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Characteristics of Starch-Based Carbon Foams Filled with Zeolite and Activated Carbon

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Activated carbon, Carbon dioxide absorption, Compressive strength, Density, Porosity, Starch-based carbon foam, Zeolite

Abstract

This study investigates the development of starch-based carbon foam composites enhanced with zeolite and activated carbon fillers to improve structural, mechanical and absorption properties for sustainable material applications. This research addresses the environmental challenges of fossil fuel-based materials and aims to produce environmentally friendly alternatives using renewable tapioca starch. A systematic methodology was adopted, starting with preparing samples containing various concentrations of zeolite and activated carbon (0%, 2%, 4%, 6%, and 8%). The process involved mixing, drying and carbonization at 800°C, followed by extensive characterization using density and porosity tests, compression tests, Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and carbon dioxide absorption analysis. The results showed that increasing the filler content increased the density, reduced the porosity and increased the compressive strength. The zeolite filler was more effective in reducing porosity and increasing the density, while the activated carbon filler exhibited superior mechanical strength and stress distribution due to its better compatibility with the starch matrix. SEM and FTIR analyses confirmed the structural and chemical changes, highlighting the stronger bonding of the activated carbon with the matrix. Both fillers increased the carbon dioxide absorption capacity, with zeolite showing a higher absorption rate at a concentration of 6%. These findings underline the potential of these composites for environmental applications, especially in CO_2 capture and storage. In conclusion, this study provides insights for optimizing the filler concentration to tailor the material properties for specific applications, advancing sustainable materials for green technologies. Future work should explore thermal insulation, electrical conductivity, and chemical stability for broader applications.

1. Introduction

Carbon foam is an advanced structural material that is praised for its advantages, including low density, strong mechanical strength and electrical conductivity and aids in absorption and catalysis. The qualities of carbon foam make it useful for various applications, including thermal insulation, electromagnetic interference shielding, filtration and energy storage in batteries and supercapacitors [1,2]. This thesis explores the enhancement of starch-based carbon foams by combining zeolite fillers and activated carbon at different concentrations, aiming to create high-performance materials for aerospace, military and commercial applications from renewable resources instead of fossil fuels in producing foamed carbon.

Blowing and carbonization and template carbonization are two common approaches to creating carbon foams. This process converts organic materials to carbon while maintaining their porous structure. Carbonization is a stabilization process in which the foam material is heated in an oxidizing atmosphere to crosslink the polymer chains, increasing rigidity and structural stability. This technique is important for tailoring the properties of carbon materials for specific applications such as adsorption or catalysis. Both methods offer significant benefits for synthesizing carbon-based compounds, such as CO_2 adsorption and energy storage, and provide fine control over the pore size and distribution. Template carbonization produces carbon foams with a consistent porous structure suitable for energy storage, catalysis, and adsorption [1,2,3,4].

Combining carbon foam with activated carbon and zeolite produces a composite material with enhanced adsorption properties. Activated carbon is a highly porous material created by treating carbon-rich materials with heat or chemicals to increase the surface area and adsorption capacity. It is effective for water and air purification, gas adsorption, and chemical filtration. Activated carbon is particularly effective at absorbing carbon dioxide (CO_2) due to its large surface area and microporous structure. Zeolites, a group of natural and synthetic minerals with a unique porous structure, are particularly effective at adsorbing gases, including CO_2. Optimizing the composition of zeolites is predicted to increase the CO_2 adsorption capacity [6].

The rising demand for carbon foams poses environmental issues due to using fossil fuel-based precursors. Incorporating activated carbon or zeolite into carbon foams enhances characteristics such as density, absorption, thermal conductivity, and hydrophilicity, which makes them ideal for applications like air and water filtration and absorption. This research intends to create carbon foam composites from tapioca starch by incorporating activated carbon zeolite fillers at varying concentrations (0%, 2%, 4%, 6%, and 8%), with an emphasis on the structural, physical, mechanical, and absorption properties of the carbon foams. The study will examine the foaming and carbonation processes and perform thorough physical and mechanical evaluations, including tests for density, porosity, compression, FTIR, SEM, and carbon dioxide absorption on the carbon foams.

2. Methodology

The methodology section outlines the processes involved, including mixing, drying, and carbonization, for producing carbon foam composites from tapioca starch by incorporating zeolite and activated carbon fillers. Various physical testing methods, such as density measurement, porosity assessment, compression testing, as well as advanced techniques like SEM, FTIR, and carbon dioxide absorption analysis, were employed to assess the foam's structure and mechanical properties. This method aligns with prior research on starch-based carbon foams and examines the impact of varying concentrations of zeolite and activated carbon on the characteristics of the foam, thereby contributing to research on sustainable materials.

2.1 Sample Preparation

2.1.1 Mixing Process

The ingredients used for the preparation of the carbon foam sample were tapioca starch ($C_6H_{10}O_5$), sodium bicarbonate ($NaHCO_3$), distilled water (H_2O), zeolite powder, and activated carbon powder. The method of making carbon foam begins with the creation of a sodium bicarbonate solution. In this step, 3.5 grams of sodium bicarbonate are carefully combined into 160 grams of distilled water, and the liquid is continuously blended for 10 minutes to guarantee complete sodium bicarbonate dissolution. Next, the zeolite or activated carbon powder is mixed in with the 350 grams of tapioca starch, enhancing the mixture and laying the groundwork for the subsequent phases in carbon foam manufacturing. The bicarbonate solution is then added to a mixer along with the mixture. After 25 minutes of gradual mixing, the starch transforms into lumps and reaches the ideal consistency. Table 1 shows the mixing ratio used for zeolite and activated carbon with starch, and Fig. 1 shows a sample of starch and a mixture of zeolite or activated carbon with starch.

Table 1: Ratio of zeolite and activated carbon mixture to the starch.

