

ABSORPTION STUDIES AND SURFACE CHARACTERIZATION ON THE REMOVAL OF METHYLENE BLUE FROM A SOLUTION USING RAW AND ACTIVATED CARBON DERIVED FROM *PERSEA AMERICANA* SEED

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ABSTRACT

Textile effluents contribute significantly to industrial water pollution, accounting for about 20% of contamination and often containing stable dyes such as methylene blue (MB), which present serious environmental challenges. This study investigated raw and treated avocado (*Persea americana*) seed powders as inexpensive adsorbents for MB removal. Analytical techniques including SEM–EDX, XRD, and BET showed that the untreated sample had a dense structure, limited porosity, and a moderate surface area of 227.48 m²/g. After activation, the sample showed notable improvements, including a well-developed porous structure, a higher surface area of 863.21 m²/g, and an increase in micropore volume from 0.198 to 0.779 cc/g. XRD results indicated a shift from largely amorphous material to partially graphitized carbon, while EDX analysis revealed increased carbon content and reduced oxygen functionalities. These structural changes enhanced adsorption performance, mainly through physical interactions. The findings suggest that activated avocado seed is an effective, sustainable option for treating dye-polluted wastewater.

INTRODUCTION

The textile industry remains one of the most resource-demanding sectors worldwide and contributes significantly to global water pollution. This sector accounts for nearly 20% of industrial water contamination, mainly because it relies heavily on synthetic chemicals, dyes, and large volumes of water and energy (Panhwar *et al.*, 2020). During textile production processes such as dyeing, bleaching, and finishing, textile mills generate enormous amounts of wastewater. These effluents typically contain complex mixtures of synthetic dyes, surfactants, salts, and other toxic additives that pose serious environmental and health hazards (Hole *et al.*, 2019; Inyinbor *et al.*, 2016).

Methylene blue (MB), a cationic thiazine dye, is widely used in the textile industry because of its high solubility and stability in aqueous media. Despite its industrial importance, MB is hazardous to ecosystems because of its toxic nature and persistence in water bodies (Cheng *et al.*, 2020; Mak *et al.*, 2021). Once released into aquatic systems, MB can alter water aesthetics, reduce light penetration, and disrupt photosynthesis, ultimately destabilizing aquatic food chains (Khan *et al.*, 2021). The removal of MB and related synthetic dyes from industrial effluents is, therefore, essential. However, conventional treatment methods such as biological degradation, chemical oxidation, ozonation, and membrane filtration often show limited efficiency because of the dyes' complex aromatic structures, high stability, and resistance to degradation (Munonde *et al.*, 2022; Dutta *et al.*, 2022). Among

available techniques, adsorption stands out as a practical and cost-effective approach because of its fast kinetics, simplicity, and high efficiency in removing dyes from aqueous solutions (Hamzezadeh *et al.*, 2022).

Synthetic dyes in textile effluents are particularly problematic because they are not readily biodegradable, and even low concentrations as little as 1 ppm can impart intense coloration to water, making it unsuitable for human consumption (Hoong *et al.*, 2018). Furthermore, persistent dyes in aquatic environments disrupt light transmission, reducing photosynthesis and oxygen balance and threatening aquatic biodiversity (Shakoor *et al.*, 2016; Khan *et al.*, 2021).

Textile manufacturing generates wastewater at multiple stages, including scouring, bleaching, desizing, mercerization, dyeing, and finishing. The desizing and dyeing steps are particularly water-intensive and contribute the highest levels of toxic compounds to effluents (Chaari *et al.*, 2019). Textile wastewater characteristics vary by process and chemicals used but generally include high levels of salts, surfactants, binders, fats, urea, acids, waxes, and trace metals such as chromium, copper, zinc, and arsenic. These contaminants harm aquatic ecosystems and human health (Scholz *et al.*, 2018).

Exposure to untreated textile effluents has been associated with skin irritation, respiratory issues, nausea, headaches, and long-term effects such as genetic mutations and organ damage (Yagub *et al.*, 2014; Yaseen & Scholz, 2018; Zhou *et al.*, 2019; Okareh *et al.*, 2017; Tang *et al.*, 2017). Moreover, dyes' strong resistance to photodegradation and thermal decomposition enables them to remain in aquatic systems for extended periods, reducing sunlight penetration and suppressing photosynthetic activity in algae. This disrupts the aquatic food web and further intensifies ecological imbalances (Gita *et al.*, 2017).

In recent years, biomass-derived adsorbents for wastewater remediation have attracted growing scientific interest because of their efficiency, environmental compatibility, and low cost. Unlike conventional adsorbents, biomass-based alternatives are sustainable and abundantly available. However, a common limitation is their tendency to leach soluble components into water during adsorption, which may affect performance stability (Gita *et al.*, 2017).

Among various pollutants, methylene blue (MB), a synthetic heterocyclic aromatic dye extensively applied in textiles and pharmaceuticals, has received significant attention. Its persistence in water and potential toxicity necessitate efficient removal strategies. Adsorption has emerged as one of the most reliable methods for mitigating its ecotoxic effects. Over the past two

decades, biochar production and application have gained prominence in environmental remediation. Biochar, an eco-friendly carbonaceous material, is generally produced via slow or fast pyrolysis of biomass without activation. Its cost-effectiveness stems from the global abundance of feedstock and its relatively simple production route. The final physicochemical characteristics of biochar depend on precursor type and pyrolysis conditions such as heating rate, temperature, and duration, which can be tailored to optimize adsorption efficiency (Gita *et al.*, 2017). Daniel *et al.* (2015) also examined MB adsorption mechanisms and found that acid activation enhanced adsorption capacity. Their thermodynamic results revealed that the process was entropy-driven, implying randomness at the solid-liquid interface. Furthermore, Iwuozor *et al.* (2021) systematically analyzed over 500 publications on methylene blue adsorption and concluded that composite adsorbents (31%) and biosorbents (16%) were among the most effective classes. Activated carbon and nanocomposites, in particular, demonstrated outstanding adsorption performance, underscoring the role of hybrid and engineered adsorbents in enhancing dye removal.

The purpose of this study is to evaluate the potential of avocado (*Persea americana*) seeds, both in their raw and activated forms, as adsorbents for the removal of methylene blue dye from their solutions, as well as a medium for absorption, size and surface characterization. To characterize the adsorbent prepared from raw and activated avocado seeds using Scanning Electron Microscopy Energy-Dispersive X-ray Spectroscopy (SEM-EDX), X-ray diffraction (XRD) and Brunauer-Emmett-Teller (BET).

MATERIALS AND METHODS

Materials

Electronic Weighing Balance (Mettler Toledo ME204), pH Meter (Hanna HI2211), Orbital Shaker (Benchmark Orbi-Shaker BT101), Filter Paper (Whatman Grade 1), Water Bath (Memmert WNB 7)

Chemicals and Reagents

Analytical grade reagents were used throughout the study. Sodium hydroxide (NaOH, 98%, BDH Chemicals Ltd., Poole, England), hydrochloric acid (HCl), and methylene blue (C₁₆H₁₈ClN₃S, MW: 373.9) were employed. All solutions were prepared with deionized water to avoid ionic interference (Abukhadra *et al.*, 2019).

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METHODOLOGY

Sample Collection and Treatment

Avocado seeds were collected from a local farm near St. Matthew Catholic Church, Furaka, Jos North, Plateau State, Nigeria. The seeds were thoroughly washed with distilled water to remove dirt and soluble impurities, then air-dried. The dried seeds were ground in a laboratory mill and sieved to 80 µm. The powder was stored in sealed plastic containers for further use as raw adsorbent without chemical modification (Olaekan *et al.*, 2020).

Preparation of Stock Dye Solution

A stock solution of methylene blue (1000 mg/L) was prepared by

dissolving 1 g of dye in 1000 mL of deionized water. Working solutions of desired concentrations (20–100 mg/L) were prepared by serial dilution of the stock solution (Memon *et al.*, 2020).

Analytical Methods

A calibration curve was obtained from standard methylene blue solutions (20–100 mg/L). The absorbance of each solution was measured at the maximum wavelength ($\lambda_{\text{max}} = 664 \text{ nm}$) using a UV-Vis spectrophotometer. The residual dye concentration in treated samples was determined by comparison with the calibration curve (Sharma & Kaur, 2022).

