

Production and Characterization of Zeolite Y/Coconut Shell Activated Carbon Hybrid Adsorbent and its Suitability for Textile Wastewater Treatment

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ABSTRACT

Textile effluents contain persistent organic dyes and ionic pollutants that are inadequately treated by single-component adsorbents. In this work, hybrid adsorbents composed of Zeolite Y and Coconut Shell-derived Activated Carbon (CSAC) were synthesized at different mass ratios using a wet impregnation technique. Structural and surface properties were characterized using Scanning Electron Microscopy (SEM), X-ray Fluorescence (XRF), X-ray Diffraction (XRD), Fourier Transform Infrared Spectroscopy (FTIR), and Brunauer, Emmett and Teller (BET)-N₂ adsorption analyses, revealing complementary features of the two components. Thus, CSAC exhibited heterogeneous porosity and abundant oxygenated functional groups favourable to hydrophobic dye adsorption, whereas Zeolite Y displayed large crystallite sizes, high crystallinity, and revealed ion-exchange capacity. The hybrids displayed coexisting amorphous carbon domains and crystalline Faujasite phases, alongside oxygenated carbon functionalities and aluminosilicate framework vibrations. Textural properties improved significantly after activation and hybridization, yielding high surface areas ($\approx 512\text{--}580\text{ m}^2\text{ g}^{-1}$) and hierarchical micro-mesoporous structures. Carbon-rich hybrids showed potential affinity towards adsorbing bulky hydrophobic dyes through pore filling and $\pi\text{--}\pi$ interactions, whereas zeolite-rich materials enhanced electrostatic adsorption and ion exchange towards cationic dyes. Incorporation of zeolite into carbon matrices enhanced mechanical stability, rigidity, and selective ion-exchange behaviour, while retaining the broad adsorption capacity of activated carbon. These findings underscore the promise of Zeolite Y/CSAC hybrids as sustainable, high-performance adsorbents for complex textile wastewater remediation.

Keywords: Coconut shell, zeolite Y, activated carbon, hybrid

1.0 INTRODUCTION

The textile industry is recognized as one of the most water-intensive manufacturing sectors, requiring large volumes of freshwater and chemical auxiliaries during dyeing and finishing processes, which consequently leads to the generation of highly polluted wastewater streams (Althuri *et al.*, 2020). Textile effluents are commonly characterized by strong coloration, high chemical oxygen demand (COD) and biological oxygen demand (BOD), turbidity, total dissolved solids (TDS), high salt content, surfactants, and persistent organic substances, which can cause severe environmental degradation and pose risks to human health if discharged without adequate treatment (Al-Tohamy *et al.*, 2022; Periyasamy, 2024). Synthetic dyes, such as azo dyes, are particularly challenging pollutants due to their stable aromatic structures, resistance to biological degradation, and toxic, mutagenic, and carcinogenic properties (Qasim & Ibrahim, 2025). Therefore, the development of effective, environmentally friendly, and economically feasible treatment technologies for dye-contaminated wastewater remains a critical global concern.

Among available treatment methods, activated carbon has been extensively applied as a commercial

adsorbent owing to its large specific surface area, well-developed pore structure, and strong affinity for a broad range of organic contaminants (Tan *et al.*, 2017). Nevertheless, the high production cost of coal-derived activated carbon, coupled with challenges related to regeneration and disposal, has stimulated interest in alternative, low-cost, and renewable carbon sources (Tan *et al.*, 2017). Biomass-derived activated carbons have emerged as sustainable substitutes due to their abundance, renewability, and favourable physicochemical properties. Agricultural by-products such as coconut shell, palm kernel shell, rice husk, corncob, and sawdust have shown strong potential as precursors for producing high-performance carbon adsorbents (Altenor *et al.*, 2009). In particular, coconut shell has attracted considerable attention because of its high lignin content and dense structure, which facilitate the formation of mechanically stable, micro/mesoporous carbon materials with excellent adsorption capacity (Ajie *et al.*, 2023).

Despite these advantages, biomass-based activated carbons exhibit limited selectivity toward ionic and polar contaminants due to surface charge heterogeneity and restricted accessibility of some pore domains (Vu

et al., 2025). To overcome these limitations, the development of hybrid adsorbent systems that integrate carbonaceous materials with inorganic porous structures has gained increasing research interest. Zeolites are attractive inorganic components due to their crystalline aluminosilicate framework, uniform microporous channels, high thermal stability, and exceptional ion-exchange properties (Kordala & Wyszowski, 2024; Shah *et al.*, 2023). The permanent negative charge of zeolite frameworks, resulting from the substitution of Si^{4+} by Al^{3+} in the tetrahedral structure, enables strong electrostatic interactions with positively charged pollutants, thereby enhancing adsorption performance in aqueous systems (Kordala & Wyszowski, 2024). Zeolite Y, a Faujasite-type zeolite, is particularly suitable for adsorption applications due to its large pore apertures, high surface area, and three-dimensional pore network that promotes efficient mass transfer (Ibrahim & Al-Slaty, 2023). However, pristine zeolites, being highly crystalline aluminosilicates with uniform micropores, are excellent adsorbents for small polar molecules. Nevertheless, their hydrophilic nature and narrow pore sizes limit their effectiveness for bulky or hydrophobic organics. This can lead to reduced adsorption capacity and even pore blockage when large molecules attempt to enter the microporous framework (Guo *et al.*, 2021; Zhan *et al.*, 2025). Conversely, activated carbon exhibits strong hydrophobic adsorption and π - π electron donor-acceptor interactions but lacks inherent ion-exchange functionality (Bernal *et al.*, 2022). Combining these two materials into a hybrid composite offers a promising strategy to exploit their complementary adsorption mechanisms while mitigating their individual shortcomings. In addition, hybridization can improve mechanical stability and structural durability, both of which are essential for long-term operation in wastewater treatment systems. Despite increasing interest in hybrid adsorbents, systematic investigations focusing on the integration of Coconut Shell-derived Activated Carbon with Zeolite Y for the treatment of textile wastewater remain limited. Most previous studies have primarily examined biomass-based activated carbon or synthetic zeolites as individual adsorbents, with relatively few reports addressing the structure-property relationships of combined systems. Studies have documented composite adsorbents derived from Coconut Shell Activated Carbon and Zeolite for oilfield wastewater treatment (Oyedoh & Igbokwe, 2025), sugarcane bagasse activated carbon-zeolite composite (Mohamed *et al.*, 2022), and algae-based zeolite composite (Dryaz *et al.*, 2021). However, these studies largely employed fixed mass ratios, typically equal proportions of both components, which restricts understanding of how compositional variation influences properties of the composites, which in turn can affect their adsorption performance. This limitation highlights a clear research gap and provides strong

