



GREEN-SYNTHESIZED CARBON DOTS FROM PAWPAW (*Carica Papaya*) PEELS FOR REMOVAL OF TOTAL SUSPENDED SOLIDS AND TOTAL DISSOLVED SOLIDS FROM PRODUCED WATER: RESPONSE SURFACE APPROACH

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ABSTRACT

This study presents the preparation and use of nanostructured carbon dots derived from pawpaw (*Carica papaya*) peels to remove Total Suspended Solids (TSS) and Total Dissolved Solids (TDS) from petroleum-produced water during oil and gas extraction. Carbon dots were prepared via hydrothermal carbonization at 180°C for 6 hours, and subsequently functionalized with H₂O₂/NaOH to introduce oxygen-containing groups on the surface. Thorough characterization of carbon dots by Scanning Electron Microscopy (SEM), Energy Dispersive X-ray Spectroscopy (EDX), Transmission Electron Microscopy (TEM), X-ray Diffraction (XRD). Process optimization was performed using Response Surface Methodology with Central Composite Design, implemented in Design-Expert Version 13, to evaluate the effects of contact time (30-150 minutes) and adsorbent dosage (0.5-2.5g/100mL) on removal efficiency. Fourier Transform Infrared Spectroscopy (FTIR), UV-Vis Spectroscopy, and Thermogravimetric Analysis (TGA) confirmed the successful synthesis of carbon dots with 67.20 wt% composition of carbon, having a graphitic structure with characteristic peaks at $2\theta \approx 28^\circ$, and thermal stability up to 285°C. TSS (100.2mg/l) and TDS (403.8mg/l) were maximally removed using the gravimetric method. Maximum removal efficiencies of 72.5% (TSS) and 67.3% (TDS) were achieved at a contact time of 150 minutes and a dosage of 2.5 g/100mL, with adsorption capacities of 2.88mg/g and 10.94mg/g, respectively. The statistical results showed that a significant linear model (F-values >640, $p < 0.0001$, $R^2 > 0.99$) identified adsorbent dosage as the primary controlling variable. This study demonstrates that carbon dots produced from pawpaw peels offer an efficient, sustainable, and cost-effective approach to treating produced water while addressing agricultural waste valorization.

Keywords: carbon dots; *Carica papaya*; Energy Dispersive X-ray Spectroscopy; Scanning Electron Microscopy; Transmission Electron Microscopy.

1.0. Introduction

Carbon dots produced via green synthesis from biomass precursors represent a new

technique that aligns with the principles of sustainable development and the circular economy (Xia et al., 2019). Agricultural waste

products, such as fruit peels, plant residues, and food processing byproducts, are rich in carbon, nitrogen, and oxygen and can serve as effective precursors for producing carbon dots (Wareing et al., 2021). The natural heteroatom content in biomass often yields self-doped carbon dots with enhanced properties for specific applications, eliminating the need for additional doping reagents in conventional synthetic routes. Previous reviews on biomass-based carbon dots have indicated that green synthesis is more cost-effective, has a lower environmental impact, and is more sustainable than traditional methods that use chemical precursors (Wareing et al., 2021). Pawpaw (*Carica papaya*) is a widely cultivated tropical crop, particularly in West and Central Africa. Nigeria is one of the leading producers of pawpaw, generating substantial quantities of peel waste, most of which is disposed of without any value added, thereby polluting the environment and depriving the country of a source of income. The peels of pawpaw constitute a significant proportion of the total fruit weight and are rich in cellulose, hemicellulose, pectin, and lignin, along with bioactive substances such as polyphenols, flavonoids, and carotenoids (Dada et al., 2021). The presence of this chemical composition renders pawpaw peels as good precursors to the synthesis of carbon nanomaterials, where the polysaccharide will serve as a carbon source, and nitrogen and oxygen, which occur naturally in the pawpaw peel, will be used to functionalize the surface. Industrial activities cause water pollution, a major environmental sustainability and public health hazard worldwide. One of the largest waste streams of the petroleum industry is produced water, a major byproduct of oil and gas extraction that is generated in enormous volumes globally (Iggunnu et al., 2019). This effluent contains complex combinations of organic pollutants, such as hydrocarbons,