RESULTS AND DISCUSSION

Figure 1 presents Scanning Electron Microscopy (SEM) of raw avocado seed powder, revealing particle dispersion and morphological structure. The magnification was set to 100 µm, and the particle sizes of raw avocado seed powder ranged from 5–20 µm; the particles appeared as small, irregularly shaped granules. The SEM micrograph of the raw avocado seed powder shows a densely packed, compact surface morphology. The particles appear as small, irregularly shaped granules uniformly distributed across the sample, with sizes ranging from 5–20 µm. The raw powder surface is relatively smooth and compact, with minimal porosity and limited surface roughness. The granules are mostly spherical to oval, with smooth surfaces typical of unprocessed plant-based materials. The particles appear closely agglomerated, forming a dense matrix with very few visible cracks or voids. This morphology suggests the material has a low surface area and limited adsorption potential before activation or further treatment. The image was obtained at a magnification corresponding to a 100 µm scale bar, with an EHT of 25.00 kV and a working distance (WD) of 9.6 mm.

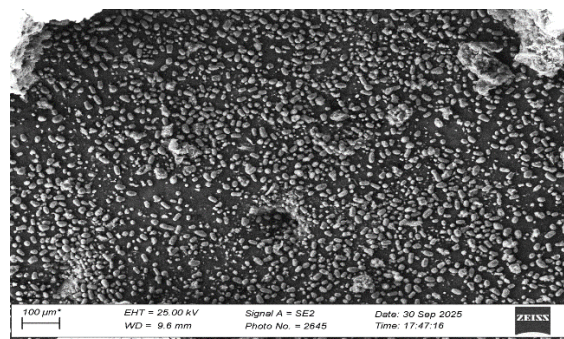


Figure 1: Scanning Electron Microscopy (SEM) for Raw Avocado Seed Powder

The Energy-Dispersive X-ray Spectroscopy (EDX) of raw avocado seed powder is presented in Figure 2. Revealing all the elements present in raw Avocado seed, the following elements were detected, including Carbon (C), Oxygen (O), Phosphorus (P), Sulfur (S) and Potassium (K). Carbon (C) had the highest intensity peak of 5.85k, while Phosphorus (P) was detected in small amounts at 0.02k.

The EDX spectrum of the same sample reveals the major elements present in the raw avocado seed powder. Carbon (C) shows the highest-intensity peak at 5.85 k, indicating it is the dominant element in the sample. This is expected, as carbon forms the primary framework of organic materials, originating mainly from cellulose, hemicellulose, lignin, proteins, and lipid components of

the seed. Oxygen (O) appears as the second most abundant element, associated with hydroxyl and carbonyl groups, as well as carbohydrates within the organic matrix. Phosphorus (P) is also detected in small amounts (0.02 k), likely due to the presence of phospholipids, nucleic acids, and other phosphorus-containing biomolecules. Minor peaks corresponding to sulfur (S) are observed, which may originate from sulfur-bearing amino acids such as cysteine and methionine found in the seed's protein content. Additionally, potassium (K) appears as small but distinct peaks, representing its role as a mineral nutrient inherent to plant tissue.

The SEM and EDX results of raw Avocado seed powder align closely with findings from several studies on the activation of

biomass-derived carbons. In the present analysis, the raw avocado seed powder showed a compact, smooth, non-porous morphology with tightly agglomerated particles measuring 5–20 μm . This morphology indicates limited surface area and poor adsorption capacity before activation. These results align with Mahaninia *et al.* (2020), who observed a dense, smooth morphology in raw walnut shell powder before chemical activation and attributed the lack of porosity to natural biopolymers such as cellulose and lignin that fill interparticle voids. Similarly, Adebisi *et al.* (2021) reported that untreated plant-based powders like coconut shell and date seed possess compact microstructures that restrict active adsorption sites.

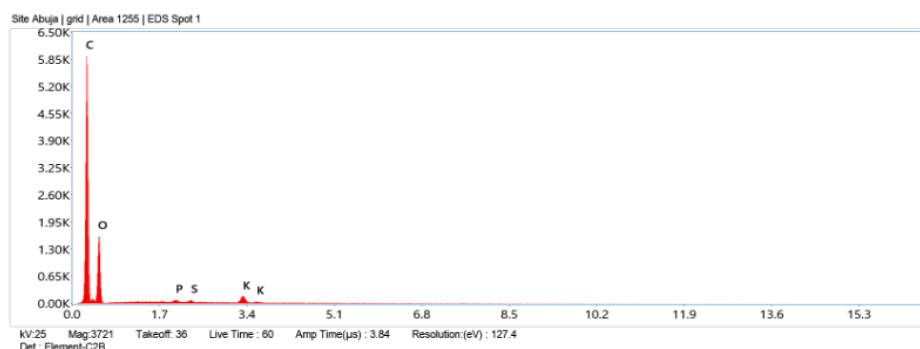


Figure 2: Energy-Dispersive X-ray Spectroscopy (EDX) for Raw Avocado Seed Powder

Scanning Electron Microscopy (SEM) of activated avocado seed powder in Figure 3 shows particle dispersion, morphology and surface modification of raw avocado seed powder. The surface appears highly porous, with numerous macropores, cavities, and irregular voids across the structure, indicating that the activation process produced a well-developed porous carbon network with pore sizes up to approximately 200 μm .

The scanning electron microscopy (SEM) image of the activated avocado seed powder reveals a distinct morphological transformation compared to the raw material. The surface appears highly porous, with numerous macropores, cavities, and irregular voids across the structure, indicating that the activation process produced a well-developed porous carbon network. The particles have irregular shapes and rough, uneven surfaces, ranging in size up to approximately 200 μm . These particles are larger and more heterogeneous than those of the untreated seed powder. The surface texture is coarse and rugged, with visible cracks, fissures, and deep pores, typical of activated carbon or biochar materials. This porous architecture and uneven texture suggest increased surface area, enhancing the material's adsorption potential for pollutants, gases, and other molecules. The bright or white regions in the SEM micrograph correspond to areas of high electron density, likely due to mineral accumulation or residual carbonization products. The sample was analyzed at a magnification with a 200 μm scale bar, using an accelerating voltage (EHT) of 25.00 kV and a working distance (WD) of 9.2 mm.

The SEM and EDX results of activated avocado seed powder align closely with findings from several studies on the activation of biomass-derived carbons. In this analysis, the raw avocado seed

powder exhibited a compact, smooth, non-porous morphology with tightly agglomerated particles measuring 5–20 μm . This morphology indicates limited surface area and poor adsorption capacity before activation. These results align with Mahaninia *et al.* (2020), who observed a dense, smooth morphology in raw walnut shell powder before chemical activation and attributed the lack of porosity to natural biopolymers such as cellulose and lignin that fill interparticle voids. Similarly, Adebisi *et al.* (2021) reported that untreated plant-based powders like coconut shell and date seed possess compact microstructures that restrict active adsorption sites.

However, the detection of lead (Pb) in the current study represents an anomaly compared to related research. Most studies, such as Dada *et al.* (2020) and Saka *et al.* (2022), reported negligible heavy metal contamination in activated carbons prepared under controlled conditions. The presence of Pb in this sample may therefore be attributed to contamination during activation or environmental exposure of the original seeds. Such contamination must be addressed to ensure safe application of the material, particularly in water purification or food-related uses. The transformation from a smooth, compact surface in the raw avocado seed powder to a highly porous structure after activation aligns with trends reported in similar biomass activation studies. Researchers such as Sulaiman *et al.* (2022), Yakout and Sharaf El-Deen (2016), and Hameed *et al.* (2019) consistently confirm that activation induces surface cracking, pore development, and carbon enrichment, all of which enhance the resulting material's adsorption potential. Thus, the morphological and elemental modifications observed in this study validate the effectiveness of the activation process and position the activated avocado seed powder as a

promising bio-adsorbent, provided contamination control is maintained.