motivation for the present study, which explores the synthesis and characterization of Zeolite Y and coconut shell activated carbon hybrids at multiple composition ratios to optimize adsorbent performance. Scanning Electron Microscopy (SEM) (morphology); X-ray Fluorescence (XRF) (elemental composition); X-ray Diffraction (XRD) (crystallinity); Fourier Transform Infrared Spectroscopy (FTIR) (functional groups); and Brunauer, Emmett and Teller (BET)- N_2 adsorption (surface area and pore properties); provides a comprehensive understanding of material properties and their treatment potential. The outcome of this work is expected to contribute to the development of sustainable and high-performance hybrid adsorbent materials for textile wastewater treatment and broader environmental remediation applications.

2.0 MATERIALS AND METHODS

2.1 Materials

The materials, chemicals/reagents used in this study are of analytical grade, and the reagents were prepared using the established standard procedures. Phosphoric acid, H_3PO_4 (98% purity) (BDH Chemicals Limited, Poole, England) was used for the activation process. Others include Glassware, Whatman filter papers, distilled water, Electric Oven, muffle furnace, magnetic stirrer, water bath shaker, weighing balance, and funnels, Coconut shell (Central Market, Kaduna), and Faujasite-type Zeolite Y (obtained from KRPC, Kaduna, which was used without further modification).

2.2 Preparation of Activated Carbon from Coconut Shell

The coconut shells used in this study were collected from Bakindogo Central Market, Kaduna State, Nigeria. The shells were thoroughly washed repeatedly with tap water after picking foreign materials, followed by distilled water washing, and then sundried for five days, and thereafter oven-dried overnight. This was followed by crushing using an industrial crusher and grinding machine (Olatunji *et al.*, 2024). The ground coconut shell was sieved with a mesh size of 280 microns to obtain a uniform particle size.

The method described by Onigemo *et al.* (2024) was modified and used to prepare activated carbon from coconut shell powder. The ground coconut shell was placed in a crucible and subsequently carbonized in a muffle furnace at 500 °C for 1 hr. After cooling in a desiccator, the carbonized shell was impregnated with a 1M solution of phosphoric acid in a ratio of 2:1 (volume of acid per gram of precursor) for 24 hr at room temperature to obtain activated carbon (Oyedoh & Igbokwe, 2025). The resultant paste was washed with distilled water to a neutral pH of 7.0, and this was followed by activation at a temperature of 350 °C for 2 hr in a furnace (Olatunji *et al.*, 2024), and later oven-dried overnight at 120 °C. The oven-dried sample was cooled in a desiccator and stored in an air-tight labeled

sample bottle for characterization and blending with Zeolite Y at different proportions.

2.3 Formulation of Zeolite/Coconut Shell Activated Carbon (CSAC) hybrid Adsorbent

The Zeolite/CSAC hybrid adsorbent was prepared by mechanical mixing (Nwafulugo, 2014), followed by a wet impregnation technique similar to that used in the work of Dryaz *et al.* (2021); Mohamed *et al.* (2022). To ensure an even particle-size distribution, particle-size analysis for both the Zeolite and activated carbon was carried out by milling and sieving using a 150 μm aperture sieve. Specific weight ratios as shown in Table 1 were measured, mixed, and properly blended with a domestic blender to ensure homogeneity of the

resulting zeolite and activated carbon hybrid (Nwafulugo, 2014). To ensure the penetration of zeolite into the activated carbon and to achieve effective impregnation with strong bonding between the two materials, the blends were subsequently mixed with distilled water in ratio of 1:10 (mass of blend to distilled water) (Dryaz *et al.*, 2021) and stirred at 500 rpm for 60 min on a magnetic stirrer and then in water bath at 300rpm for 60 min at room temperature. This was repeated for three times, and after that, the resultant Zeolite/CSAC hybrid was filtered and washed with distilled water for several times (to remove unreacted ions) and thereafter, dried in an oven at 60 °C for 24 h.