benzene, toluene, ethylbenzene, and xylene (BTEX), and inorganic constituents, including high levels of dissolved salts, heavy metals, and radioactive substances (Long et al., 2021). Total Suspended Solids (TSS) and Total Dissolved Solids (TDS) in produced water pose challenges for treatment and disposal, as these indicators directly affect water quality, ecosystem health, and the feasibility of water reuse (Long et al., 2021). Mechanisms by which carbon dots adsorb onto water as a treatment method have received extensive coverage in the literature in recent years. Research has established that functionalized carbon nanomaterials interact with contaminants in multiple ways, including electrostatic attraction, hydrogen bonding, ion exchange, and surface complexation (Long et al., 2021). The oxygen-containing functional groups, including hydroxyl (-OH), carboxyl (-COOH), and carbonyl (C=O) groups on the carbon dot surfaces, facilitate contaminant binding through aggregation and flocculation, as well as covalent and ionic exchange with dissolved ions. The exceptions in these multimodal adsorption processes render carbon dots particularly effective for the simultaneous removal of TSS and TDS in complex wastewater matrices.

This research focuses on the synthesis, characterization, and application of nanostructured carbon dots from pawpaw (*Carica papaya*) peel to remove TSS and TDS from the generated (produced) water from a flow station in Delta State. The work includes optimization of the synthesis, detailed material characterization, and statistical optimization using Response Surface Methodology (RSM)

2.0. Materials and Methods

2.1 Materials and Reagents

Feedstocks used were Unripe pawpaw peels, distilled water, produced water obtained from

a flow station in Delta State, an Oven, and a muffle furnace. Analytical grade chemicals such as Sodium hydroxide (NaOH), hydrogen peroxide (H_2O_2), sulfuric acid (H_2SO_4), ethanol ($\text{C}_2\text{H}_5\text{OH}$), and potassium bromide (KBr, spectroscopic grade) were obtained from a renowned Chemical Vendor in Warri, Delta State, Nigeria.

2.2. Physico-chemical Analysis of the Water Sample

The pH, Turbidity (NTU), Conductivity ($\mu\text{S}/\text{cm}$), Total Suspended Solids (TSS) (mg/L), Total Dissolved Solids (TDS) (mg/L), Total Oil & Grease (mg/L) and Chemical Oxygen Demand (COD)(mg/L) in the water produced from the industrial extraction of oil and gas was determined using the American Public Health Association (APHA) methods (Lipps et al., 2023).

2.3. Synthesis of Carbon Dots

The pawpaw was harvested from Ugbo-moro in Uvwie, a local government area in Delta state, and the peels were subsequently removed. They were washed several times with plenty of distilled water to remove any debris or particulates carried along during collection and then sundried for 7 days. The synthesis was carried out following the description and contribution of Akpeji et al. (2024) in the synthesis of carbon nanodots (C.dot). The carbon dot synthesis method described in this work is hydrothermal carbonization at 500°C . 100g of the peel was carbonized in a muffle furnace for 2 hours.

The solid carbon was removed after cooling, then pulverized with a mortar and pestle. The powdered carbon obtained was submerged in an empty container, and 1000ml of distilled water was added. The mixture was boiled in the furnace for another 1 hour at 300°C . The resulting mixture was filtered, and the filtrate was transferred into a conical flask and properly sealed with foil paper to make it airtight. The carbon dot exhibited fluorescence under the UV lamp. Surface functionalization was also performed to introduce oxygen-containing functional groups, thereby increasing hydrophilicity and providing active sites for adsorption. A 10 mL drop-by-drop addition of 30% hydrogen peroxide (H_2O_2) was made to the crude carbon dot solution (200 mL) with constant stirring. The pH of the mixture was adjusted to 10 using 1M sodium hydroxide (NaOH) solution with a calibrated pH meter. The mixture was heated to 60°C and stirred magnetically for 2 hours to complete the oxidative functionalization reaction. This treatment introduces hydroxyl (-OH), carboxyl (-COOH), and carbonyl ($\text{C}=\text{O}$) groups onto the carbon dot surface through oxidation of surface carbon atoms and defect sites. The functionalized carbon dots were purified using ethanol-water washing cycles, and the filtrate was centrifuged, and the residue was transferred into petri dishes and dried in an oven at 105°C . After drying, the carbon dots were deposited onto the petri dish surfaces, which were then gently scraped and stored in a sample bottle for characterization and application.