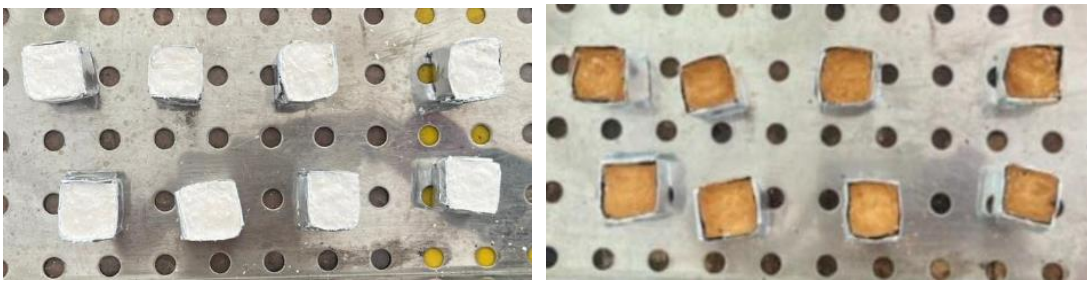
Filler	Name of sample	Mass Ratio in gram (Starch: Filler)
Zeolite	CFZ (0%)	0
	CFZ (2%)	120:2.4
	CFZ (4%)	120:4.8
	CFZ (6%)	120:7.2
	CFZ (8%)	120:9.6
Activated Carbon	CFAC (0%)	0
	CFAC (2%)	120:2.4
	CFAC (4%)	120:4.8
	CFAC (6%)	120:7.2
	CFAC (8%)	120:9.6

(a) (b) (c)

Fig. 1: *Sample mixture (a) starch, (b) starch mixed with Zeolite, (c) starch mixed with Activated Carbon.*

2.1.2 Carbon Foaming

The samples were dried in an oven for one hour at 200 °C. This drying process improved the sample's structure and properties for various applications, including air and water filtration, by removing moisture and preparing it for subsequent activation operations. This procedure eliminated moisture and enhanced the viscosity of the precursor substance through a dehydration reaction, as illustrated in Fig. 2.



(a) (b)

Fig. 2: *Sample before (a) and after the foaming process (b).*

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2.1.3 Carbonization

Carbonization involves heating the sample in an argon atmosphere at a temperature of 800°C. This method is carried out in a tube furnace, where the foam is exposed to the appropriate temperature for two hours at a constant heating rate of 10 °C per minute. The temperature profile for this process is illustrated in Fig. 3. The use of an argon atmosphere is necessary because it creates an oxygen-free environment, preventing oxidation during the carbonization process. The carbon foam resulting from the carbonization process is shown in Fig. 4.

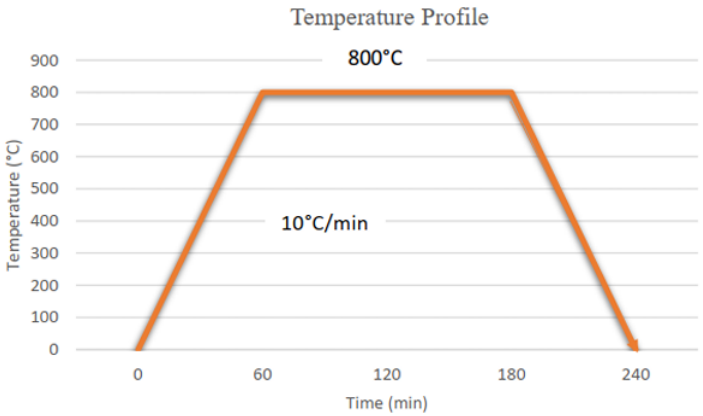

Fig. 3: *Temperature profile for carbonization process*

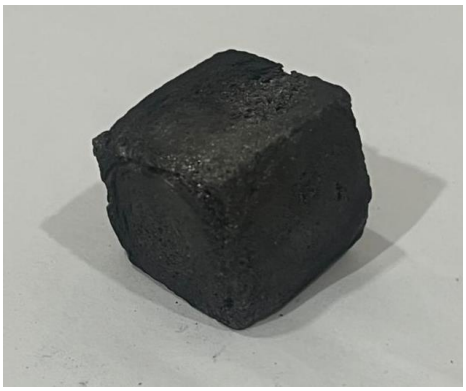

Fig. 4: *Carbon foam sample after carbonization*

3. Results and Discussion

This chapter presents and analyzes the results from laboratory experiments on various carbon foam samples. The samples were named (CFZ) for Carbon foam with zeolite filler and (CFAC) for Carbon foam with activated carbon filler. Subsequently, samples with different filler percentages are shown for each carbon foam, namely 0%, 2%, 4%, 6% or 8%. A total of eight samples were prepared for each parameter. A total of five samples underwent mechanical and structural evaluations, with dimensions measuring 20.0mm. These evaluations included compression tests to assess mechanical strength, along with measurements of density and porosity to evaluate physical properties. Scanning Electron Microscopy (SEM) was utilized to analyze the microstructure, while Fourier Transform Infrared Spectroscopy (FTIR) helped identify the chemical functional elements present. Furthermore, three samples from each group were analyzed for their capacity to absorb carbon dioxide. The results from these tests were average to yield comprehensive findings.

3.1 Physical Properties

3.1.1 Density and Porosity

The density and porosity of carbon foam samples incorporating zeolite and activated carbon fillers were assessed using a technique grounded in Archimedes' principle. Porosity represents the fraction of volume occupied by pores in the material. Simultaneously, the porosity of the material was evaluated by analyzing the void spaces within the foam structure. There is a close relationship between the porosity of the material and its density. It can be stated that an increase in density leads to a decrease in porosity. This technique involves initially weighing the dry sample, then immersing it in boiling water to ensure complete saturation for measuring the submerged weight and finally weighing it while it remains submerged to ascertain the submerged weight. A volume of 1 cm^3 of the carbon foam sample was measured.
The density of carbon foam is calculated using the formula:

$$\rho = \frac{m}{V} \tag{1}$$

where ρ is the density, m is the mass, V *is the volume*.

$$B = \frac{D}{V} - \frac{D}{W - S} \tag{2}$$

where B is the bulk density, D is the dry weight of the sample, V is the external volume, W is the wet weight of the sample in air, S is the submerged weight of the sample.