Table 1: Mixed Ratio of Zeolite Y and Coconut Shell Activated Carbon (CSAC)

Hybrid Ratio (in grams)		Identity
Zeolite Y (%)	CSAC (%)	
0	100	Sample 1
20	80	Sample 2
50	50	Sample 3
80	20	Sample 4
100	0	Sample 5

2.4 Sample Characterizations

The Shimadzu Fourier Transform Infrared Spectrophotometer (FTIR 8400 S) was used for functional group determination using the dry KBr pellet method. The Rigaku MiniFlex 600 XRD Diffractometer with a copper X-ray tube (Cu K α 1 and Cu K α 2) as radiation sources was used to determine the crystallinity of the samples. The instrument settings for each test at a temperature of 21-23 °C were: voltage = 40kV, Current = 30 mA. The adsorbent sample was scanned at a 2 θ interval between 2° and 70°. PhenomWorld Pro X capable SEM of high-resolution imaging working at a high voltage of 15 kV was used for analysing the morphology of prepared adsorbents. Genius-IF Xenometric XRF was used to determine the elemental oxide composition of the adsorbents. To determine the surface area and the pore properties, nitrogen (N₂) physical adsorption-desorption isotherm measurement (NOVA 4200e Quantachrome) was used.

3.0 RESULTS AND DISCUSSION

3.1 SEM Analysis

SEM analysis of the adsorbents as presented in Figure 1, provides critical morphological insights into Coconut Shell Activated Carbon (CSAC), Zeolite Y, and their hybrids, showing how surface texture and porosity can influence adsorption performance. Thus, CSAC micrograph (Figure 1a) reveals a highly heterogeneous and irregular fractured edges, full of various sizes of cavity, and a rough surface which indicates micro- and meso-porosity resulting from

carbonization and activation-features that gives CSAC a large accessible surface area and pore accessibility favorable for non-specific adsorption. The removal of volatiles widens micropores into mesopores, producing interconnected channels suitable for dye diffusion and physisorption. These pores promote π - π interactions with synthetic dyes and increase accessibility to surface sites (Bernal *et al.*, 2022; Yang *et al.*, 2021). While, Zeolite Y micrograph (Figure 1e), by contrast, has a well-defined, crystalline octahedral morphology with uniform, smooth, and brighter particle sizes - characteristic of aluminosilicate frameworks; its pore structure is dominated by micro-porosity, which underlines zeolite's strong ion-exchange and molecular-sieving capabilities (Perez-Botella *et al.*, 2022). This morphology aligns with that observed by (Ibrahim & Al-Slaty, 2023; Moneim & Ahmed, 2015). Unlike CSAC, the zeolite's adsorption relies less on broad porosity and more on framework-driven ion exchange. The hybrids (Figure 1b, 1c and 1d) show transitional morphology that correlates with composition. The zeolite and activated carbon are well combined during synthesis, with zeolite particles covering some of the pores of the activated carbon and forming agglomerated particles. This may likely reduce the pore size of the activated carbon, leading to physical and chemical reactions. This observation is in line with what was reported by Mohamed *et al.* (2022) after synthesizing an Activated Carbon/Zeilite nanocomposite from sugarcane and zeolite for methylene blue adsorption.

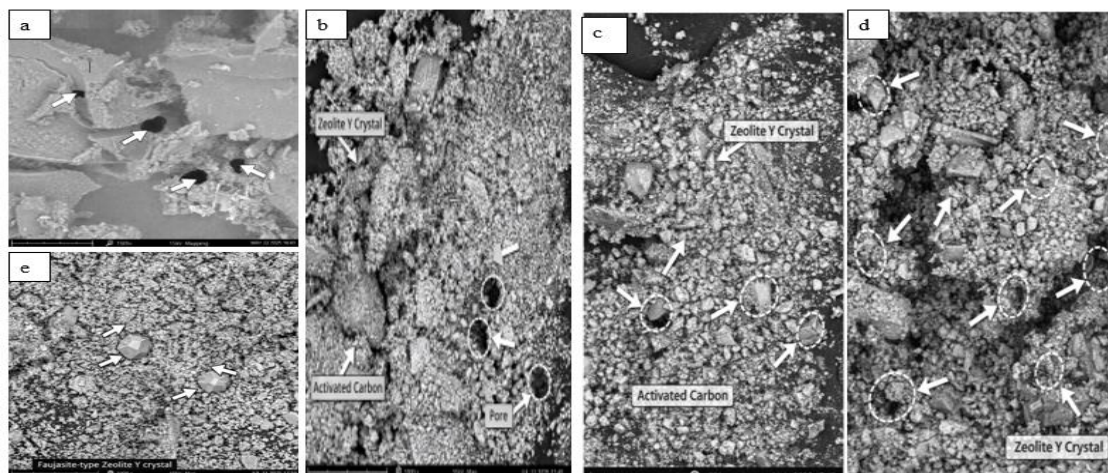


Figure 1: SEM images of (a) CSAC, (b) CSAC-80%/Zeolite-20%, (c) CSAC-50%/Zeolite-50%, (d) CSAC-20%/Zeolite-80%, and (e) Zeolite Y

The CSAC-rich (Figure 1b) hybrid is dominated by the carbon's rough porous matrix with zeolite crystals dispersed throughout, preserving large accessible voids while introducing some ordered domains from zeolite. This structure can enhance dye adsorption capacity with some ionic selectivity without significant pore blockage, as observed by Alawam *et al.* (2025). The 50% CSAC/50% Zeolite hybrid (Figure 1c) exhibits an integrated morphology in which CSAC's porous network and zeolite's crystalline domains coexist and appear well interfaced, producing a more uniform pore distribution. This dual-phase morphology provides synergistic functionality, where carbon pores enable broad adsorption, while zeolite domains contribute ion-exchange capacity. This balanced ratio and morphology may enhance superior dye removal as observed by Alawam *et al.* (2025). Conversely, the zeolite-rich composite (Figure 1d) is largely crystalline with reduced surface roughness. The reduced carbon fraction may diminish the capacity for bulky, non-ionic dyes removal, though stability and framework selectivity may also be improved. Thus, increasing the CSAC content enhances surface heterogeneity and larger pore volumes. This is beneficial for bulk adsorption, where higher zeolite content increases crystalline order and micro-porosity. This is also beneficial for selective ion exchange.

