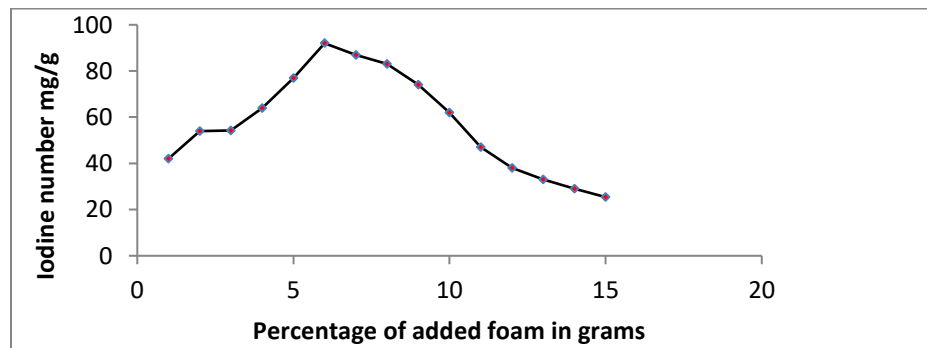
#### 4. Activation process by microwave radiation:-

The samples were reactivated through thermal activation using 720 W of microwave radiation for 10 minutes. In Table 3-6, we observe a gradual increase in the iodine value, starting from 723 mg/g with a foam content of 50 grams and reaching a peak of 1042 mg/g at 100 grams of foam. This trend indicates that increasing the foam content up to this activation stage enhances the structure of the activated carbon by promoting the formation of micropores. This improvement results from the structural interaction between the foam and asphalt, in addition to the microwave heating, which facilitates regular and controlled thermal cracking.

However, beyond this optimal point, the iodine values begin to decline, dropping to 813 mg/g with the addition of 190 grams of foam. This decrease is attributed to the structural saturation of the matrix; specifically, an excess amount of foam can lead to pore clogging or deterioration of the porous structure due to structural collapse caused by excessive expansion. Researchers [25-26].have indicated that there is an optimal limit for adding activators, such as gases or organic components, and exceeding this limit results in decreased effectiveness of the final material due to poor pore distribution. Furthermore, microwave activation provides uniform internal heating that quickly penetrates the molecules, leading to more efficient activation compared to conventional heating, as explained in study [27]. The results indicate that the optimal ratio for adding foam is 100 grams per 100 grams of asphalt, as this ratio achieves the highest iodine number, which correlates with the largest effective surface area. Exceeding this level results in performance deterioration; therefore, the proportions of components must be carefully adjusted during the manufacturing of activated carbon to ensure maximum porous effectiveness, as illustrated in Table 2 and Figure 3.

**Table 2: Specifications of Activated Carbon Prepared from Qayyarah Asphalt Treated with Foam via Microwave Activation**

Models	Amount of asphalt in grams	Percentage of filtered foam in grams	Iodine number after microwave activation
C1	100	50	723
C2	100	60	882
C3	110	70	952
C4	100	80	965
C5	100	90	1031
C6	100	100	1042
C7	100	110	1036
C8	100	120	1001
C9	100	130	992
C10	100	140	984
C11	100	150	935
C12	100	160	900
C13	100	170	921
C 14	100	180	823
C15	100	190	813



**Figure 3 .Relationship Between the Percentage of Foam Added (in Grams) and the Iodine Number (mg/gm) After Microwave Activation 5-Atomic Absorption Spectrometer (AAS)**

To load nickel onto activated carbon, an aqueous sulfate solution was prepared by dissolving sulfate in distilled water. The activated carbon was then immersed in this solution for a specified duration, with occasional stirring. A solution containing 17,920 ppm of nickel was prepared for the soaking process. The activated carbon was soaked in the nickel sulfate solution in two stages, with each stage containing an equal amount of nickel.