The porosity percentage of carbon foam can be determined by:

$$P = \frac{W - D}{W - S} \times 100\% \quad (3)$$

Comparison between bulk density and porosity of carbon foam samples for zeolite and activated carbon fillers is illustrated in Fig. 5 and Fig. 6, respectively.

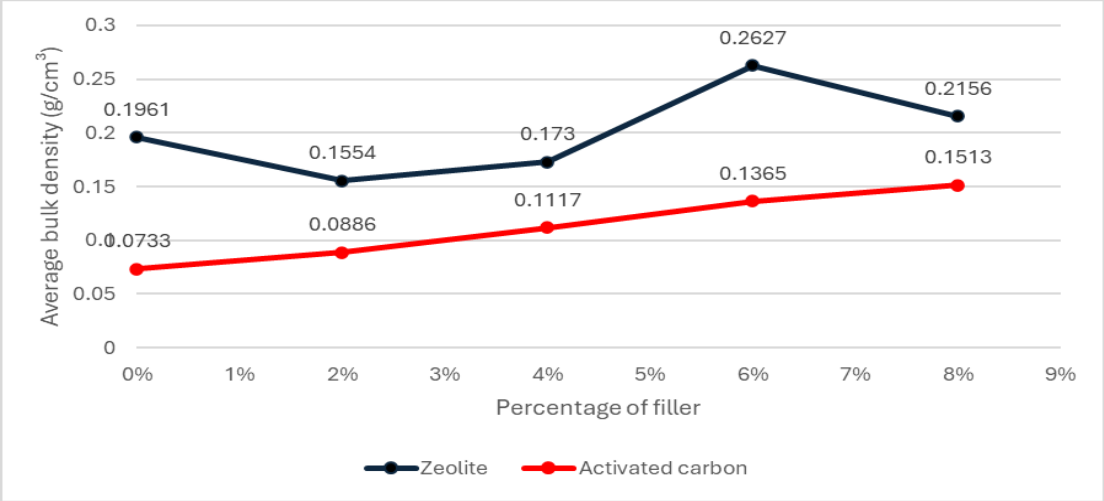

Fig. 5: *Bulk density comparison graph between zeolite and activated carbon fillers.*

The graph of carbon foam with zeolite-based fillers shows that the bulk density starts at 0% filler percentage, decreases slightly at 2%, and then increases at 6%. However, it decreases again at 8%. The crystalline arrangement of zeolite leads to an increased initial bulk density. When the filler percentages are low, the dispersion and incorporation within the matrix may result in a slight reduction in density. The observed 6% increase can be linked to improved packing efficiency as the zeolite particles occupy the voids, thereby enhancing the density. Conversely, the following reduction at 8% suggests that excessive loading results in poor packing or clumping, diminishing the overall bulk density.

Carbon foam incorporating activated carbon-based fillers exhibits a bulk density that starts at 0% filler and progressively increases up to 8%. The inherent porosity of activated carbon makes it naturally less dense. As the percentage of filler rises, its slightly porous characteristics increasingly contribute to the density of the composite material. This consistent rise could suggest a uniform distribution and integration within the matrix. In contrast, carbon foams containing zeolite display a higher bulk density owing to zeolite's denser and crystalline structure, with variations resulting from interactions between the fillers and the matrix, as well as packing efficiency. Meanwhile, activated carbon maintains generally lower bulk density because of its porous attributes but demonstrates a consistent upward trend as more material is introduced. The graph for carbon foams with zeolite and activated carbon filler shows a decrease in porosity with increasing filler content, which decreases from 0% to 8%. Zeolite particles can fill voids in the material matrix and reduce overall porosity. Their rigid structure and ability to occupy empty spaces contribute to this trend.

Zeolite has a more pronounced effect than activated carbon in reducing porosity due to its dense particle structure and better void-filling ability, as evidenced by Scanning Electron Microscopy (SEM) tests. Activated carbon has a naturally porous structure, resulting in a decrease in the overall porosity of the material while maintaining a larger number of interconnected pores. In conclusion, carbon foams with zeolite fillers are more effective at reducing porosity due to their dense structure and ability to occupy voids, making them suitable for applications with lower porosity.

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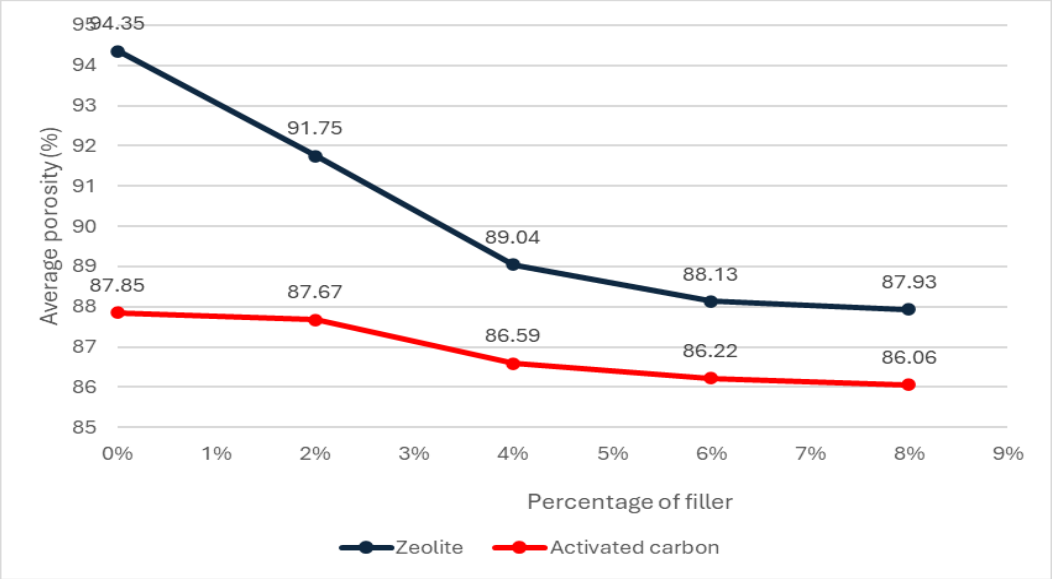