3.2 XRF Analysis

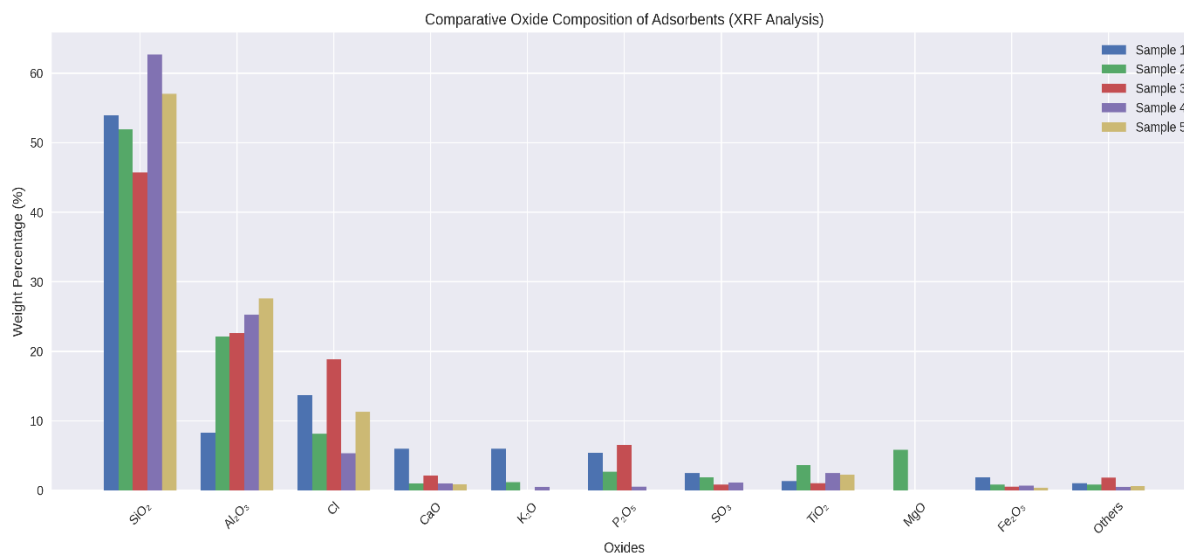
The elemental oxide compositions of the prepared CSAC, hybrid adsorbents, and Zeolite Y were obtained through XRF analysis, and the results are as presented in Table 2 and Figure 2. The chemical composition shows various concentration of metal oxides, which plays an important role in catalysis and adsorption (Ukonkwo *et al.*, 2022). The CSAC contained high levels of SiO₂ (53.954 wt%), CaO (5.974 wt%), and K₂O (5.963 wt%), reflecting the mineral-rich composition typical of biomass. The relatively high

concentration of silica has the potential to enhance structural stability and provide more active sites for dye uptake, consistent with observations by Janekarn *et al.* (2020). The higher ratio of silica to alumina of the CSAC can be attributed to the presence of a lower content of Al_2O_3 and a higher content of SiO_2 in the form of quartz in the sample, which is supported by the XRD pattern in Figure 3 and in line with the result obtained by Nwafulugo (2014). The Faujasite-type Zeolite Y exhibited a composition dominated by SiO_2 (57.032 wt%) and Al_2O_3 (27.605 wt%), corresponding to a $\text{SiO}_2/\text{Al}_2\text{O}_3$ ratio of about 2.1. This is well within the characteristic range reported for Faujasite Zeolite Y in the literature (Kordala & Wyszowski, 2024).

The hybrid samples showed a gradual transition toward the oxide profile of Zeolite Y as the zeolite fraction increases. SiO₂ rose from 51.894 wt% (20% zeolite) to 62.693 wt% (80% zeolite), while Al₂O₃ increased from 22.122 wt% to 25.231 wt%. At the same time, there is a reduction in the amount of CaO, K₂O, and characteristic of biomass-derived carbons due to the compositional dilution effect. The presence of chloride ions can modify the electrical double layer surrounding adsorbent surfaces, thereby influencing surface charge density and weakening electrostatic attraction between adsorbents and charged dye molecules through charge screening and competitive ion effects (Wu *et al.*, 2020). The presence of transition metal oxides such as TiO₂ and Fe₂O₃ can enhance catalytic activity and facilitate advanced oxidation processes by promoting the formation of highly reactive species, such as hydroxyl radicals, thereby improving the degradation efficiency of organic pollutants (Anucha *et al.*, 2022; Ukonkwo *et al.*, 2022). These findings confirm that zeolite-carbon composites combine adsorption, ion exchange, and catalytic functions to enable effective treatment of textile wastewater.