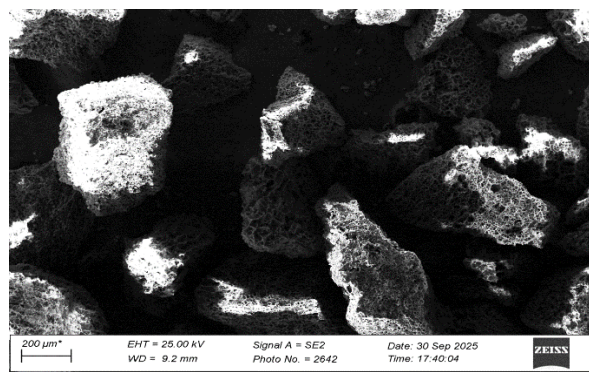


Figure 3: Scanning Electron Microscopy (SEM) for Activated Avocado Seed Powder

The Energy-Dispersive X-ray Spectroscopy (EDX) of raw avocado seed powder in Figure 4 shows all the elements present in activated Avocado seed powder, with Carbon (C) remaining the dominant element, with a peak intensity of about 1.53K counts, Oxygen (O), lead (Pb), Aluminum (Al), Molybdenum (Mo) and Potassium (K). Lead (Pb) is observed at different energy levels, and it was the lowest, as displayed (0.01)K counts

The energy-dispersive X-ray (EDX) analysis of the activated avocado seed powder shows notable changes in elemental composition. Carbon (C) remains the dominant element, with a peak intensity of about 1.53K counts, though slightly reduced compared to the raw sample. This reduction suggests partial decomposition of volatile organic compounds during activation. Oxygen (O) shows a marked decrease in intensity (approximately 0.34K counts), indicating the removal of oxygen-containing groups through dehydration, decarboxylation, and dehydroxylation reactions. Interestingly, several peaks corresponding to lead (Pb) appear at different energy levels, suggesting possible contamination during processing or storage, use of lead-containing activation chemicals, environmental exposure, or instrumental artifacts. Minor aluminum (Al) peaks are detected, which may

originate from activation agents or incidental contamination. A weak signal for Molybdenum (Mo) appears, possibly due to trace residues from equipment or reagents. Potassium (K) is present in small amounts, significantly lower than in the unactivated sample. The EDX results confirm a substantial reduction in oxygen content, consistent with successful carbonization and the removal of functional groups such as hydroxyl and carbonyl. The increased carbon-to-oxygen (C/O) ratio indicates carbon enrichment, typical of activated carbon materials. However, lead detection raises potential safety concerns, particularly for applications involving water purification or food contact. Despite this, the developed porous structure and altered elemental composition indicate that activated avocado seed powder has strong potential for adsorption-based applications, provided contamination issues are effectively addressed.

Following activation, the avocado seed powder in this study showed a dramatic morphological transformation, characterized by irregularly shaped particles with macropores, cavities, and fissure features that indicate the formation of a highly porous carbon network. This observation agrees with Sulaiman et al. (2022), who reported that thermal and chemical activation of avocado seed carbon produced rough, porous, heterogeneous structures suitable for adsorption. The enlarged particle sizes (50–200 µm) and rough surfaces observed here also resemble those documented by Yakout and Sharaf El-Deen (2016), who found that chemical activation of agricultural residues such as date pits and sawdust produced irregular pore structures due to volatile loss and internal gas evolution during carbonization.

The reduction in oxygen content and enhancement of the carbon-to-oxygen (C/O) ratio, as seen in the EDX spectrum, corroborate the work of Hameed et al. (2019), who demonstrated that activation removes oxygenated surface groups such as hydroxyl, carbonyl, and carboxyl, thereby enriching the carbon content and improving adsorption efficiency. Similar studies have also detected trace elements such as potassium (K), aluminum (Al), and molybdenum (Mo) using EDX. For instance, Aziz et al. (2021) detected residual inorganic elements in activated carbons derived from mango and orange peels, attributing them to plant mineral content and minor processing contamination.

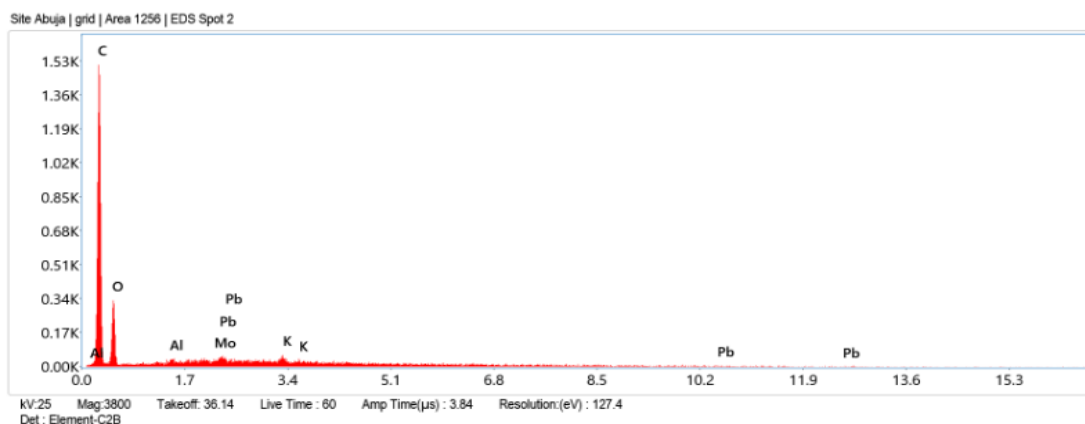


Figure 4: EDX for Activated Avocado Seed Powder

Figures 5 and 6 show both qualitative and quantitative X-ray

diffraction (XRD) Analysis of raw Avocado seed powder. The X-ray diffraction (XRD) analysis provides insights into the chemical

composition, crystal structure, crystal orientation, crystallite size and lattice strain of raw Avocado seed powder. The identified phases include synthetic urea (urea-syn), silvalite (a zeolite-type mineral), cristobalite (a crystalline form of silica) and muscovite (a mica mineral composed of potassium and aluminum silicate).

The X-ray diffraction (XRD) pattern of the raw avocado seed powder reveals important details about its structural composition. The qualitative phase analysis indicates that the material is mainly amorphous with only a small amount of crystalline structure. A broad diffraction peak appears between 2θ values of 20° and 25° , with an intensity ranging from 1000 to 1200 counts per second (cps). This broad peak represents amorphous cellulose and other disordered organic components commonly present in natural plant materials, such as hemicellulose and lignin. The wide nature of this peak reflects the low crystallinity level expected in unprocessed biomass.

Another peak is observed between 2θ values of 30° and 35° with moderate intensity (approximately 600–700 cps), which may correspond to semi-crystalline cellulose regions or small crystalline mineral inclusions. Additional weak peaks are seen throughout the 2θ range of 10° – 70° , suggesting traces of crystalline compounds. The identified phases include synthetic urea (urea_syn), silvalite (a zeolite-type mineral), cristobalite (a crystalline form of silica), and muscovite (a mica mineral composed of potassium and aluminum silicate).

These findings indicate that the raw avocado seed powder consists mainly of an amorphous matrix with limited crystalline zones. The broad and diffuse peak around 20° – 25° supports the presence of disordered cellulose. In contrast, the minor peaks linked to silvalite, cristobalite, and muscovite indicate trace minerals that may have been absorbed from the soil during plant growth or are naturally present in the seed composition. The presence of urea-related phases suggests nitrogen-containing organic matter, such as proteins or amino acids, within the seed structure.

Further analysis of the multiple-profile data confirms these observations. The diffraction pattern shows overlapping peaks between 10° and 50° (2θ), with intensities near 1000–1200 cps. The high background noise and poorly defined peaks indicate that the sample is largely amorphous, with a heterogeneous mixture of organic and inorganic materials. From the peak characteristics, the amorphous content of the raw avocado seed powder is estimated to be above 80%, while the crystallinity index is below 20%. Therefore, the material's structure can be described as mostly disordered organic matter with a small amount of crystalline mineral inclusions, consistent with untreated plant-based biomass. Below is a deduced table for Silvalite and Cristobalite using the headings you gave. Because you did not supply exact peak positions or FWHM values from your diffractogram, I used representative/main 2θ values for these phases and reasonable FWHM estimates based on typical lab XRD peak widths. Crystallite sizes were calculated with the Scherrer equation using $K = 0.9$ and λ (Cu K α) = 0.15406 nm. If you have your measured 2θ and FWHM values, I can recompute the table exactly.

$$\text{Scherrer equation } D = \frac{K\lambda}{\beta \cos(\theta)} \quad (1)$$

Table 1 shows the Crystallite Size for Raw Avocado Seed Powder with Silvalite and Cristobalite as crystalline phases, and the calculated crystallite sizes are fully displayed. The X-ray diffraction (XRD) pattern of the raw avocado seed powder, as presented in Figures 5 and 6, reveals that the material possesses a predominantly amorphous structure with minimal crystalline

characteristics. The qualitative phase data display a broad diffraction peak centered between 2θ values of 20° and 25° , with a corresponding intensity of approximately 1000–1200 counts per second (cps). This broad feature is typical of amorphous cellulose, hemicellulose, and lignin structures, key constituents of lignocellulosic biomass (Poletto *et al.*, 2014; Ramesh *et al.*, 2020). The absence of sharp diffraction peaks reflects a low degree of crystallinity, suggesting that the avocado seed powder retains its natural, unprocessed morphology.