Fig. 6: *Porosity comparison graph between zeolite and activated carbon fillers.*

3.2 Mechanical Properties

3.2.1 Compression Test

The compression test conducted on carbon foam aims to evaluate its ability to endure compressive stress without failing or deforming. The mechanical properties of carbon foam samples were thoroughly examined using a NISTRON Universal Testing Machine (UTM) to investigate their compressive strength under load. Each sample, precisely shaped into a 20 mm cube, was subjected to compression at a regulated crosshead speed of 0.5 mm/min in accordance with ASTM C365 standards. The testing continued until the sample achieved its maximum load capacity, with the UTM apparatus calibrated to handle loads up to 5 kN. This evaluation yields essential information regarding the material's mechanical characteristics, such as compressive strength, yield strength, and modulus of elasticity, which are crucial for various applications in construction and manufacturing. Fig. 7 shows a graph of the maximum stress comparison between zeolite and activated carbon filler, while Fig. 8 shows a graph of the maximum strain comparison between zeolite and activated carbon filler.

The graph indicates an initial rise in maximum stress of up to 2% for zeolite fillers. The stress then experiences a significant decrease gradually from 4% to 8%. This decline can be linked to poor compatibility between the zeolite and the base material (carbon). The zeolite particles might not be uniformly distributed or effectively bonded within the matrix, resulting in weak areas and stress concentrations as the filler content increases. Consequently, this leads to diminished mechanical performance.

In contrast, the graph for activated carbon-based fillers shows a consistent rise in maximum stress as the filler content increases from 0% to 8%. This suggests that activated carbon demonstrates superior compatibility with the base material. The bonding and distribution of carbon in the matrix enhance its capacity to efficiently transfer and distribute stress, thereby augmenting the material's strength.

Zeolites display lower compatibility, which leads to microstructural inconsistencies that can hinder their effectiveness in managing stress as the filler content increases. Activated carbon serves as a more effective reinforcing filler due to its compatibility and its ability to enhance stress distribution, whereas zeolite, because of its weak interaction with the base material, undermines the structural integrity of the material as its proportion rises [6].

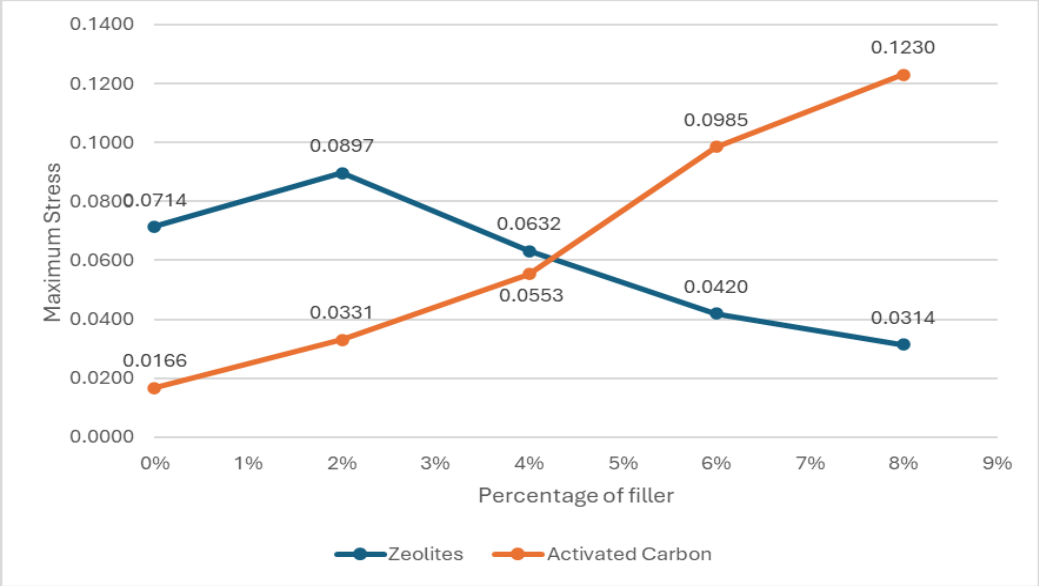

Fig. 7: *Maximum stress comparison graph between zeolite and activated carbon fillers.*