Table 2. Elemental Oxides Composition of Prepared Adsorbents from XRF

Oxide (wt %)	Sample 1	Sample 2	Sample 3	Sample 4	Sample 5
SiO ₂	53.954	51.894	45.696	62.693	57.032
Al ₂ O ₃	8.308	22.122	22.620	25.231	27.605
Cl	13.703	8.117	18.85	5.334	11.280
CaO	5.974	1.017	2.076	0.966	0.844
K ₂ O	5.963	1.176	ND	0.48	ND
P ₂ O ₅	5.39	2.692	6.532	0.543	ND
SO ₃	2.438	1.903	0.796	1.134	ND
TiO ₂	1.296	3.598	1.058	2.475	2.261
MgO	ND	5.821	ND	ND	ND
Fe ₂ O ₃	1.914	0.829	0.536	0.667	0.407
Others	1.06	0.831	1.835	0.477	0.571
SiO ₂ /Al ₂ O ₃	6.5	2.2	2	2.5	2.1

**Figure 2: Comparative oxide composition of adsorbents**

3.3 XRD Analysis

XRD analysis was conducted to gain insight into the crystal structure of the samples. The various crystalline phases present in CSAC, Zeolite Y, and the hybrid adsorbents in the range of 2θ from 2° to 70° are shown in Figure 3. The XRD peaks list and crystal size are displayed in Table 3. For CSAC, a main peak appears at 14.74° with a crystallite size of $29 \text{ \AA}$. The broad diffraction peak at approximately $2\theta=24^\circ$ corresponds to the (002) of the hexagonal carbon-like reflections (Mohamed *et al.*, 2022). The broad nature of the main peaks at low 2θ and the existence of short-range order in the carbon structure from the XRD pattern indicate high amorphousness with increased

porosity as a result of chemical activation (Mohamed *et al.*, 2022). The lack of sharp graphitic reflections suggests that activation enhances disordered carbon domains and widens pore structures rather than producing crystalline carbon. This disorder is favorable for adsorption applications because it increases surface defects and functional sites for dye interaction (Zerin *et al.*, 2025). The CSAC sample contains significant amounts of silicon oxide (34%) and graphite (23%), with woodhouseite (19.8%) and calcium aluminum silicate hydrate (14.8%). Minor phases include goethite (3.5%), sylvine (1.6%), and refikite ($\text{C}_{20}\text{H}_{32}\text{O}_2$, 2.8%). The higher graphite content indicates increased carbon ordering, though still disordered rather than highly

crystalline. This combination of amorphous-graphitic carbon with mineral inclusions is typical for activated carbons derived from biomass (Yang *et al.*, 2021). The Zeolite Y sample displays multiple sharp peaks at 6.44°, 10.44°, 12.21°, 16.00°, and 24.12°, with crystallite sizes ranging from approximately 535 Å to 1725 Å (average around 922 Å). This indicates high crystallinity and an ordered faujasite structure in Zeolite Y. These prominent reflections align with a well-ordered aluminosilicate lattice, consistent with reports in the literature on Zeolite Y (Marnani *et al.*, 2024). The large crystallite sizes highlight the mechanical stability and structural rigidity of the zeolite framework compared to disordered carbons (Auerbach *et al.*, 2003). Zeolite Y predominantly consists of the faujasite phase (81%), which is the characteristic aluminosilicate framework responsible for ion exchange and adsorption. Minor phases include silicon oxide (8%) and albite (NaAlSi₃O₈, 10%). The high purity of faujasite confirms the structural integrity of the zeolite used. This crystallinity, which may improve the selectivity of the adsorbents, is reflected in zeolite-rich hybrids (Marnani *et al.*, 2024). Sample 2 (CSAC 20%/Zeolite 80 %) hybrid exhibits peaks at 14.8°, 17.2°, and 22.06°, with crystallite sizes ranging between 30-37 Å (average 33 Å). The coexistence of broad carbon-related peaks with sharper reflections from zeolite, which diminishes as zeolite content increases, indicates the successful integration of the two phases. Carbon maintains structural disorder (amorphous contribution), while zeolite provides crystallinity. This dual nature reflects a synergistic microstructure, retaining porosity from carbon while incorporating zeolitic ion-exchange frameworks. This carbon-dominant hybrid is rich in SiO₂ (59%) and

contains alunogen ($\text{Al}_2(\text{SO}_4)_3 \cdot 16\text{H}_2\text{O}$, 34%) and minor sanjunitite ($\text{Al}_2(\text{PO}_4)(\text{SO}_4)(\text{OH}) \cdot 9\text{H}_2\text{O}$, 4.8%) plus traces of KCl. The strong silica signal reflects zeolite incorporation, while aluminum sulfate phases suggest ion-exchange or framework modifications during synthesis. Sample 3 (CSAC 50%/Zeolite 50%) hybrid showed a dominant sharp reflection at 23.75° ($d \approx 14.42 \text{ \AA}$) with a moderate crystallite size of $386 \text{ \AA}$. The peak intensity suggests zeolite influence dominates. In this balanced hybrid, silicon oxide dominates (63%), while zeolite-specific sodium aluminum silicate (20.5%) appears, confirming the integration of the zeolite crystalline framework. Secondary phases include brushite ($\text{CaHPO}_4 \cdot 2\text{H}_2\text{O}$, 10%) and residual graphite (6%). This phase distribution reflects a synergistic coexistence of crystalline zeolite domains and carbon, with phosphate phases likely arising from the biomass precursor. For sample 4 (CSAC 80%/Zeolite 20 %) hybrid, with increasing zeolite content, peaks become sharper and more numerous, notably at 6.36° , 67.36° and 67.85° corresponding to the zeolite's crystalline framework. Crystallite sizes are very large ($450\text{-}548 \text{ \AA}$), with an average of $502 \text{ \AA}$, indicating that highly crystalline zeolite phases dominate the structure, with the highest coherent scattering domain exhibiting a perfect arrangement of unit cells (Akande, 2015). This composite exhibits the ordered zeolite framework; silicon oxide (51%) and aluminum-silicon oxide (17%) dominate, accompanied by significant graphite (28%) and minor woodhouseite (3.6%). This hybrid preserves zeolite crystallinity while embedding dispersed carbon domains. The XRD results correlate well with the morphological features observed in the SEM micrographs (Figure 1).