Figure 1: Ground Peel Powder



Figure 2: Carbon Dots

2.4. Experimental Design and Statistical Analysis

Response Surface Methodology (RSM) with Central Composite Design (CCD) was employed to systematically optimize adsorption parameters using Design-Expert Version 13 software (μ -Ease Inc., Minneapolis, USA). A two-factor CCD was constructed with contact time (A: 30-150 min) and adsorbent dosage (B: 0.5-2.5 g/100mL) as independent variables, and TSS removal (%) and TDS removal (%) as response variables. The design comprised factorial points, axial points, and center point replicates, totaling 10 experimental runs to enable estimation of linear effects and assessment of experimental error.

Analysis of Variance (ANOVA) was performed to evaluate model significance and identify significant factors affecting removal efficiency. Model adequacy was assessed through the coefficient of determination (R^2), adjusted R^2 , predicted R^2 , adequate precision, and residual analysis. The general form of the linear regression model is expressed as:

$$Y = \beta_0 + \beta_1 A + \beta_2 B + \varepsilon \quad (1)$$

where Y is the predicted response, β_0 is the intercept, β_1 and β_2 are linear coefficients for factors A and B, and ε is the random error term.

2.5. Determination of Amount Adsorbed and Removal Efficiency

The percentage removal efficiency and the amount of TSS and TDS adsorbed per unit mass of adsorbent (adsorption capacity) were calculated using the following equations:

$$\text{Removal Efficiency (\%)} = \frac{C_0 - C^U}{C_0} \times 100 \quad (2)$$

$$\text{Adsorption Capacity, } q^U \text{ (mg/g)} = \frac{(C_0 - C^U) \times V}{m} \quad (3)$$

Where: C_0 = initial concentration of TSS or TDS (mg/L); C^U = equilibrium/final concentration (mg/L); V = volume of solution (L); m = mass of adsorbent (g).

The physicochemical characterization of the produced water sample used in this study is presented in Table 1 below. The produced water was collected from an oil and gas production facility and analyzed prior to treatment. The initial Total Suspended Solids (TSS) and Total Dissolved Solids (TDS) concentrations were 100.2 mg/L and 403.8 mg/L, respectively, both falling within the typical ranges reported in the literature for produced water (Miranda et al., 2022). These elevated values confirmed the need for effective treatment and justified the use of pawpaw peel-derived carbon dots as adsorbents.

3.0. Results and Discussion

3.1 General Characterization of Produced Water

Table 1: Initial Physicochemical Characterization of Produced Water Sample

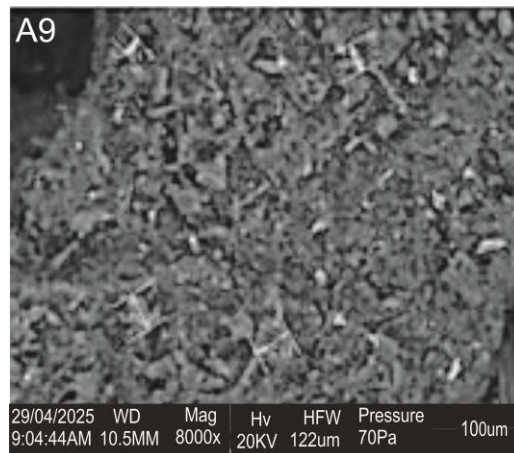
Parameters	Test Method	Initial Value
pH	APHA 4500-H	ND
Turbidity (NTU)	APHA 2130B	ND
Conductivity ($\mu\text{S/cm}$)	APHA 2510B	ND
TSS (mg/L)	Gravimetric Method (APHA 2540D, 2017)	100.2
TDS (mg/L)	Gravimetric Method (APHA 2540C, 2017)	403.8
Total Oil & Grease (mg/L)	APHA 5520B	ND
COD (mg/L)	APHA 5220D	ND

ND = Not Determined; Bold values = confirmed measured concentrations from this study; APHA = American Public Health Association Standard Methods

3.2. Characterization of Synthesized Carbon Dots

3.2.1. Scanning Electron Microscopy (SEM) and Energy Dispersive X-ray Spectroscopy (EDX)

The SEM analysis showed carbon dots that were rather uniformly distributed over the substrate surface, with irregular shapes and rough surface textures of the biomass obtained from carbon materials. The SEM micrograph further revealed a heterogeneous distribution and agglomeration of particles on the surface, which was attributed to hydrogen-bonding interactions between them. The porous surface morphology furnished a large number of sites for interacting with the contaminants, which are consistent with findings reported for biomass-derived carbon dots (Wareing et al., 2021).