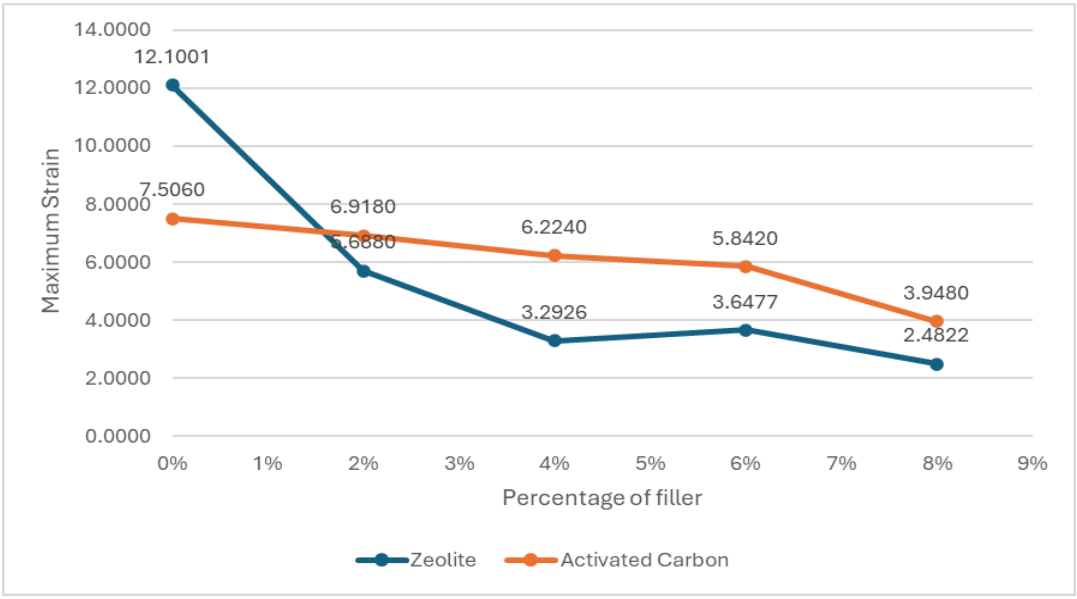

Fig. 8: *Maximum strain comparison graph between zeolite and activated carbon fillers.*

3.3 Sample Morphology

3.3.1 Scanning Electron Microscopy (SEM)

Scanning Electron Microscopy (SEM) is a flexible method that produces high-resolution images and detailed surface information from materials. Scanning Electron Microscopy (SEM) is a useful tool for analyzing organic and inorganic materials at the nanometer to micrometer scale. To investigate the morphology of carbon foam samples using a scanning electron microscope (SEM), a Hitachi S-4800 Type II FE-SEM instrument was used to examine the samples for each sample parameter and filler type. SEM investigation was used to closely examine the surface morphology and microstructure of carbon foam samples. SEM analysis allows observation of structural properties such as pore size, distribution, and shape, which provides valuable information about the internal architecture of the material. Table 2 shows the differences in sample structure at 300x magnification for zeolite and activated carbon fillers.

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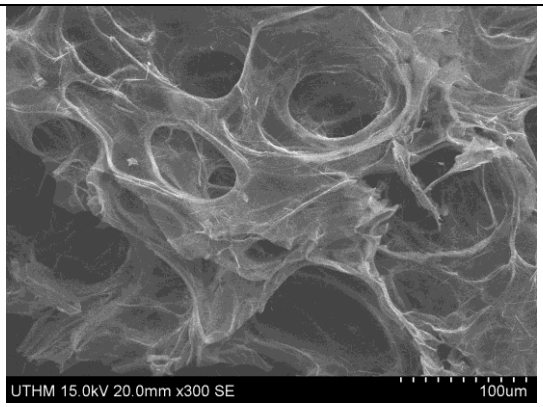
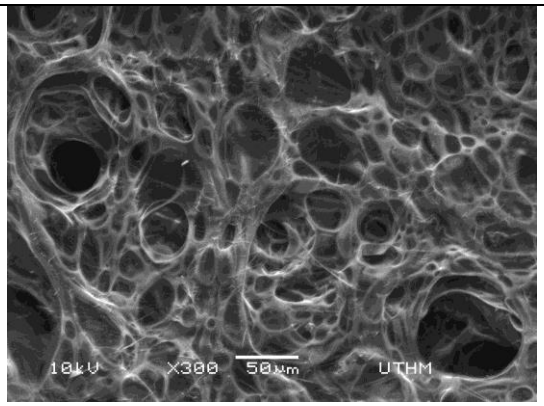
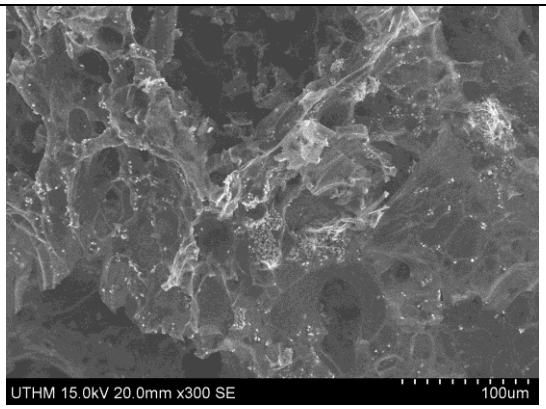
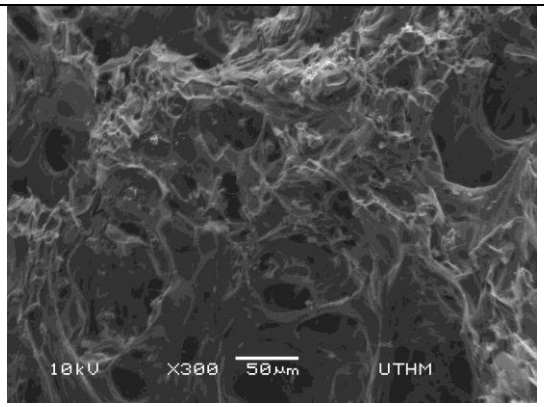
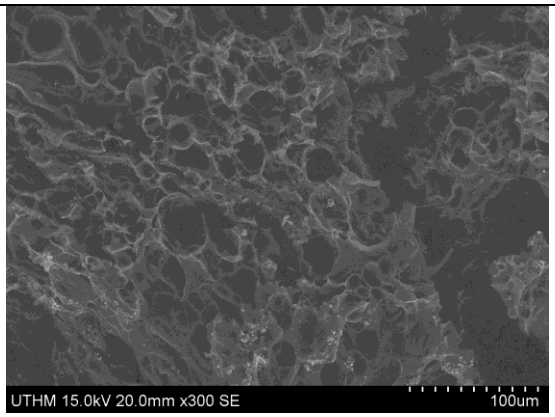
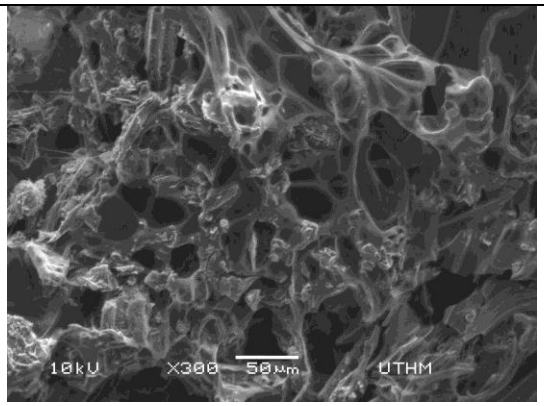
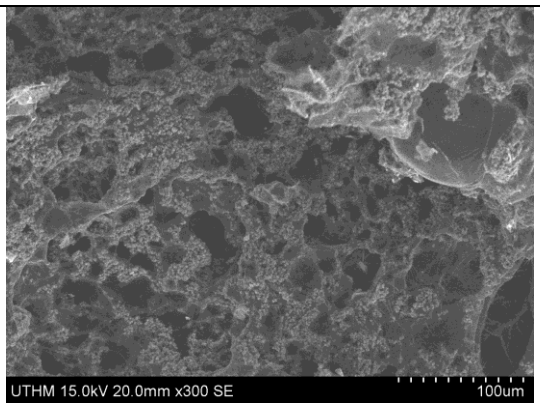
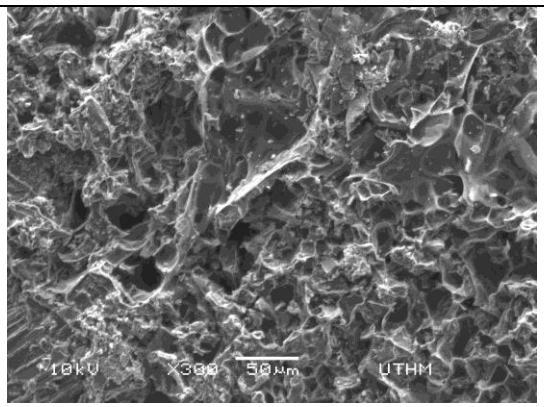
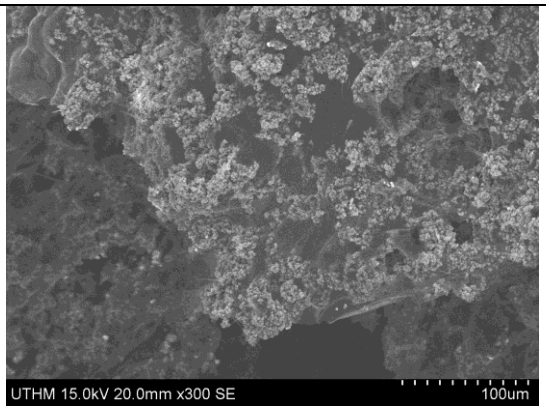
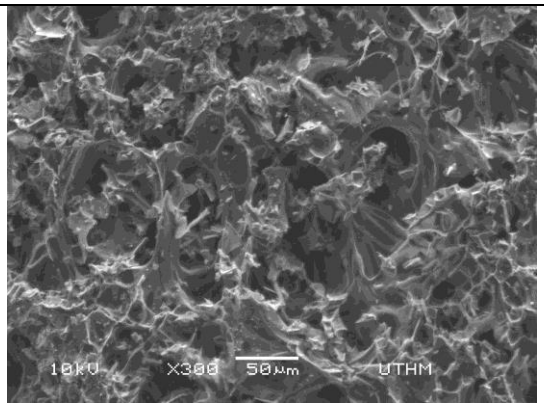
Table 2: *SEM Observation of Pores Structure under 300x magnification.*