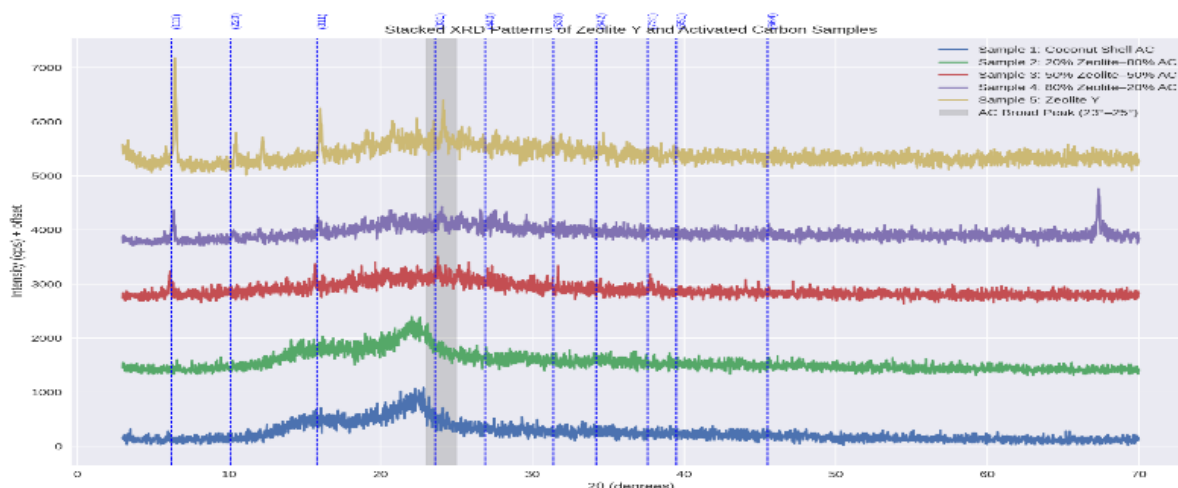


Figure 3: XRD patterns of CSAC (Sample 1), Zeolite Y (Sample 5), and hybrid adsorbents of CSAC and Zeolite Y (Samples 2, 3, and 4)

This synergistic interaction enables multifunctional treatment performance by simultaneously facilitating adsorption of hydrophobic organic dyes through carbonaceous domains and selective removal of ionic

species through zeolitic ion-exchange mechanisms, which is particularly advantageous for addressing the complex and heterogeneous contaminant profile typical of textile wastewater streams.

Table 3: XRD Peaks List and Crystal Size

Sample	2 θ , °	dÅ	FWHM	Crystal size (Å)	Ave. size (Å)
Sample 1	14.74 (12)	6.01 (5)	2.9 (3)	29 (3)	29
Sample 2	14.8 (4)	5.99 (17)	2.3 (5)	37 (8)	
	17.2 (2)	5.14 (6)	2.8 (8)	30(8)	33
	22.06 (11)	4.03 (2)	2.66 (17)	32 (2)	
Sample 3	6.123 (9)	14.42 (2)	0.22 (2)	386 (41)	386
Sample 4	6.36 (3)	13.89 (5)	0.16 (4)	508 (109)	
	67.365 (8)	1.38894 (15)	0.22 (2)	450 (45)	502
	67.85 (3)	1.3801 (5)	0.18 (9)	548 (267)	
Sample 5	6.444 (14)	13.71 (3)	0.155 (15)	535 (52)	
	10.44 (3)	8.46 (2)	0.15 (3)	574 (130)	
	12.21 (3)	7.242 (18)	0.15 (3)	547 (94)	922.6
	16.008 (8)	5.532 (3)	0.07 (2)	1232 (432)	
	24.12 (2)	3.687 (3)	0.05 (2)	1725 (727)	

3.4 FTIR Spectra Analysis

The presence of various functional groups on the surface of the samples was analyzed using a Fourier Transform Infrared (FTIR) spectrometer (Shimadzu 8400s), and the IR spectra are shown in Figure 4. The FTIR spectra provide important information on the functional groups responsible for dye adsorption during textile wastewater treatment. The CSAC shows characteristic peaks of lignocellulosic biomass. A broad band around 3402.54 cm^{-1} corresponds to O–H stretching, attributed to alcohols, phenols, and absorbed water-common in lignin and cellulose structures (Hussaini *et al.*, 2025). The peaks near 2924.18 cm^{-1} indicate C–H stretching vibrations of aliphatic $-\text{CH}_2$ and $-\text{CH}_3$ groups, typical of hemicellulose and cellulose (Salih *et al.*, 2022). This peak is more prominent in Sample 1 and moderately present in Sample 2, and gradually weakens with increasing zeolite content. This decline signifies progressive elimination of hydrocarbon moieties and increased dominance of inorganic aluminosilicate structures in zeolite-rich composites. The peak detected around 1743.71 cm^{-1} is linked to the C=O stretching of carbonyl and ester groups found in hemicellulose (Al-Bayati *et al.*, 2025). Peaks near 1643.41 cm^{-1} represent aromatic C=C and conjugated carbon structures, which are typical of activated carbon's graphitic domains (Alawam *et al.*, 2025; Janekarn *et al.*, 2020). Overall, the CSAC contains numerous oxygen-containing functional groups, which can promote surface hydrophilicity and enhance π – π interactions with aromatic dyes such as methylene blue and disperse blue (Luz Godino-Salido *et al.*, 2014). The presence of C–O and C–O–C stretching bands at

1033.88 cm^{-1} indicates residual oxygenated sites capable of forming hydrogen bonds and electrostatic interactions important for adsorbing anionic dyes (Dai *et al.*, 2023). These findings confirmed that activation increases surface heterogeneity and creates more active functional groups, thereby enhancing adsorption capacity for both cationic and anionic dyes.