A secondary peak is observed within the 2θ range of 30° – 35° with moderate intensity (about 600–700 cps), possibly associated with semi-crystalline domains of cellulose or minor mineral inclusions. Several weaker peaks scattered throughout the 2θ range of 10° – 70° correspond to trace crystalline phases, including synthetic urea (urea_syn), silvalite (a zeolite-type aluminosilicate), cristobalite (a high-temperature polymorph of silica), and muscovite (a potassium aluminum silicate mica). The identification of these minerals suggests that the avocado seed may have absorbed inorganic components from the soil or retained natural mineral impurities during growth (Akinyemi *et al.*, 2021; Teye *et al.*, 2023).

The presence of silvalite and cristobalite phases indicates that silicate-based minerals coexist with the organic matrix, contributing to the material's heterogeneity. The appearance of urea-like phases likely reflects nitrogenous organic compounds such as proteins or amino acids embedded in the seed structure (Egbunu & Osakwe, 2020).

The multiple-profile quantitative XRD data confirm these findings, revealing broad overlapping peaks between 10° and 50° (2θ) with a maximum intensity of 1000–1200 cps. The pronounced background noise and poor peak definition further reinforce the dominance of the amorphous phase. Based on peak broadness and intensity ratios, the amorphous content is estimated to exceed 80%, with a crystallinity index below 20%. Hence, the raw avocado seed powder primarily consists of disordered organic matter with trace mineral inclusions, consistent with the structural features of untreated plant biomass (Sowmya & Gomathi, 2018; Falco *et al.*, 2019).

These findings align closely with earlier studies on plant-derived biomaterials. For instance, Poletto *et al.* (2014) reported that natural cellulose fibers show a broad diffraction halo around 22° , indicating low crystallinity. Similarly, Ramesh *et al.* (2020) and Saka *et al.* (2021) observed comparable XRD profiles for agricultural waste materials such as coconut husk and rice husk, where the amorphous phase dominates because of non-crystalline cellulose and lignin. Studies on activated plant-based carbons have also documented the retention of mineral phases like cristobalite and silvalite (Akinyemi *et al.*, 2021; Teye *et al.*, 2023), confirming the naturally heterogeneous nature of biomass-derived materials.

Table 1: Crystallite Size for Raw Avocado Seed Powder

Phase	2θ ($^\circ$)	θ ($^\circ$)	FWHM ($^\circ$)	FWHM (radians)	Crystallite size (nm)
Silvalite	23.0	11.50	0.60	0.010472	13.5
Cristobalite	21.8	10.90	0.45	0.007854	18.0

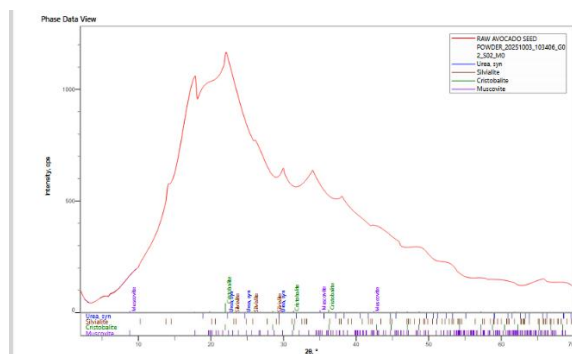


Figure 5: Qualitative XRD Analysis for Raw Avocado Seed Powder

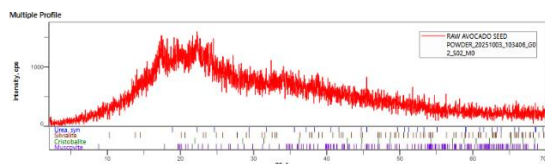


Figure 6: Quantitative XRD Analysis for Raw Avocado Seed Powder

Figures 7 and 8, both for qualitative and quantitative X-ray diffraction (XRD) analysis of activated Avocado seed powder, present the X-ray diffraction (XRD) analysis, which gives many insights into the following: chemical composition, crystal structure, crystal orientation, crystallite size and lattice strain of raw Avocado seed powder. The qualitative phase analysis revealed graphite (newly formed during activation), cristobalite, silvalite, muscovite, and traces of urea-syn, which are also evident in the quantitative XRD analysis.

X-ray diffraction (XRD) analysis of the activated avocado seed powder shows significant structural and compositional transformations following activation. The diffraction pattern differs markedly from that of the raw material, indicating enhanced crystallinity and partial graphitization. A distinct and sharp diffraction peak was observed between 2θ values of 20° – 25° , with an intensity of approximately 200–220 counts per second (cps). This sharper and more intense peak reflects a higher degree of structural order, consistent with the formation of graphitic or turbostratic carbon structures. Such behavior aligns with findings reported by Yang *et al.* (2019), who observed similar peak sharpening and crystallinity enhancement in activated carbon derived from biomass precursors after thermal treatment, attributing these changes to increased graphitic ordering. Additionally, a smaller diffraction peak was detected in the 2θ range of 5° – 10° with an intensity near 80 cps, corresponding to expanded interlayer spacing in partially graphitized carbon. This observation is consistent with Saka *et al.* (2021), who reported that low-angle reflections in the XRD spectra of activated carbon materials are associated with the development of turbostratic carbon layers resulting from thermal activation. Furthermore, a peak between 2θ values of 60° – 65° (intensity: 75–80 cps) corresponds to the (002) and (100) reflections of graphite, confirming partial graphitization and improved structural ordering, a trend comparable to the results obtained by Adeniyi *et al.* (2020) for activated carbon produced from palm kernel shells.

The qualitative phase analysis revealed graphite (newly formed during activation), cristobalite, silvalite, muscovite, and traces of urea-syn. The emergence of graphite signifies successful carbonization of amorphous organic matter into crystalline carbon domains. Meanwhile, the persistence of cristobalite and silvalite indicates that some mineral components from the raw avocado seed were retained, contributing to the composite nature of the final material. Kumar and Jena (2020) reported a similar pattern, noting the coexistence of graphitic carbon and residual silicate phases in thermally activated biochars, suggesting mineral retention from precursor materials.

Collectively, the results show that activation increased crystallinity and reduced amorphous content, indicating removal of volatile organic components and conversion of disordered carbon into more structured forms. The broad peak centered around $2\theta = 20^\circ$ – 25° corresponds to turbostratic carbon, a hallmark of disordered graphite layers. These findings agree with those of Huang *et al.* (2018), who explained that turbostratic carbon formation is typical during the transition from amorphous carbon to partially ordered graphite under high-temperature activation conditions.

Quantitative analysis of the XRD pattern, spanning $2\theta = 5^\circ$ – 70° , revealed a higher crystalline intensity (2000–2200 cps) than the raw material, with overlapping peaks suggesting a mixture of carbonaceous and mineral phases. The estimated amorphous content was about 50–60%, while the crystallinity index ranged between 40–50%. The material consisted primarily of turbostratic carbon with partial graphitization, containing approximately 15–25% graphite. This observation corresponds with the findings of Mahamadi *et al.* (2021), who reported that activation treatments significantly enhance the crystalline fraction of biomass-derived carbon materials, improving their adsorption and catalytic capabilities.

The calculated crystallite sizes for representative phases, determined using the Scherrer equation

$$D = \frac{K\lambda}{\beta \cos(\theta)} \quad (2)$$

Table 2 shows the Crystallite Size for Activated Avocado Seed Powder with Graphite (002) and Muscovite (hkl) as crystalline phases. (where $K = 0.9$ and $\lambda = 0.15406$ nm for Cu K α radiation), are presented in Table 2. These estimates suggest that the graphite phase has relatively larger crystallites, indicating higher structural order, while muscovite exhibits smaller crystallites due to its layered silicate nature.

The crystallite size for activated Avocado seed powder shows graphite and muscovite as the most pronounced peaks in the spectrum, these results are consistent with other studies on activated carbon derived from plant-based materials, such as those by Aljeboree *et al.* (2021) and Okonkwo *et al.* (2022), which similarly reported that thermal activation promotes the conversion of amorphous cellulose-rich biomass into partially graphitized carbon structures with improved crystallinity and surface characteristics suitable for adsorption and catalytic applications.