Sample	Zeolite filler	Activated carbon filler
CF (0%)		
CF (2%)		
CF (4%)		
CF (6%)		

CF (8%)		

The morphology form and pore distribution in the foam matrix are seen at 300x magnification. According to the observations for both fillers, the average pore size decreases when the filler percentage increases from 0% to 8%. This reduction in pore size is directly related to the decrease in porosity. The addition of zeolite and activated carbon fillers contributes to the density of the carbon foam matrix, as the fillers fill the voids in the foam, reducing both the pore diameter and the overall porosity.

The walls of the pores become uneven and rough, signifying an increased level of interaction among the starch matrix, zeolite, and activated carbon particles. The presence of the filler creates smaller pores by occupying space within the carbon structure, thereby restricting the development of larger pores during the manufacturing process. Additionally, a higher concentration of filler results in a more compact structure as more voids are occupied, which decreases the overall air space in the material. Starch-derived foams exhibit more irregular pores compared to genuine carbon foams made from polymer precursors or phenolic resins, which generally possess a more uniform and denser carbon matrix with smaller and more consistent pore sizes [7].

3.3.2 Fourier-transform Infrared Spectroscopy (FTIR)

Fourier Transform Infrared (FTIR) spectroscopy is the preferred method for non-destructive analysis of samples, providing enhanced speed, sensitivity, and precision compared to earlier techniques. It employs a Fourier transform infrared spectrometer to assess the energy that is imparted to the sample through infrared radiation. Infrared spectroscopy depends on the unique absorption of infrared radiation caused by molecular vibrations (either stretching or bending). Different types of bonds or functional groups absorb energy at distinct frequencies, resulting in a specific emission pattern that facilitates accurate molecule identification. In the evaluation of carbon foam, FTIR is primarily employed to ascertain the functional groups within the material by grinding a sample into a fine powder and positioning it on the detector. This analytical approach can effectively identify the chemical makeup and molecular configuration of the sample. Fig. 9 and Fig. 10, respectively, illustrate the wavenumber of the raw material and carbon foam sample for zeolite filler. Meanwhile, Table 3 shows a comparison of functional groups and wavenumber for zeolite filler and activated carbon.