Zeolite Y displays a distinct peak in the 1150-1000 cm^{-1} region, a characteristic of crystalline zeolite frameworks. The hybrid spectra show a combination of activated carbon and zeolite-specific peaks (Si-O-Si/Al-O-Si). The wide and broad transmission band between 3600–3200 cm^{-1} corresponds to O–H stretching of structural hydroxyl groups and adsorbed water in zeolite frameworks as reported by Oyedoh *et al.* (2025). This band, whose intensity slightly diminishes with increasing zeolite content due to reduced surface hydroxylation in crystalline zeolite structures, is located at 3402.43 cm^{-1} for all the hybrids and at 3394.83 cm^{-1} for zeolite. The appearance of peaks at 1080.17, 1087.89, and 1087.89 cm^{-1} for Samples 3, 4, and 5, respectively, is more pronounced than in CSAC, indicating asymmetric stretching of Si–O–Al and Si–O–Si (Alawam *et al.*, 2025), confirming the incorporation of zeolite into the hybrid adsorbents, and indicating the retainment of zeolite structure. Additionally, characteristic low-wave number bands at 455.22 and 833.28 cm^{-1} (zeolite lattice bending modes) for zeolite-rich samples correspond to symmetric stretching and bending of Si–O–Al, identifying the crystalline nature of the zeolitic component (Alawam *et al.*, 2025; Shah *et al.*, 2023). Similar FTIR bands were observed among the samples; differences in band

intensities and small wave number shifts provide evidence of successful formation and interfacial interaction within the hybrid materials.

From an adsorption standpoint, the carbon phase provides aromatic domains and oxygen-containing surface functionalities that promote hydrophobic interactions, hydrogen bonding, and π - π electron

donor–acceptor interactions with organic dye molecules, while the zeolite phase introduces permanently negatively charged framework sites that enhance electrostatic attraction and ion-exchange capacity toward cationic dyes such as methylene blue (Arias *et al.*, 2026).

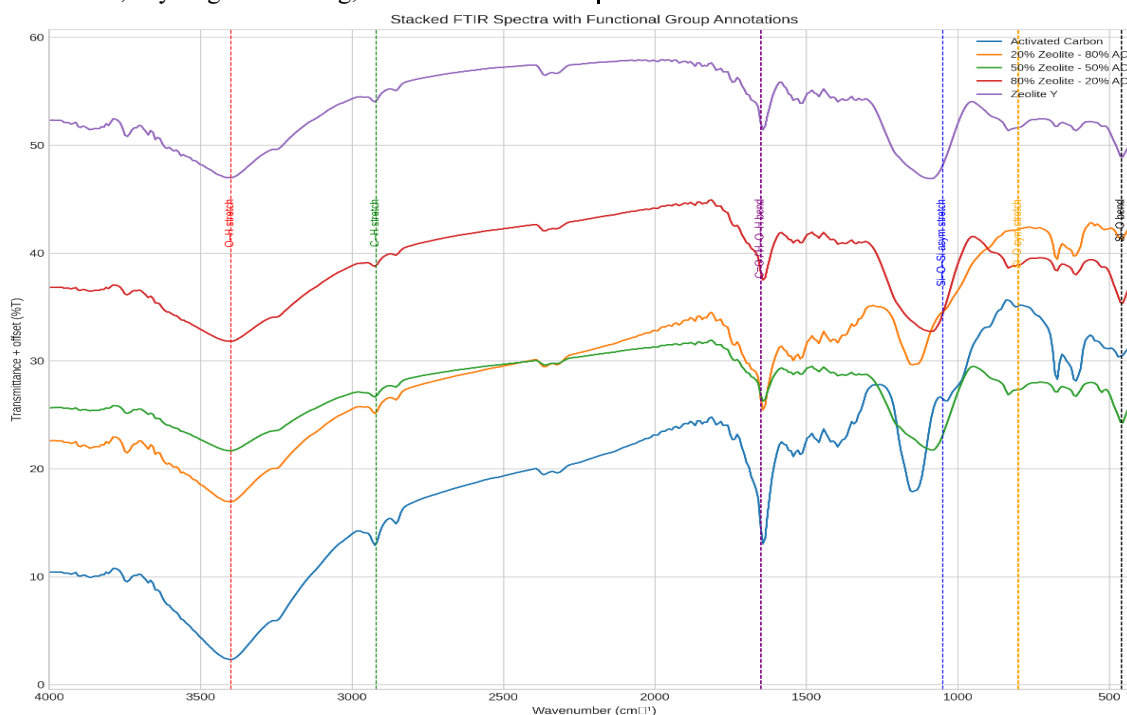


Figure 4: FTIR Spectra of CSAC (Sample 1), Zeolite Y (Sample 5), and hybrid adsorbents of CSAC and Zeolite Y (Samples 2, 3 and 4)