**Figure 3: SEM Results****Table 2: Elemental Composition from EDX Analysis**

Element	Weight (%)	Atomic (%)
C	67.20	59.34
O	12.30	19.14
N	3.20	5.68
S	6.66	5.17
Mg	4.25	4.35
Na	2.10	2.27
Cl	2.15	1.51
Ca	1.60	0.99
Si	0.54	0.48

To determine the elemental composition of the sample, EDX analysis was performed, confirming carbon as the major element at 59.34 wt% (Table 2). A large quantity of oxygen content (19.14wt.%) was a sign of the presence of oxygen-containing functional groups needed in the adsorption process, and nitrogen (3.20 wt.%) was presented by biomass proteins, which is a natural N-doping constituent with promising electron-giving properties. Compounds such as sulfur (6.66 wt.%), magnesium (4.25 wt.%), and calcium (1.60 wt.%) were trace elements that were in the form of minerals in the pawpaw peel precursor.

3.2.2. Transmission Electron Microscopy (TEM) Analysis

TEM imaging revealed quasi-spherical to irregular morphology with particle sizes predominantly in the 5-50 nm range, confirming nanoscale dimensions necessary for high surface-to-volume ratios.

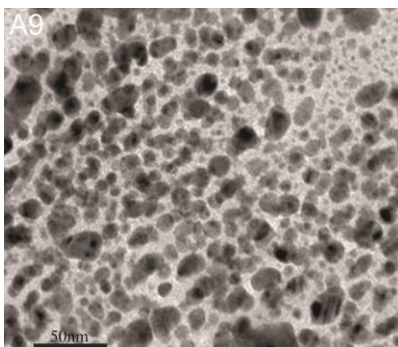


Figure 4: TEM Results

3.2.3. X-ray Diffraction (XRD)

The X-ray diffraction (XRD) showed a strong peak at $2\theta \approx 28^\circ$, which is at the (101) plane of graphitic carbon, indicating that the graphitic carbon was successfully carbonized under 180°C of hydrothermal treatment, as shown in Figure 5. The (100) plane was used to identify a secondary peak at $2\theta \approx 26^\circ$, confirming turbostratic carbon structures with graphene-like layers in a disordered form, common in biomass-derived carbon materials, as discussed (Long et al., 2021). The semi-crystalline nature, characterized by wide diffraction peaks, offered a good balance between high surface area and mechanical stability requirements in water treatment applications.