In the first stage of soaking, the remaining nickel content was measured at 5,080 ppm. In the second stage, the nickel content was calculated to be 3,955 ppm. These results indicate a high nickel loading efficiency of 72% on the activated carbon. During the second stage, the nickel loading rate on the activated carbon reached 78%.

This high loading rate can be attributed to the availability of large adsorption sites that effectively trap heavy metal ions. The presence of active functional groups, such as hydroxyl and carboxyl groups, enhances surface interactions with these ions. Additionally, the internal structure of the carbon is proportional to the size of the ions, facilitating their entry into the carbon's internal framework. These findings align with recent studies, such as Study [28-29], which demonstrated that activated carbon derived from agricultural or industrial waste exhibits high adsorption efficiency for heavy metal ions, including nickel. The study also indicates that repeated treatment (from Step 1 to Step 2) resulted in a significant improvement in efficiency (from 5080 to 3955 ppm), highlighting the importance of contact time and the amount of adsorbent in the removal of contaminants. Indicating that contact time and the quantity of adsorbent play important roles in pollutant removal. According to (Table 4).

**Table 4 atomic absorption data of activated carbon**

N.O	Ni ppm
Nickel sulfate before loading	17920
Step 1	5080
Step 2	3955

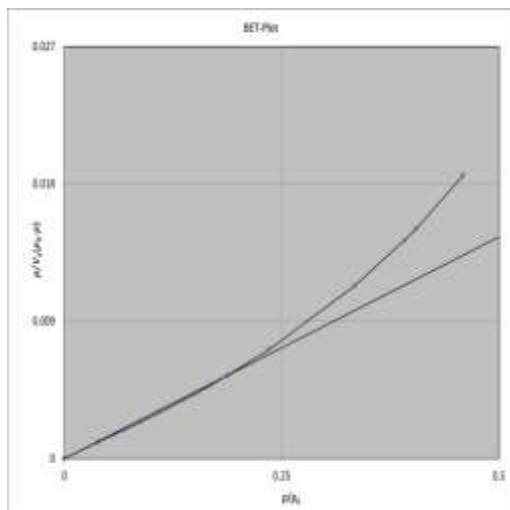
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**Surface Area Emission Testing (BET) Measurement: Brunner Amit Teller**

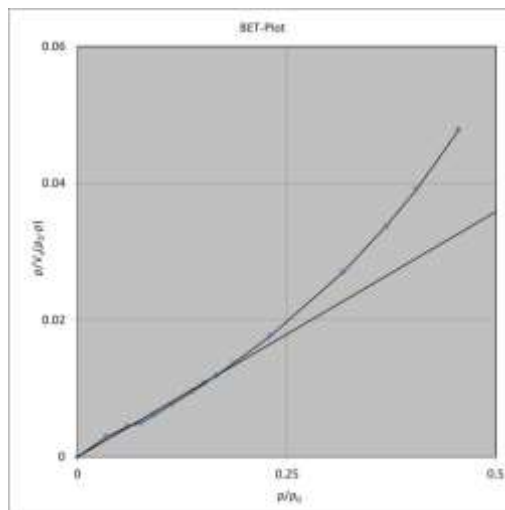
BET analysis results revealed a significant contrast in the surface properties between the raw activated carbon and the sample loaded with nickel sulfate (NiSO<sub>4</sub>). Table 5 presents the BET analysis results, indicating that the prepared activated carbon C6 had a surface area of 203 m<sup>2</sup>/g. Upon loading with nickel sulfate, this surface area decreased to 130.72 m<sup>2</sup>/g, while the total pore volume increased from 0.035542 cm<sup>3</sup>/g to 0.085559 cm<sup>3</sup>/g. This enhancement is attributed to the crucial role of nickel in modifying the porous structure during preparation, as illustrated in Table 5 and Figures 4a and 4b below.