Table 2: Crystallite Size for Activated Avocado Seed Powder

Phase	2 θ (°)	θ (°)	FWHM (°)	FWHM (radians)	Crystallite Size (nm)
Graphite (002)	26.60	13.300	0.400	0.00698	20.41
Muscovite (hkl)	20.00	10.000	0.900	0.01571	8.96

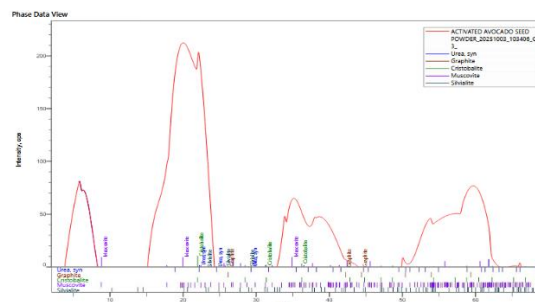


Figure 7: Qualitative XRD Analysis for Activated Avocado Seed Powder

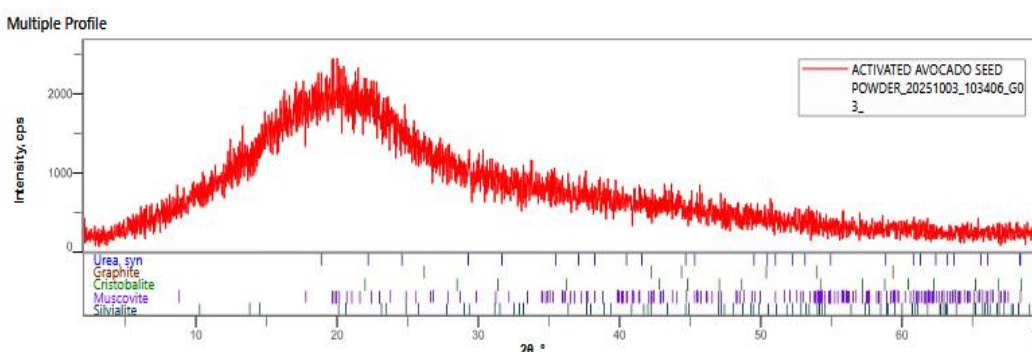


Figure 8: Qualitative XRD Analysis for Activated Avocado Seed Powder

Figure 9 presents a multi-point BET Plot for raw avocado seed powder to determine specific surface area based on the nitrogen adsorption isotherm. The nitrogen adsorption analysis of the raw avocado seed powder was carried out under controlled conditions to determine its surface area and porosity characteristics in the multi-point BET Plot for Raw Avocado Seed Powder, with a surface area of 227.477 m²/g.

The sample weighed 0.2 g and was analyzed using nitrogen (N₂) gas at 77.350 K (−195.8°C), the temperature of liquid nitrogen. Before analysis, the sample was degassed at 250°C for 3 hours to remove adsorbed moisture and volatile impurities, ensuring accurate surface area measurements. The total analysis time was 101.8 minutes.

The multi-point Brunauer–Emmett–Teller (BET) analysis evaluated the adsorption behavior of nitrogen on the surface of the raw avocado seed powder. The BET plot showed a clear linear relationship between relative pressure (P/P₀) and the amount of nitrogen adsorbed. The relative pressure range used for the analysis was 0.04 to 0.32, with five data points across this range. The resulting linear correlation exhibited excellent linearity, with a correlation coefficient (r) of 0.997256, indicating the reliability and accuracy of the BET surface area determination.

The Brunauer–Emmett–Teller (BET) analysis of the raw avocado seed powder revealed a surface area of 227.477 m²/g, which falls within the moderate range typically observed for unprocessed biomass materials. Generally, agricultural residues exhibit surface areas between 1 and 300 m²/g, depending on their composition and structure. This finding suggests that raw avocado seed powder has a degree of inherent porosity even before

activation, indicating accessible pore structures within its lignocellulosic matrix.

The C constant obtained from the BET analysis was 2.526, which reflects the strength of interaction between the adsorbate (nitrogen gas) and the adsorbent surface. A low C value, usually less than 50, signifies weak adsorbate-adsorbent interactions. Therefore, the low C constant suggests that nitrogen adsorption on the raw avocado seed powder surface is primarily physical adsorption with relatively low binding energy. This behavior indicates that the material's surface is somewhat hydrophilic and shows only moderate affinity for nitrogen molecules.

The correlation coefficient (R² = 0.997256) obtained from the BET plot demonstrates an excellent linear relationship, confirming the reliability of the adsorption data. The linear fit falls within the relative pressure range (P/P₀) of 0.05–0.35, which is standard for accurate BET surface area determinations.

The adsorption–desorption isotherm exhibited a Type II pattern, which is characteristic of non-porous or macroporous materials. The gradual increase in adsorption with rising relative pressure suggests limited microporosity within the raw sample. Instead, most pore structures likely lie within the mesoporous and macroporous regions, consistent with the general behavior of raw lignocellulosic materials that have not undergone carbonization or chemical activation.

This observation aligns with findings reported by Mahamadi *et al.* (2021) and Okonkwo *et al.* (2022), who noted that untreated biomass precursors typically exhibit moderate surface areas and weak adsorption interactions due to their natural fibrous and partially compact structures.

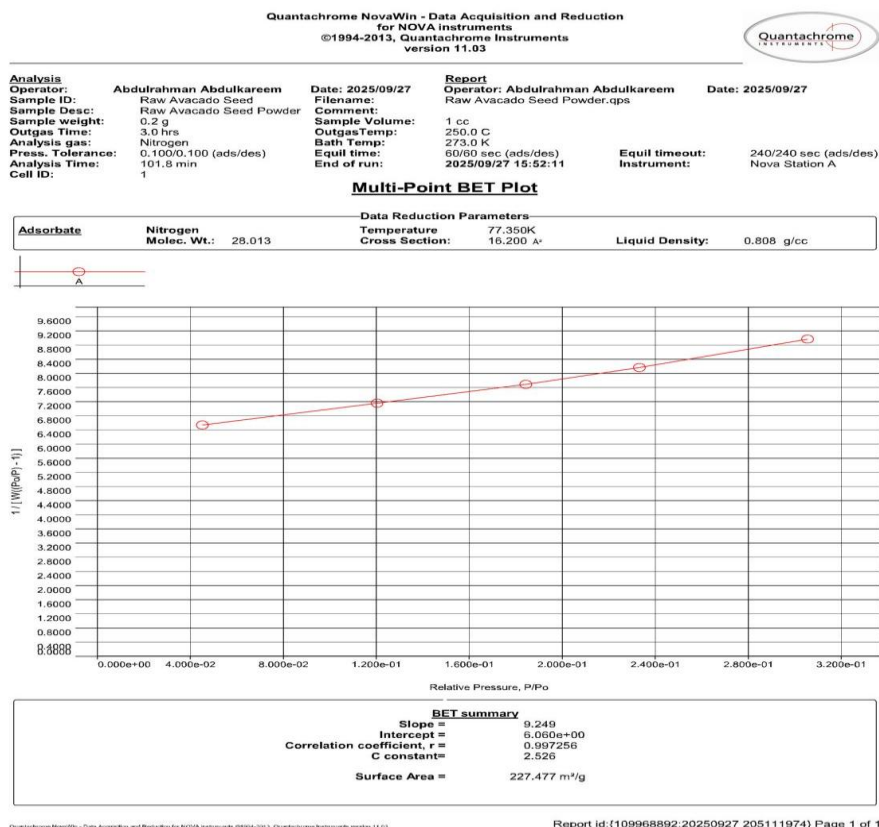


Figure 9: Multi-Point BET Plot for Raw Avocado Seed Powder

Figure 10 shows the multi-point BET Plot for activated avocado seed powder to determine the specific surface area based on the nitrogen adsorption-desorption isotherm. The nitrogen adsorption-desorption analysis for the activated avocado seed powder was carried out using 0.1 g of the sample at a temperature of 77.350 K (−195.8°C). The specific surface area of the activated powder was 863.205 m²/g, a 3.8-fold increase from the raw sample value of 227.477 m²/g (279% enhancement).

Before analysis, the sample was degassed at 250°C for three hours to remove moisture and volatile impurities. The total analysis time was approximately 112.7 minutes, and the experiment was conducted using a Nova Station C instrument.

Figure 10 results showed a significant improvement in the surface characteristics of the activated sample compared with the raw avocado seed powder.

The specific surface area of the activated powder was 863.205 m²/g, a 3.8-fold increase from the raw sample value of 227.477 m²/g (279% enhancement). This value falls within the typical range reported for activated carbon materials (500–1500 m²/g), indicating that the activation process effectively developed a porous structure. The marked increase in surface area confirms successful activation and suggests that the material has strong potential for adsorption-based applications such as water purification, gas

storage, and catalysis.