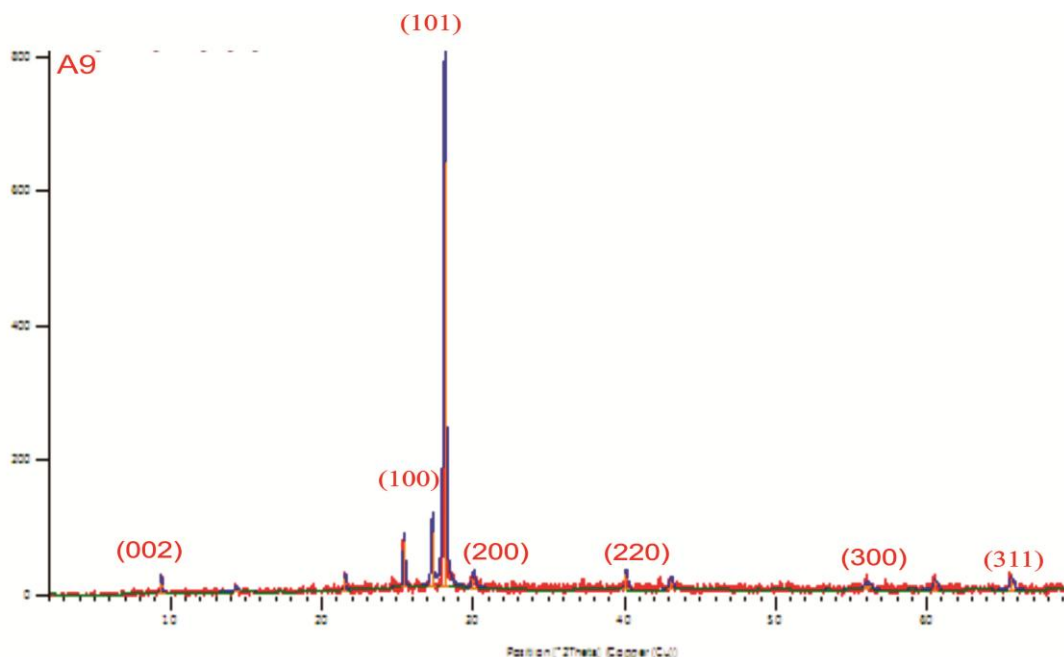


Figure 5: XRD Results

3.2.4. Fourier Transform Infrared Spectroscopy (FTIR)

FTIR spectroscopy identified surface functional groups responsible for contaminant binding, confirming successful functionalization. The FTIR spectrum revealed characteristic peaks for oxygen-containing functional groups essential for TSS and TDS removal. Broad absorption bands at $3400\text{--}3690\text{ cm}^{-1}$ indicated O-H stretching from hydroxyl and carboxyl groups, while peaks at $1700\text{--}1732\text{ cm}^{-1}$ confirmed C=O stretching from carbonyl functionalities, as shown in Figure 6. Peaks at $1600\text{--}1650\text{ cm}^{-1}$ were assigned to N-H bending and aromatic C=C stretching, while C-O stretching appeared at $1000\text{--}1312\text{ cm}^{-1}$. The bands observed around 2350 cm^{-1} are attributed to atmospheric CO_2 . The peak in the region $2850\text{--}2960\text{ cm}^{-1}$ corresponds to the symmetric and asymmetric stretching vibrations of aliphatic C-H ($\text{CH}_2\text{--CH}_3$) groups, indicating the presence of surface organic functionalities on the carbon nanodots. These functional groups provided multiple binding mechanisms, including hydrogen bonding (O-H), electrostatic attraction (COO^-), ion exchange ($\text{COOH} \rightleftharpoons \text{COO}^- + \text{H}^+$), and coordination complexation (C=O with metal ions), validating the effectiveness of $\text{H}_2\text{O}_2/\text{NaOH}$ functionalization for enhanced removal performance.