**Table 5: Surface Area Emission Testing (BET) of activated carbon**

The property	before loading	after loading
Surface area(m <sup>2</sup> /g)	203.42	130.72
Pore volume cm <sup>3</sup> /g	0.085559	0.035542
Pore diameter nm	2.2857	2.3404



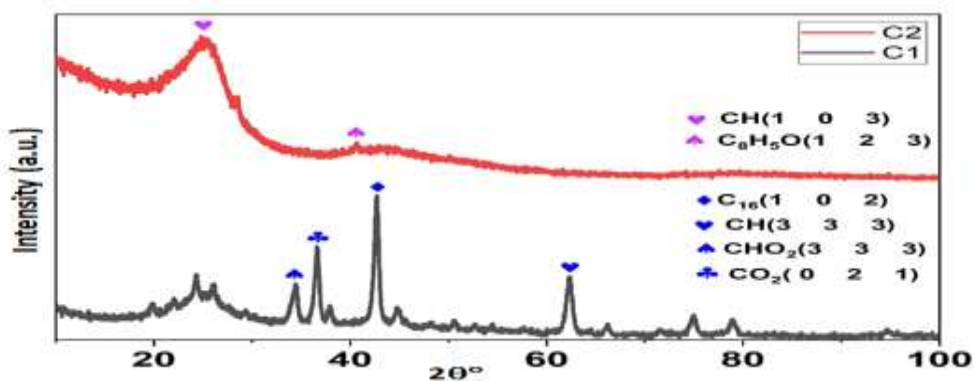
A- Before downloading



B- After downloading

### 7- X-ray diffraction (XRD)

Studying the properties of materials necessitates conducting various processes on the material, whether thermal or mechanical. These processes lead to the formation of a crystalline structure, which subsequently influences the material's properties. Understanding these processes is essential for assessing the efficiency of the product. The most critical parameter for evaluating material properties is the crystalline structure. Consequently, an X-ray diffraction analysis was conducted on a raw carbon sample used in the activated carbon preparation process, as well as on a prepared activated carbon sample, to assess the crystalline structure and determine its degree of crystallinity or amorphous nature. The results are illustrated in the figures below:



The X-ray diffraction (XRD) pattern of sample C1 reveals distinct crystalline peaks at  $2\theta = 26.5^\circ$  and  $43.3^\circ$ , corresponding to reflections from the (002) and (100) planes, respectively, which are indicative of the graphite phase. This pattern suggests the presence of a semicrystalline structure within the material, likely resulting from thermal decomposition under relatively mild conditions. The appearance of additional peaks at higher angles may signify impurities, such as metal oxides, which often persist from the initial materials or arise from chemical activation processes. These peaks indicate that the carbon retains some of its ordered structure, thereby enhancing its electrical conductivity and its ability to interact with organized surfaces. This type of carbon is particularly beneficial for applications that require partial crystal stability, such as catalysis or energy storage.

$2\theta^\circ$

In contrast, the X-ray diffraction (XRD) pattern of sample C2 exhibited a broad spectrum devoid of distinct crystalline peaks, particularly in the range of 20° to 30°. This observation indicates a completely amorphous nature. This phenomenon can be attributed to a high thermal activation process or the use of catalysts, which resulted in the disintegration of the crystal structure and the formation of an irregular carbon structure. This finding confirms the successful and practical preparation of activated carbon in this study. Such an amorphous structure is ideal for adsorption applications, as it is associated with a high surface area and a wealth of active sites.