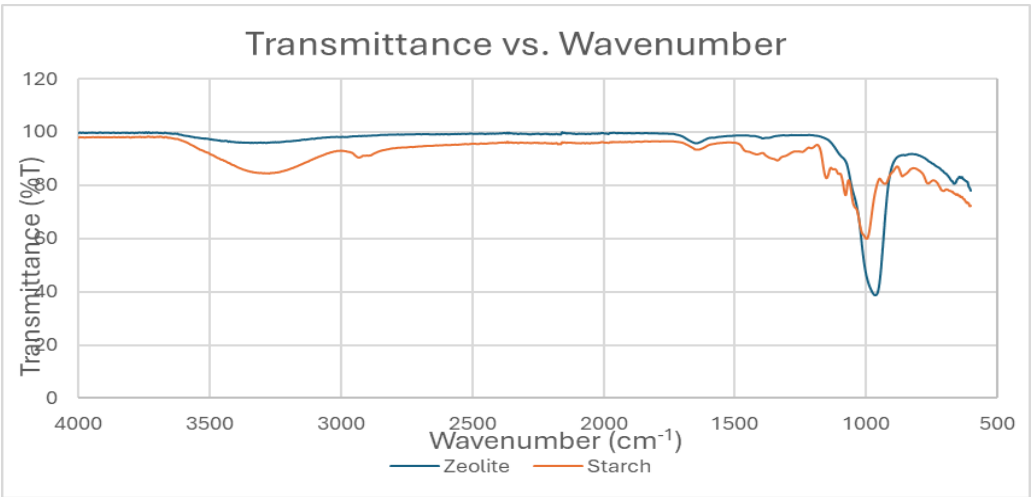


Fig. 9: *FTIR analysis of raw material samples.*

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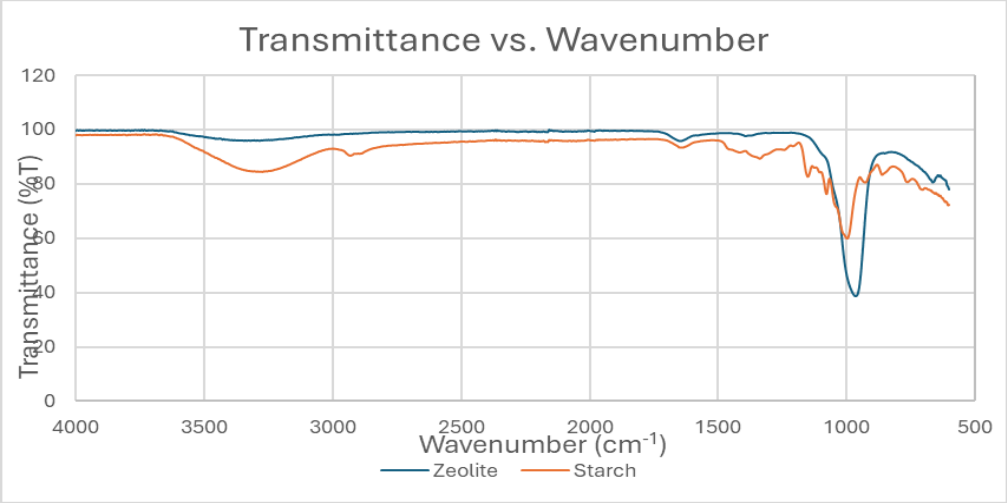

Fig. 10: *FTIR analysis of raw material samples*

Table 3: *Functional group and wavenumber for zeolites and activated carbons filler*

Functional group	Wavenumber (cm^{-1})							
	Zeolite				Activated Carbon			
	CFZ (2%)	CFZ (4%)	CFZ (6%)	CFZ5 (8%)	CFAC (2%)	CFAC (4%)	CFAC (6%)	CFAC (8%)
OH	-	-	-	-	3280.89	3267.46	3282.20	3280.35
CH	871.6	627.06	947.98	675.1	2932.99	2932.61	2932.46	2932.90
C=O	-	-	-	-	1638.77	1639.20	1639.05	1639.04
C-O-C	-	-	-	-	1338.65	1338.42	1338.36	1338.16
C-OH	995.09	-	-	-	999.08	998.80	998.70	997.12

The FTIR test results illustrate the characteristic functional groups found in zeolite and activated carbon fillers in carbon foams. For zeolite fillers, CH groups were detected at all concentrations (2%, 4%, 6%, 8%) at specific wavenumbers. C-OH groups were also consistently detected at all levels with wavenumbers ranging from 995.09 to 999.08 cm^{-1}. Whereas for activated carbon fillers, OH groups were detected at high wavenumbers (~3280 cm^{-1}). In addition, CH, C=O, and C-O-C groups were detected at all conditions with stable wavenumbers. C-OH groups were also consistently detected at wavenumbers ranging from 997.12 to 999.08 cm^{-1}.

Zeolites showed the presence of CH and C-OH groups, indicating that there were certain chemical interactions with the carbon foams. However, the absence of OH and C=O groups indicated that zeolites did not provide significant polar properties. Therefore, zeolites are likely to only form physical bonds with foamed carbon without strong chemical interactions. On the other hand, activated carbon shows the presence of polar groups such as OH, C=O, and C-O-C, which potentially result in stronger chemical interactions with foamed carbon. Previous studies have shown that FTIR is also used in operando studies to monitor changes in the structure of zeolites during chemical reactions. For example, studies on the decomposition of low-density polyethylene (LDPE) have shown that the pore structure and acidic nature of zeolites can accelerate catalytic reactions [8].