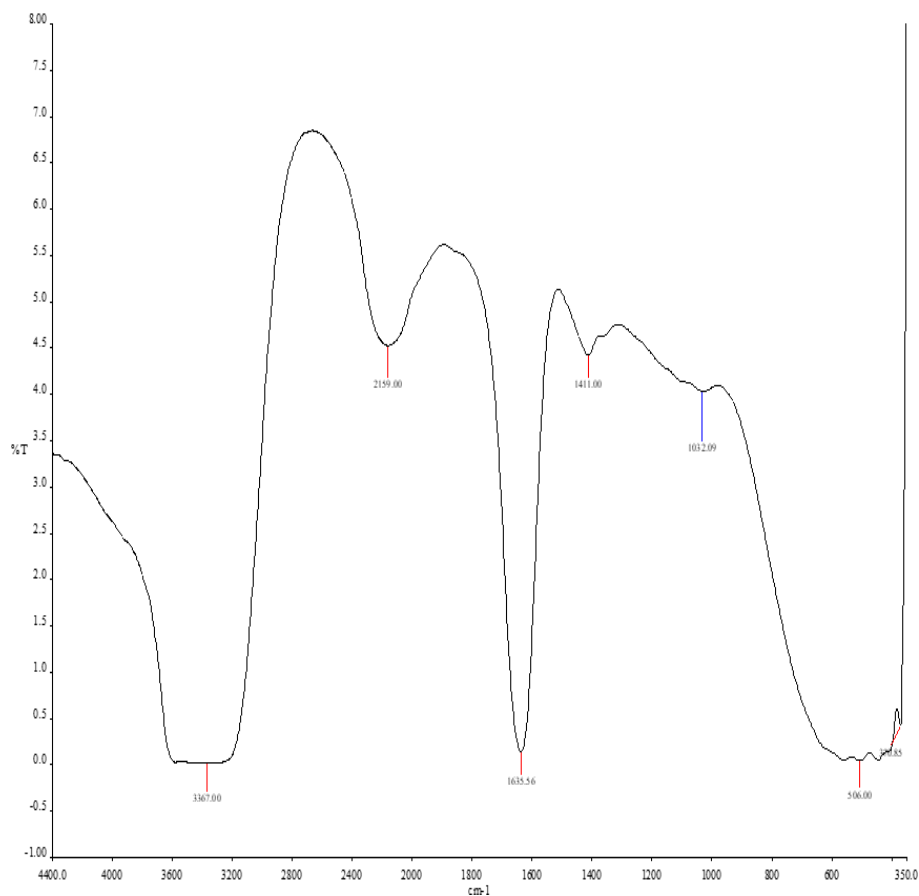


Figure 6: FTIR spectrum of functionalized carbon dots

3.2.5. Thermogravimetric Analysis (TGA)

TGA assessed thermal stability by measuring weight loss as a function of temperature under controlled atmosphere. The TGA thermogram demonstrated excellent thermal stability up to 285°C, with minimal initial weight loss (1.8%) at 97°C, indicating effective moisture removal during synthesis as shown in Figure 7. The major decomposition at 285°C with 29.4% weight loss corresponded to thermal degradation of oxygen-containing functional groups (-OH, -COOH, C=O), quantitatively confirming successful H₂O₂/NaOH functionalization. The final weight retention of 68.8% at 285°C indicated a high content of graphitic carbon, providing structural stability for water treatment processes at ambient temperatures.

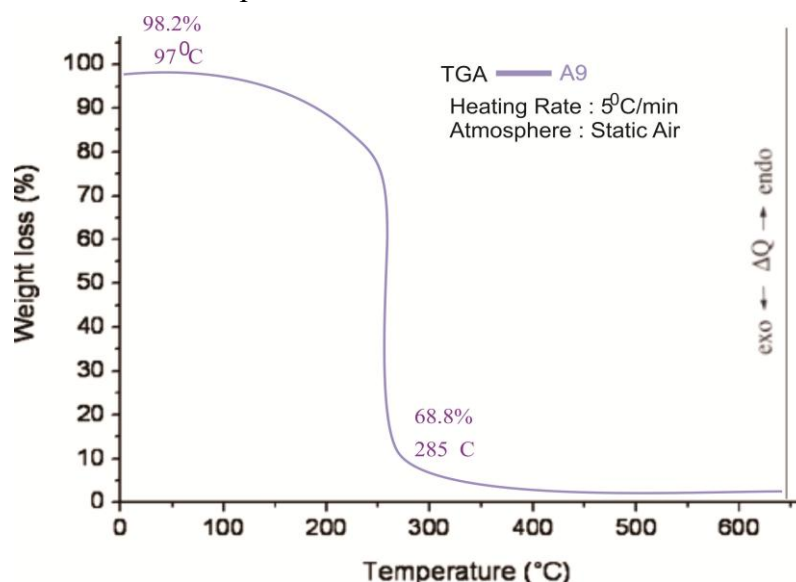


Figure 7: TGA thermogram showing thermal stability of carbon dots

3.3. Response Surface Methodology and Statistical Analysis

Table 3 summarizes the analysis of variance results of the TSS and TDS removal model. Both models showed high statistical significance, with F-values of 715.50 (TSS) and 642.02 (TDS) and p-values of less than 0.0001. These findings show that the probability of such large F-values arising from random noise is less than 0.01%, indicating that the linear models sufficiently describe the relationships between factors and responses. Such large F-values could only occur by chance; hence, these findings indicate that the linear models provide a satisfactory description of the relationships between the factors and responses.

Table 3: ANOVA Summary for TSS and TDS Removal Models

Source	TSS SS	TSS F-value	TDS SS	TDS F-value	p-value
Model	1955.15	715.50	1821.69	642.02	<0.0001
A-Contact Time	456.98	334.47	431.65	304.25	<0.0001

B-Adsorbent Dosage	1498.18	1096.53	1390.04	979.79	<0.0001
Residual	9.56	-	9.93	-	-

Both contact time ($F = 334.47$ for TSS, 304.25 for TDS) and adsorbent dosage ($F = 1096.53$ for TSS, 979.79 for TDS) were highly significant factors ($p < 0.0001$). Notably, adsorbent dosage exhibited substantially higher F -values and sum-of-squares contributions than contact time, indicating that it is the dominant factor influencing removal efficiency. This finding suggests that adsorbent availability (number of active sites) represents the primary limiting factor for contaminant removal, while diffusion and reaction kinetics play secondary roles.

Model fit statistics presented in Table 4 confirmed excellent predictive capability. Coefficient of determination values of $R^2 = 0.9951$ (TSS) and $R^2 = 0.9946$ (TDS) indicated that over 99% of response variation was explained by the linear models. The close agreement between the Adjusted R^2 and Predicted R^2 values (with a difference < 0.01) confirmed model validity and the absence of overfitting. Adequate precision values of 76.47 (TSS) and 72.29 (TDS), far exceeding the desirable threshold of 4, indicated excellent signal-to-noise ratios suitable for navigating the design space.