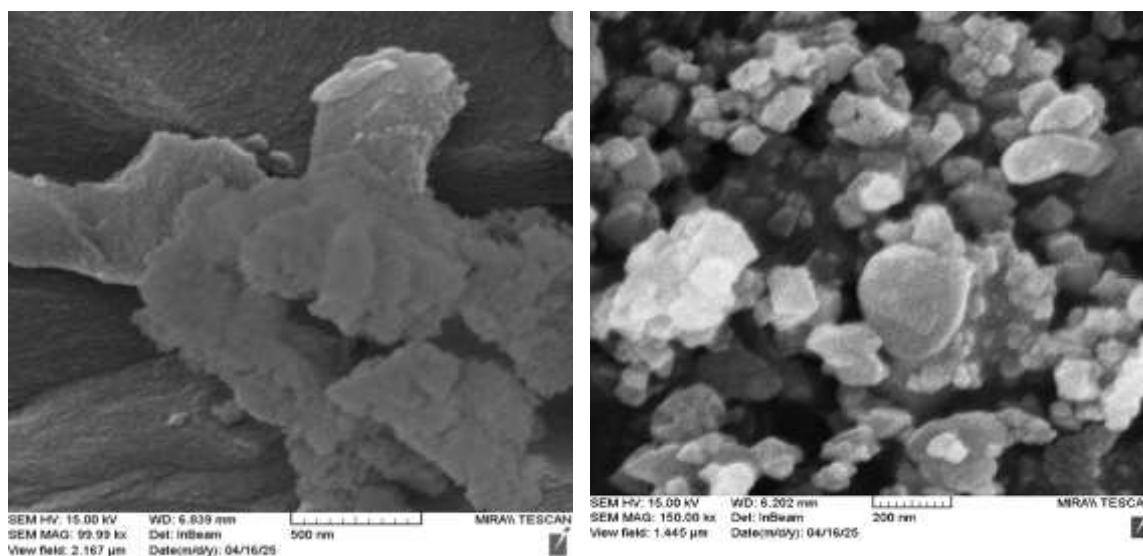
Table (6) indicate the structural properties of activated carbon before and after loading with nickel

Property	Sample C1	Sample C2
Crystal Pattern	Sharp graphite phase peaks	Wide curves, no sharp points
Characteristic Angles	54.7°, 43.3°, 26.5°	20°–30° wide range
Crystal Density	High intensity	Low intensity
Potential Applications	Electrical conductivity, catalytic	Adsorption, contaminant removal

Thus, the X-ray diffraction (XRD) results revealed a distinct difference between the two samples. Sample A exhibited a semi-crystalline structure, whereas Sample B was entirely amorphous. These findings confirm that the degree of crystallinity significantly influences the functional performance of activated carbon. An irregular structure is associated with enhanced environmental adsorption efficiency, while an ordered structure is advantageous for electromechanical applications.

#### 8-Scanning electron microscope SEM :

The samples were imaged using scanning electron microscopy (SEM). The images of the activated carbon before and after loading by nickel demonstrate that the grain size in both cases was within the nanoscale. Additionally, there was an increase in grain size after nickel loading, attributed to the growth and aggregation of the grains.



Figure(6) Scanning electron microscope of activated carbon before and after loading by nickel

#### CONCLUSIONS

This study demonstrated that combining asphalt with sulfur-rich foam residues is an effective and cost-efficient strategy for producing activated carbon with promising adsorption characteristics.

The adsorption results for methylene blue and iodine indicated that samples C5, C6, and C7 exhibited the highest adsorption capacities, demonstrating the development of micro- and mesoporous structures that are well-suited for capturing organic pollutants.

Microwave-assisted activation significantly improved the adsorption properties compared to steam activation alone, as evidenced by the notable increase in the iodine number. This underscores the effectiveness of microwave energy in enhancing pore formation and surface area.

X-ray diffraction (XRD) analysis revealed a predominantly amorphous structure in most activated carbon samples, confirming the disruption of crystallinity during the carbonization and activation processes, which enhances surface reactivity.

The BET surface analysis of NiSO<sub>4</sub>-loaded samples demonstrated a significant increase in both specific surface area and pore volume. This indicates that the incorporation of nickel enhances structural modification and improves porosity without leading to pore blockage.

Atomic absorption analysis confirmed the material's high efficiency in removing Ni<sup>2+</sup> ions from aqueous solutions, reducing concentrations from 17,920 ppm to less than 4,000 ppm. This underscores the material's potential for heavy metal remediation.

Overall, the findings emphasize that meticulous control of precursor ratios, activation methods, and process parameters is crucial for tailoring the textural and functional properties of activated carbon for environmental applications.

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