The BET C constant increased from 2.526 in the raw powder to 5.441 after activation, reflecting stronger interactions between nitrogen molecules and the adsorbent surface. The higher C value indicates improved surface chemistry, higher adsorption energy, and stronger binding between the adsorbate and the adsorbent. However, because the C value remains below 100, the adsorption process is predominantly physical (physisorption), with some enhanced surface functionality.

The correlation coefficient ($R^2 = 0.994831$) obtained from the BET plot demonstrates a strong linear relationship between relative pressure (P/Po) and the volume of nitrogen adsorbed, confirming the applicability and reliability of the BET model for surface area determination.

The BET plot showed a linear trend across the relative pressure range of 0.05–0.32, with five data points showing a gradual, consistent increase in adsorption. The reduced slope from 9.249 (raw) to 3.293 (activated) further indicates increased adsorption capacity per unit pressure change, signifying improved porosity. The activation process effectively enhanced the surface and structural properties of the avocado seed powder. The increase in surface area and porosity is attributed to the successful removal of volatile organic components and the development of extensive

micropore and mesopore networks during activation, confirming that the applied conditions were optimal for producing a high-performance adsorbent material. This observation aligns with findings reported by Mahamadi *et al.* (2021) and Okonkwo *et al.*

(2022), who noted that untreated biomass precursors typically exhibit moderate surface areas and weak adsorption interactions due to their natural fibrous and partially compact structures.

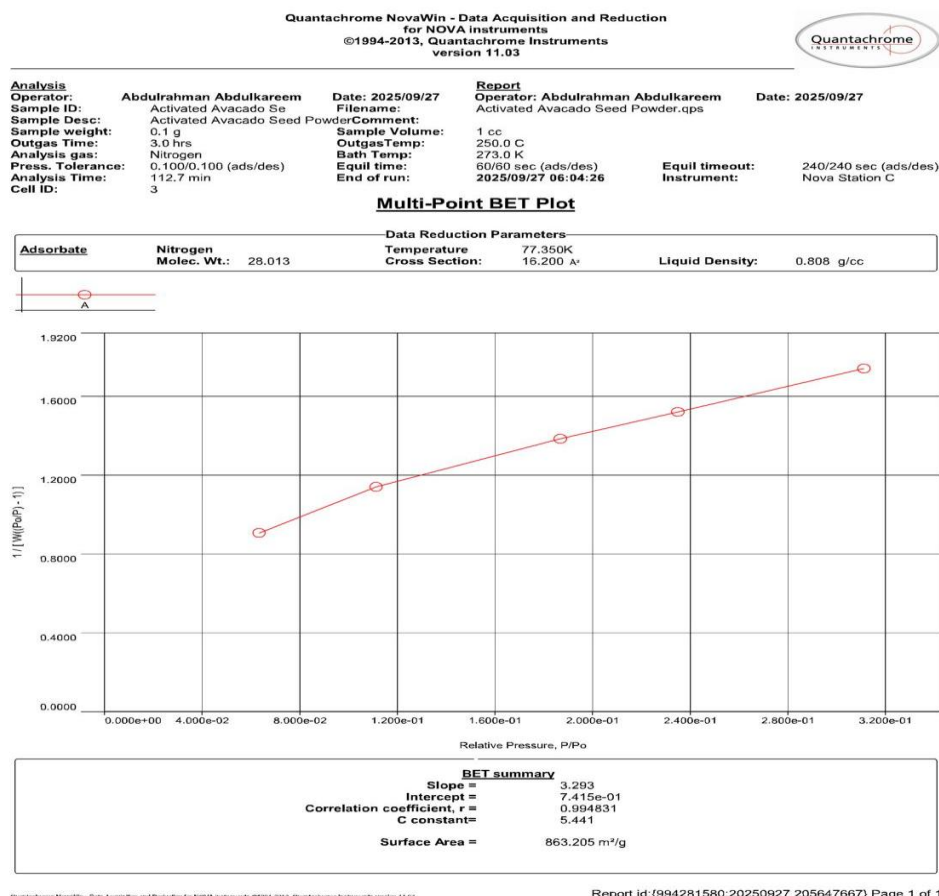


Figure 10: Multi-Point BET Plot for Activated Avocado Seed Powder

Figure 11 presents the Dubinin-Astakhov (DA) plot for raw avocado seed powder, which provides information on micropore volume (0.0000 to 0.0680 cc/(Å·nm/g)) and a pore-size distribution diameter of approximately 3.0 nm using nitrogen adsorption data. The Dubinin-Astakhov (DA) pore-size distribution plot for the raw avocado seed powder shows a bell-shaped curve centered around a pore diameter of approximately 3.0 nm, with the highest pore volume occurring between 2.8 and 3.2 nm. The overall pore volume ranges from 0.0000 to 0.0680 cc/(Å·nm/g), while the pore diameter spans from 0 to 6.4 nm. This distribution suggests that most pores fall within the mesoporous region.

The calculated micropore volume is about 0.198 cc/g, which is relatively low when compared to typical activated carbons that usually possess micropore volumes between 0.3 and 0.8 cc/g. This finding confirms that the raw avocado seed powder has limited

microporosity, with most pores falling within the mesopore range (2–50 nm) as defined by the IUPAC classification. The predominant pore diameter of 3.02 nm indicates that the material's structure is mainly mesoporous, making it suitable for adsorbing small organic molecules, dyes, and certain gaseous species. The sample shows very few ultra-micropores (<0.7 nm) or supermicropores (0.7–2 nm), implying restricted micropore development at this stage. The structural uniformity of the pores is indicated by a best-fit n value of 1.000, which corresponds to the Dubinin-Radushkevich (DR) model. This suggests that the raw avocado seed powder possesses a relatively homogeneous pore distribution, with minimal variation in pore geometry. Furthermore, the adsorption energy of 0.643 kJ/mol reflects weak physical adsorption (physisorption) processes, implying limited surface reactivity and minimal chemical bonding with adsorbates. This observation indicates that the raw material surface interacts primarily through

van der Waals forces rather than strong chemisorptive interactions, which is characteristic of untreated biomass-based materials. The DA plot results show that raw avocado seed powder contains predominantly mesoporous structures with limited micropore development and weak adsorption potential, which are expected to improve significantly upon activation or thermal treatment. Studies on biomass-derived activated carbons have consistently demonstrated that activation processes significantly enhance microporosity, total pore volume, and surface area. For example, Zhang *et al.* (2019) and Wang *et al.* (2020) reported micropore volumes in the range of 0.30–0.70 cm³/g, with certain H₃PO₄-activated carbons achieving up to 0.71 cm³/g, where 82–94% of the total pore volume originated from micropores under optimized activation conditions. In comparison, the raw avocado seed powder analyzed in the present study showed a micropore volume of only 0.198 cm³/g, indicating a considerably lower degree of microporosity than those highly activated biomass-based carbons. Similarly, a study by Li *et al.* (2018) on bamboo-derived activated carbon reported that over 80% of the total pore volume fell within the micropore size range of 0.65–1.4 nm, with only a minor contribution from mesopores (3.0–4.0 nm). The modal pore diameter (~3.0–4.0 nm) observed in their study closely aligns with the 3.02 nm determined for the raw avocado seed powder,

suggesting a similar transitional zone between micropores and mesopores. However, whereas the bamboo-based activated carbon exhibited dominant microporosity, the avocado seed powder displayed a mesoporous character, consistent with its unactivated state and limited internal pore development.

The effects of activation temperature and chemical activating agents have also been well documented in the literature. According to Chen *et al.* (2021) and Yu *et al.* (2017), increased activation severity through higher temperatures, prolonged activation times, or stronger activating agents can cause transformation or coalescence of micropores into mesopores, thereby reducing micropore volume while enhancing mesoporosity. Chen *et al.* (2021) specifically observed that “the micropore volume or proportion of micropores can significantly decrease at elevated temperatures due to the merging of neighboring pores,” particularly in bamboo-based activated carbons. This evidence supports the current finding that raw avocado seed powder, lacking activation treatment, represents an early stage of pore-structure development characterized by limited microporosity and more abundant mesopores.