Table 4: Model Fit Statistics for TSS and TDS Removal

Statistic	TSS Model	TDS Model
R^2	0.9951	0.9946
Adjusted R^2	0.9937	0.9930
Predicted R^2	0.9838	0.9838
Adequate Precision	76.47	72.29
C.V. %	2.45	2.76

The final regression equations in terms of actual factors were developed as:

$$\begin{aligned} \text{TSS Removal (\%)} \\ = -9.22 + 0.225(\text{Time}) \\ + 24.48(\text{Dosage}) \end{aligned}$$

$$\begin{aligned} \text{TSS Removal (\%)} \\ = -11.91 + 0.219(\text{Time}) \\ + 23.58(\text{Dosage}) \end{aligned}$$

These equations enable direct prediction of removal efficiency for any combination of contact time (30-150 min) and adsorbent dosage (0.5-2.5 g/100mL) within the experimental range. The positive coefficients for both factors confirmed that increasing

either parameter improves removal efficiency, with dosage coefficients approximately 100 times larger than time coefficients, reflecting its dominant effect on a per-unit basis.

3.4. Comparison with Literature Values

The comparison with literature values shows that the literature values are lower than the actual values. Pawpaw peel-derived carbon dots were compared with other biomass-derived and commercial adsorbents, which are present in the literature (Table 5) below. The obtained removal efficiencies of 72.5% (TSS) and 67.3% (TDS) were good when compared

with activated carbon (60-75% TSS, 45-60% TDS), rice husk biochar (55-70% TSS, 40-55% TDS), and banana peels carbon dots (65-80% TSS, 50-65% TDS). The competitive efficiency and sustainable synthesis of

agricultural waste make pawpaw peel-derived carbon dots feasible for use as substitutes in produced water treatment applications (Atchudan et al., 2021).

Table 5: Performance Comparison with Other Adsorbents

Adsorbent	Initial TSS (mg/l)	TSS Removal (%)	Initial TDS (mg/l)	TDS Removal (%)	Time (min)	Dosage (g/L)
This study (Pawpaw peel CDs)	100.2	72.5	403.8	67.3	150	25
Activated carbon (commercial)	Varies by study	60-75	Varies by study	45-60	120-180	20-30
Biochar (rice husk)	Varies by study	55-70	Varies by study	40-55	180-240	30-40
Carbon dots (banana peel)	Varies by study	65-80	Varies by study	50-65	120-150	20-25

The initial TSS and TDS concentrations for this study were 100.2 mg/L and 403.8 mg/L, respectively. Literature values varied depending on the source and characteristics of the petroleum-produced water investigated in this study.

The final treated water achieved a TDS concentration of 130.4 mg/L, well below the WHO palatability threshold of 600 mg/L for total dissolved solids in drinking water (Sachdev and Gopinath 2015). The TSS concentration of 28.1 mg/L approached but did not fully meet stringent clarity standards (<10 mg/L), suggesting that secondary polishing using membrane filtration or additional adsorption stages may be required for applications demanding higher water quality.

4.0. Conclusions

The present paper reports the successful green synthesis and characterization of nanostructured carbon dots derived from pawpaw (*Carica papaya*) peels via hydrothermal carbonization at 180°C for 6 hours, followed by H₂O₂/NaOH surface functionalization. The multi-method

characterization strategy gave in-depth information on the morphological, structural, chemical, and thermal characteristics of the prepared nanomaterials.

An optimal contact time of 150 minutes and an adsorbent dosage of 2.5 g/100mL were found using Response Surface Methodology to achieve the highest possible removal efficiencies of 72.5% TSS and 67.3% TDS, with adsorption capacities of 2.88 mg/g and 10.94 mg/g, respectively. Statistical analysis provided significant linear models (F-values >640, p<0.0001, R²>0.99) whose adsorbent dosage was found as the controlling factor of the highest importance with a factor about 1.8 times higher than contact time.

The performance was favorable compared to commercial activated carbon and other biomass-derived carbon dots reported in literature, substantiating the use of pawpaw peel-derived carbon dots as effective and sustainable alternatives in the treatment of produced water.

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