3.5 Surface Area and Pore Characteristics

The adsorption capacity of the prepared adsorbents was evaluated by measuring their surface area, pore volume, and pore size, parameters widely recognized as critical determinants of dye uptake from textile wastewater. The BET surface area and pore structural parameters of CSAC, Zeolite Y, and their hybrids are summarized in Table 4. The raw Coconut Shell (CS) and Coconut Shell Char (CSC) exhibit relatively low BET surface areas of 141 and 252 m²/g and total pore volumes of 0.08666 and 0.1394 cm³/g, accompanied by limited micropore development of 0.05902 and 0.09935 cm³/g, respectively. These characteristics indicate poorly evolved pore structures that restrict diffusion and limit adsorption capacity for dye molecules, which require mesoporous channels for effective diffusion and retention. This observation is consistent with previous studies, which reported that untreated lignocellulosic precursors generally possess poor porosity and limited adsorption capacity due to their dense structure, as reported by (Foo & Hameed, 2010). Although CSC shows moderate improvement over CS due to pore formation during carbonization, as observed by Mohammad (2015).

The CSAC (Sample 1) exhibits substantial improvements in textural properties, with a BET surface area of 483.1 m²/g and a pore volume of 0.2355 cm³/g. The increased micropore volume (0.1700 cm³/g) reflects effective activation, which generates abundant adsorption sites through the development of mesopores (pore size of 2.135 nm), which is highly favourable for adsorption of both cationic and anionic dyes through pore filling, hydrophobic interactions, and π - π electron donor-acceptor interactions with aromatic dye structures (Luz Godino-Salido *et al.*, 2014). The surface area, pore volume, and pore size are slightly higher than the values of 479.17m²/g, 0.15cm³/g, and 0.62 nm, respectively, obtained by Lee *et al.* (2018).

The prepared hybrid adsorbents exhibit progressively enhanced surface area and pore characteristics as the zeolite content increases, reflecting synergistic interactions between the carbon and zeolite phases. Sample 2 (20% Zeolite/80% CSAC) shows a higher BET surface area of 512.4 m²/g and pore volume 0.2866 cm³/g than CSAC, with increased micropore volume (0.1929 cm³/g). This improvement indicates that zeolite incorporation enhances microporosity while preserving carbon-derived adsorption sites.

Sample 3 (50% zeolite/50% CSAC) maintains a high surface area (554.9 m²/g) and a pore volume (0.3074 cm³/g). Sample 4 (80% Zeolite/20% CSAC) exhibits a further increase in BET surface area (580 m²/g) and the highest pore volume among the hybrid samples (0.3268 cm³/g), together with elevated micropore volume (0.2298 cm³/g). Synergistic effects of carbon–zeolite composites have been reported in previous studies, where the combination of carbon’s high surface area and zeolite’s crystalline framework improved adsorption efficiency (Alawam *et al.*, 2025; Mohamed *et al.*, 2022). Zeolite Y (Sample 5) has the highest BET surface area (659.6 m²/g) and a highly ordered microporous structure, as evidenced by its substantial micropore volume (0.2319 cm³/g). The crystalline

framework and high surface area enable selective adsorption and ion exchange for cationic dyes and small molecules (Auerbach *et al.* 2003). Overall, the results demonstrate that activation and hybrid adsorbent formation markedly improve the adsorption properties of coconut shell-derived adsorbents. Activated carbon and zeolite-rich composites provide mesoporous structures with high surface area and pore volume, enabling efficient removal of diverse dye molecules from textile wastewater. These findings corroborate earlier reports that mesoporous adsorbents with pore sizes in the range of 2-3 nm and high surface areas are optimal for dye removal (Foo & Hameed, 2010).

Table 4: Surface Area and Pore characteristics of adsorbents

Sample	S _{BET} (m ² /g)	S _{Langmuir} (m ² /g)	Pore vol _{BJH} (cm ³ /g)	Micropore vol _{DR} (cm ³ /g)	Pore size _{BJH} (nm)
CS	141	424.9	0.08666	0.05902	1.132
CSC	252	1365	0.1394	0.09935	2.126
Sample 1	483.1	11830	0.2355	0.1700	2.135
Sample 2	512.4	1452	0.2866	0.1929	2.093
Sample 3	554.9	3329	0.3074	0.2111	2.123
Sample 4	580	1490	0.3268	0.2298	2.072
Sample 5	659.6	75500	0.3303	0.2319	2.125

4.0 CONCLUSION

This study demonstrated the successful synthesis and characterization of Zeolite Y/Coconut Shell Activated Carbon (CSAC) hybrid adsorbents, highlighting their structural and functional advantages over the individual components. Multi-technique characterization validates zeolite-carbon composites as effective, sustainable adsorbents suitable for textile wastewater. SEM confirms porous morphology, XRD shows increasing crystallinity with zeolite content, FTIR reveals functional group evolution, XRF confirms elemental composition supporting multifunctional performance, and a surface area analyzer reveals high surface area of (≈512–580 m² g⁻¹). Together, these techniques demonstrate that composites strategically balance the strengths of carbon and zeolite, offering a robust solution for complex textile effluent treatment.

The results of analysis demonstrate that increasing zeolite incorporation progressively shifts surface chemistry from carbon-dominated functional groups toward aluminosilicate-rich frameworks. This transition alters the dominant adsorption mechanisms from hydrophobic and π - π interactions to electrostatic attraction and ion-exchange processes. Such tunable surface characteristics are particularly advantageous

for treating complex textile wastewater containing diverse organic and ionic dye contaminants.

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IJSGS FUGUSAU VOL. 12(3)

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