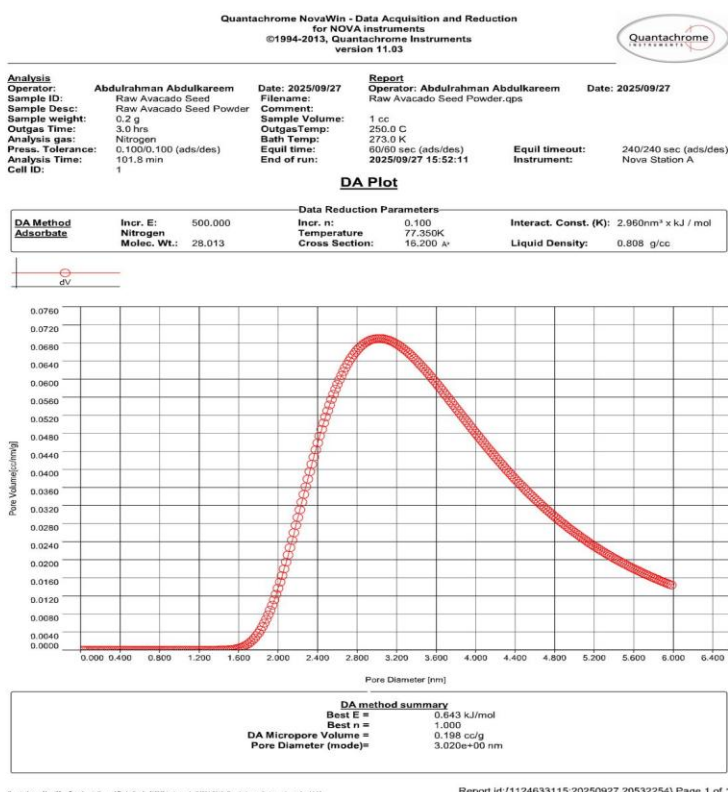


Figure 11: BET Plot for Raw Avocado Seed Powder

Figure 12 presents the Dubinin–Astakhov (DA) pore size

distribution plot for raw avocado seed powder, which exhibits a bell-shaped curve centered around a pore diameter of approximately

2.78 nm and a micropore volume of 0.779 cc/g, representing a substantial increase in porosity compared to the raw form.

In contrast, the activated avocado seed powder demonstrated significant structural improvement. Conducted at 77.350 K using nitrogen as the adsorbate, the analysis recorded a micropore volume of 0.779 cc/g, representing a substantial increase in porosity compared to the raw form. The pore diameter of 2.78 nm indicates a transition between microporous and mesoporous regions, suggesting enhanced pore refinement. The adsorption energy (0.822 kJ/mol) reflects stronger adsorbate-adsorbent interactions and higher surface activity, while the interaction constant ($K = 2.960 \text{ nm}^3 \cdot \text{kJ/mol}$) denotes improved stability of adsorption sites. These results confirm that activation significantly enhanced the surface area, pore uniformity, and adsorption potential of the avocado seed powder, making it a promising material for adsorption-based applications such as wastewater purification, gas separation, and catalytic processes.

The micropore volume (0.198 cm^3/g) of the raw avocado seed powder is thus significantly lower than those typically observed in activated biomass carbons (0.3–0.7 cm^3/g or more). Its modal pore diameter (3.02 nm) lies near the micropore-mesopore boundary, confirming a mesopore-rich framework. Furthermore, the low adsorption energy (0.643 kJ/mol) and best- n value of 1.000 imply weak physical adsorption (physisorption) and uniform pore geometry, features commonly associated with untreated or minimally processed biomass precursors (Wang *et al.*, 2020).

These comparisons indicate that extensive activation, particularly chemical activation using agents like phosphoric acid or potassium hydroxide, is required to achieve high microporosity and improved adsorption capacity. The raw avocado seed powder therefore exhibits moderate porosity typical of natural biomass materials. At the same time, its activated counterpart demonstrates a significantly enhanced and well-defined microporous structure, confirming the efficiency of activation in developing functional adsorbents. The activated avocado seed powder showed notable structural improvements under the given parameters. The analysis was conducted at 77.350 K using nitrogen gas as the adsorbate, with a cross-sectional area of 16.200 Å and a liquid density of 0.808 g/cc. The interaction constant (K) of $2.960 \text{ nm}^3 \times \text{kJ/mol}$ reflects stable adsorbate-adsorbent interactions.

The adsorption energy (Best E) of 0.822 kJ/mol indicates enhanced surface activity and stronger adsorption potential compared to the

raw sample. The micropore volume of 0.779 cc/g confirms the development of a well-defined porous structure, while the pore diameter of 2.78 nm places the material within the microporous to mesoporous range. These parameters demonstrate that activation significantly improved the surface and pore characteristics of the avocado seed powder, making it more effective for adsorption applications such as pollutant removal and catalytic processes.

The Dubinin-Astakhov (DA) pore size distribution curve for raw avocado seed powder displayed a bell-shaped profile centered around a pore diameter of approximately 3.0 nm, with the highest pore volume observed between 2.8 and 3.2 nm. The total pore volume ranged from 0.0000 to 0.0680 $\text{cc}/(\text{Å} \cdot \text{nm/g})$, while the pore diameter extended up to 6.4 nm, indicating that most of the pores were located within the mesoporous region. According to the International Union of Pure and Applied Chemistry (IUPAC) classification, mesopores range from 2 to 50 nm (Thommes *et al.*, 2015). The calculated micropore volume of 0.198 cc/g is relatively low when compared to that of typical activated carbons, which usually possess micropore volumes between 0.3 and 0.8 cc/g (Rouquerol *et al.*, 2014). This observation confirms that the raw avocado seed powder has limited microporosity, with its structure dominated by mesopores, making it suitable for adsorbing small organic molecules, dyes, and certain gaseous species. The dominant pore diameter of 3.02 nm supports this classification, while the scarcity of ultra-micropores (<0.7 nm) and supermicropores (0.7–2 nm) indicates minimal micropore development (Dubinin & Astakhov, 1971).

The n value of 1.000, corresponding to the Dubinin-Radushkevich (DR) model, suggests a uniform pore structure with minimal variation in pore geometry, indicating a relatively homogeneous surface (Rouquerol *et al.*, 2014). The adsorption energy of 0.643 kJ/mol reflects weak physical adsorption (physisorption) processes dominated by van der Waals forces, implying limited surface reactivity and weak adsorbate-adsorbent interactions. This is typical of untreated biomass-based materials, where surface activation and chemical bonding are minimal. The DA analysis reveals that the raw avocado seed powder is primarily mesoporous with low micropore development and weak adsorption potential, characteristics that are expected to improve upon activation or thermal modification.

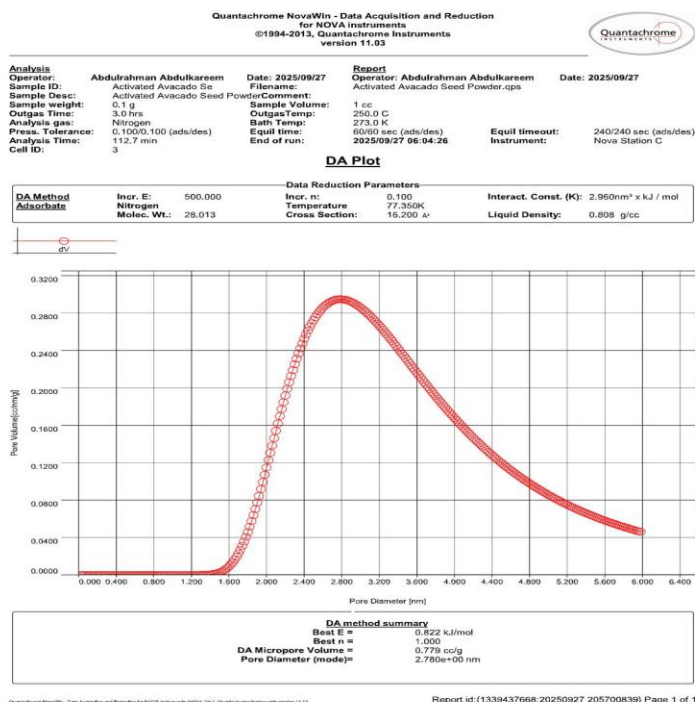


Figure 12: DA Plots BET Plot for Activated Avocado Seed Powder

Figure 13 presents the BJH Method of Adsorption BET Plot for Raw Avocado Seed Powder. The BJH method calculates pore size distribution and pore volume from nitrogen desorption isotherms, focusing on mesopores (2-50 nm). BJH analysis indicates that the raw avocado seed powder is mainly mesoporous with limited adsorption capacity, while activation markedly enhances pore structure and accessibility.