3.4 Carbon Dioxide Absorption

The carbon dioxide (CO_2) absorption test of carbon foam is an important evaluation method to measure the ability of a material to absorb CO_2. Initially, the sample is heated in an oven for 15 minutes at a temperature of 100 degrees to ensure it is dry and free of moisture before recording its initial mass. Next, the carbon foam sample is positioned in a sealed absorption chamber using a tube furnace, ensuring there is no gas leakage. Each sample is exposed to CO_2 gas for 30 minutes to aid in the absorption of carbon dioxide into the carbon foam structure. After the absorption process, the sample is carefully taken out, and its final mass is assessed to evaluate the quantity of carbon dioxide absorbed. Fig. 11 compares the percentage graphs of the carbon dioxide

absorption capacity of zeolite and activated carbon fillers. The absorption capacity can be calculated using the formula:

$$\text{Percentage of absorption, } \% = \frac{Final\ mass - Initial\ mass}{Initial\ mass} \times 100\% \quad (4)$$

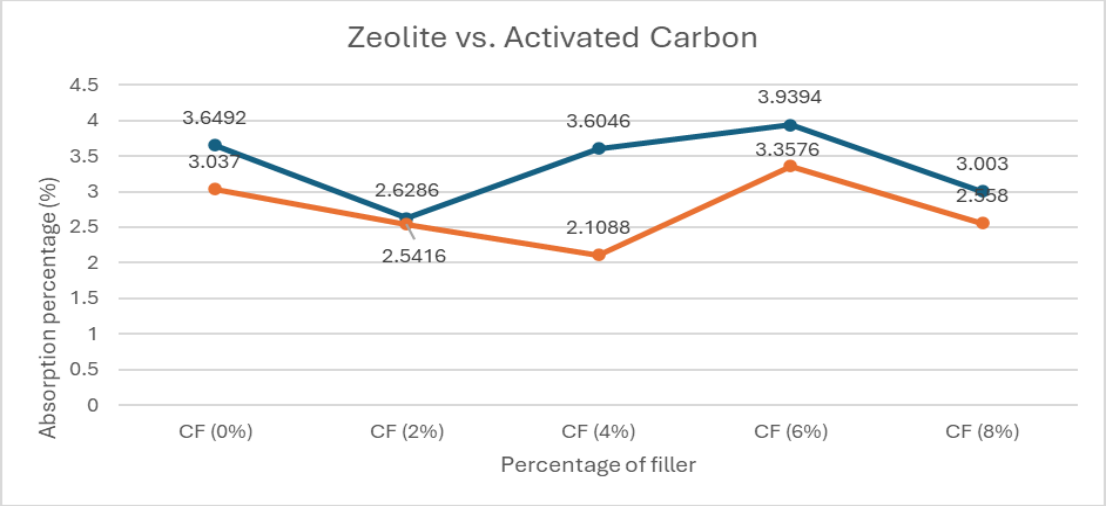

Fig. 11: *Comparison graph of the percentage of carbon dioxide absorption capacity of zeolite and activated carbon filler*

The results of carbon dioxide adsorption tests for starch-based carbon foams with varying zeolite and activated carbon contents (0%, 2%, 4%, 6%, and 8%) showed that zeolite fillers had a relatively higher adsorption percentage than activated carbon. Both fillers demonstrated optimal performance at a concentration of 6%, but their effectiveness diminished when added at 8%, potentially due to decreased pore blockage or fewer active surface sites. Zeolite exhibited a greater overall adsorption rate compared to activated carbon, particularly at the 6% filler concentration. This is attributed to zeolite's well-structured pore design and enhanced adsorption capacity, as evidenced by research on zeolite 13X, which demonstrated significant efficacy [9].

A study pointed out that zeolite, like type 5A, showed exceptional CO_2 adsorption capabilities at low pressures and room temperature, aligning well with the needs of carbon foams. These materials are especially efficient when integrated into composite formats, enhancing gas adsorption performance [10]. Previous research has indicated that zeolite frameworks, such as zeolitic imidazolate frameworks (ZIF), improve CO_2 capture by substantially boosting the material's surface area and porosity. When these frameworks are added to composite matrices, including cellulose or carbon-based foams, they enhance both the selectivity and capacity for CO_2 adsorption [11].

Conclusion

This research successfully demonstrated the production of carbon foam using cassava starch through a foaming process, demonstrating the feasibility of using renewable natural resources as a cost-effective alternative to synthetic materials. The findings revealed that adding zeolite and activated carbon fillers significantly affected carbon foams' physical and mechanical properties. The results showed that incorporating zeolite and activated carbon into starch-based carbon foams significantly affected their properties. Carbon foams containing zeolite exhibited higher bulk density than activated carbon due to the denser and more crystalline structure of zeolite. The maximum stress for the zeolite-based fillers showed poor compatibility between zeolite and the base material (carbon) compared to activated carbon, which showed a steady increase in the maximum stress. The maximum stress between zeolite and activated carbon fillers showed that the maximum strain decreased as the percentage of fillers increased, indicating a decrease in flexibility in the carbon foam structure. The Fourier-transform infrared spectroscopy (FTIR) spectrum of activated carbon showed that it was more compatible with carbon foam than zeolite due to the presence of polar groups such as OH and C=O, which allowed stronger interactions with the carbon foam structure. Scanning Electron Microscope (SEM) images also showed a denser packing of zeolite particles in the foam matrix compared to activated carbon, reducing the pore size and increasing the overall density. In terms of carbon dioxide absorption performance, both zeolite and activated

carbon enhanced the absorption capacity of carbon foam to a certain extent, reflecting the important role of fillers in improving the performance of carbon foams. In conclusion, by adjusting the filler concentration, the material properties can be optimized for specific applications, advancing the development of sustainable materials for green technology and environmental management. Future research should explore the real-world applications of carbon foams, further improve the understanding of the adsorption capacity of carbon foams, investigate the heat transfer and insulation capabilities of carbon foams, and perform additional tests for electrical conductivity, flame resistance, and chemical stability.

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Conflict of Interest

The authors declare that there is no conflict of interest regarding the paper's publication.

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