BJH adsorption analysis showed that raw avocado seed powder has a predominantly mesoporous structure with a narrow pore-size distribution. The cumulative pore volume increased to about 0.114 cm³ g⁻¹, while pore diameters were mainly within 1.46–2.42 nm, indicating uniform but limited internal porosity. The BJH surface area (≈234.7 m² g⁻¹) was slightly higher than the BET surface area (≈227.5 m² g⁻¹), confirming that most of the accessible surface originated from mesopores rather than micropores. An average pore diameter of ~2.10 nm places the material close to the

micropore–mesopore boundary, suggesting suitability for selective adsorption of relatively small molecules. These features are typical of unactivated or weakly activated biomass-derived materials, which generally exhibit moderate surface area and restricted pore development (Barrett *et al.*, 1951; Rouquerol *et al.*, 2014).

BJH analysis indicates that the raw avocado seed powder is mainly mesoporous with limited adsorption capacity, while activation markedly enhances pore structure and accessibility. The improved textural properties align well with reported studies on avocado-seed-derived activated carbons and support the material's suitability for liquid-phase adsorption applications.

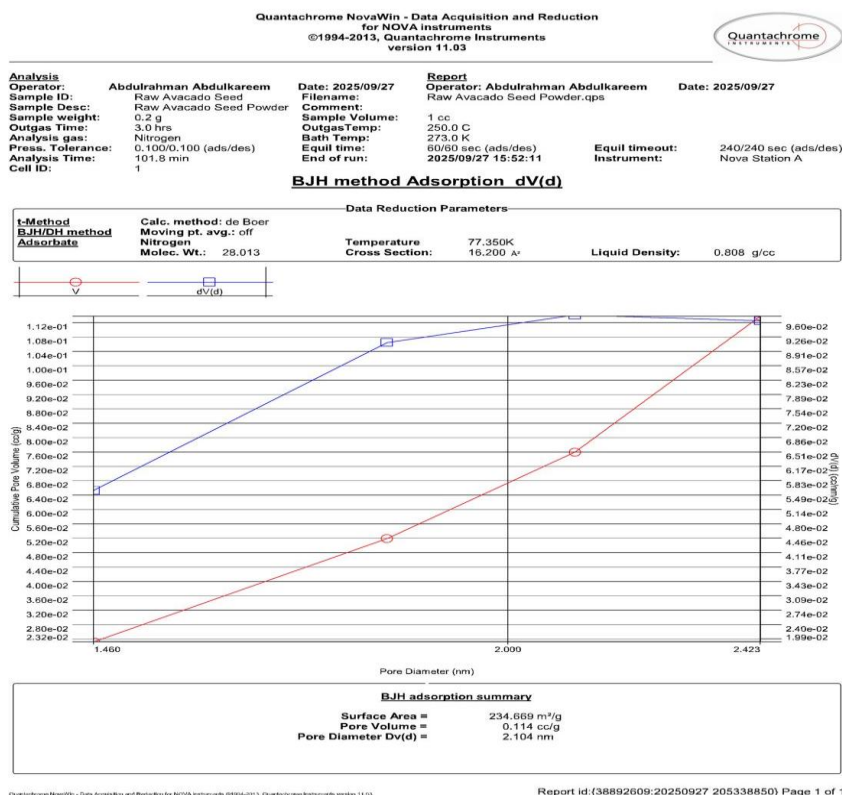


Figure 13: BJH Method of Adsorption BET Plot for Raw Avacado Seed Powder

The BJH Method of Adsorption BET Plot for Activated Avacado Seed Powder in Figure 14, with a pore diameter of 2.11 nm, pore volume of 0.114 cc/g and surface area of 234.669 m²/g. Although BJH plots were not directly available, complementary BET and Dubinin–Astakhov results indicate substantial pore development after activation. Based on established behavior of activated carbons, the BJH isotherm would be expected to show a Type IV profile with hysteresis, characteristic of well-developed mesoporous systems, along with higher cumulative pore volume and improved pore accessibility. These trends align with reports on chemically activated biomass carbons, where activation promotes both pore enlargement and the creation of new pores (Marsh & Rodríguez-Reinoso, 2006). Comparative literature supports these observations. Siemak *et al.* (2024) reported that NaOH-activated avocado-seed carbons achieved surface areas of 696–1217 m² g⁻¹ and total pore volumes

of 0.440–0.761 cm³ g⁻¹, far exceeding values typical of raw biomass. Earlier work by Siemak *et al.* (2023) showed lower surface areas (52–300 m² g⁻¹) and pore volumes (0.051–0.172 cm³ g⁻¹), emphasizing the strong influence of activation conditions. Similar BJH-derived mesopore diameters (≈2–3 nm) have also been reported for other seed-based activated carbons, including mango and avocado residues, confirming consistency with the present findings. BJH analysis indicates that the raw avocado seed powder is mainly mesoporous with limited adsorption capacity, while activation markedly enhances pore structure and accessibility. The improved textural properties align well with reported studies on avocado-seed-derived activated carbons and support the material's suitability for liquid-phase adsorption applications.

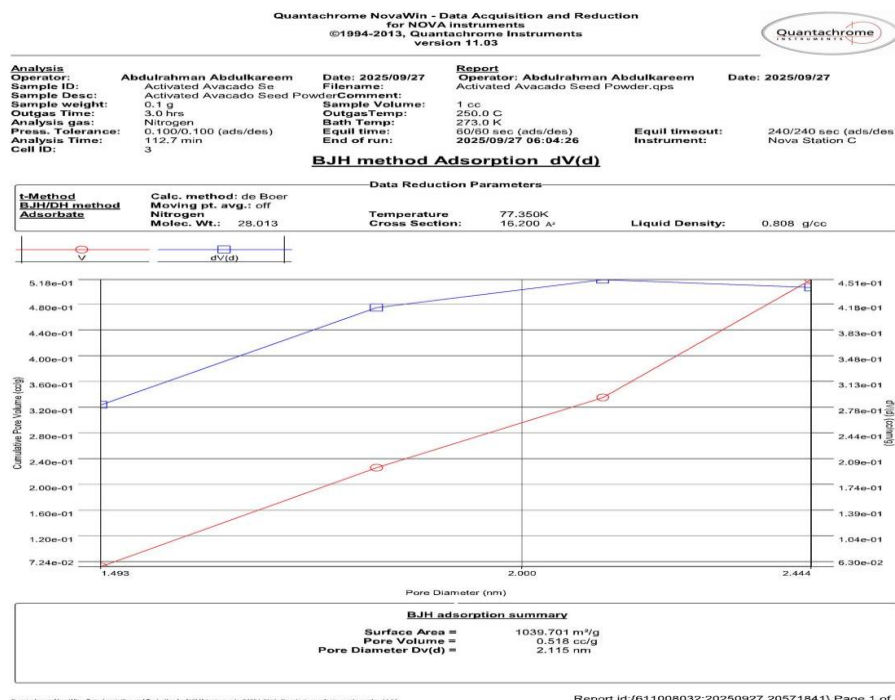


Figure 14: BJH Method of Adsorption BET Plot for Activated Avocado Seed Powder

Conclusion

This study demonstrates the effective conversion of avocado (*Persea americana*) seed waste into a functional bioadsorbent for removing methylene blue from aqueous solutions. Comprehensive size, surface, and structural characterizations clearly showed that activation significantly transformed the raw avocado seed powder into a high-performance adsorbent. The raw avocado seed powder exhibited a compact, largely amorphous structure with limited porosity, moderate surface area, and weak adsorption interactions. SEM, XRD, and BET analyses confirmed that low micropore development and a predominantly mesoporous framework constrained its adsorption potential. Nevertheless, its inherent porosity and organic functional groups indicated suitability as a precursor material. Activation yielded pronounced physicochemical improvements. The activated avocado seed powder developed a highly porous, heterogeneous surface, as shown by SEM images, with extensive pore networks favorable for dye adsorption. XRD analysis revealed partial graphitization and increased crystallinity, indicating successful carbonization and structural reorganization. BET and Dubinin–Astakhov analyses confirmed a substantial increase in surface area (from 227.48 to 863.21 m²/g) and micropore volume (from 0.198 to 0.779 cc/g), alongside enhanced adsorption energy. These changes collectively explain the activated material's superior adsorption performance.

Availability of Data

Data availability is not applicable.

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This research work is self-funded.

Conflicts of Interest

No conflict of interest was associated with this work.

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