

Article

Physicochemical Properties of Pristine and Pyrolyzed CNO Synthesized via Wick Pyrolysis

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Abstract

Carbon nano-onions (CNOs) were synthesized at ambient conditions using the wick-pyrolysis technique with ghee as a precursor. A high-purity copper substrate produced unique CNOs, differing from those obtained with other metals. To purify the nanoparticles, they underwent treatment with a solvent mixture of acetone and deionized water or were pyrolyzed at 1000 °C under nitrogen without a catalyst. Various characterization techniques, including X-ray diffraction (XRD), Field Emission Scanning Electron Microscopy (FE-SEM), High-Resolution Transmission Electron Microscopy (HR-TEM), and Raman Spectroscopy, confirmed the successful formation of CNOs. Energy Dispersive Spectroscopy (EDS) and Elemental analysis (CHN) indicated the presence of oxygen in treated CNOs. X-ray photoelectron spectroscopy (XPS) revealed binding energies linked to C–O and C=O bonds. The average particle size was found to be 30–50 nm, with some agglomeration in pyrolyzed samples. A significant increase in surface area from 79.7 m²/g to 261.8 m²/g was observed, along with changes in pore radius and volume via Brunauer–Emmett–Teller (BET) analysis. Water contact angles on the CNO surface were measured at 125° and 138°, indicating hydrophobicity. Electrochemical tests on CNO-based composite electrodes yielded a specific capacitance of 109.7 F/g with 96% capacity retention over 5000 cycles.

Keywords: carbon nano-onions; pyrolysis; flame synthesis; liquid electrolyte; supercapacitor

1. Introduction

Carbon nano-onions (CNOs) are concentric multilayer fullerene-like spheres with unique structural and electronic properties. CNOs exhibit excellent electrical conductivity, high surface area, catalytic activity, and biocompatibility, making them promising for energy storage devices, including wearable devices, biosensors, supercapacitors, and batteries. CNOs are widely used in biomedical applications, including drug delivery, bioimaging, tissue engineering, and targeted cancer treatment, due to their low toxicity and dispersibility [1–4]. CNOs range from electrochemical sensors to non-invasive therapeutic agents and photothermal therapy, as well as multifunctional nanomaterials for diverse technological advancements. As of now, CNOs are synthesized via the chemical vapor deposition (CVD) technique, which is the most versatile and controllable method for large-scale production. In this approach, hydrocarbon precursors such as methane, acetylene, or ethylene are



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decomposed over transition-metal catalysts (Ni, Fe, Co, Si) supported on oxides at temperatures typically between 600 °C and 900 °C [5]. The catalytic dissolution–precipitation of carbon and subsequent diffusion processes lead to the formation of concentric graphitic shells, often accompanied by hollow cores due to the Kirkendall effect [6]. Recent studies demonstrate that CVD enables precise control over particle size, shell number, and porosity by adjusting catalyst composition, gas flow, and reaction temperature [7]. Plasma-enhanced CVD further reduces the growth temperature to ~400 °C and enables the formation of aligned arrays, while oxygen-assisted CVD improves crystallinity by selectively removing amorphous carbon [8]. Due to their scalability, reproducibility, and compatibility with composite fabrication, CVD-derived CNOs are widely employed as electrode materials in supercapacitors and sodium-ion batteries, where they exhibit high conductivity and fast ion-diffusion pathways. CNOs synthesized via CVD exhibit uniform dimensions of 20–50 nm and tunable hollow cores, which are accessible to ions, making them ideal for supercapacitor applications [9]. Specifically, Ni/Al₂O₃-catalyzed CNOs achieve capacitance values of 250–350 F/g and retain 95% of their capacity after 10,000 charge–discharge cycles when electrodes are used in composite form [10]. In sodium-ion battery applications, CVD-synthesized CNOs serve as anodes, offering a discharge capacity of around 300 mAh/g and leveraging radial diffusion pathways for improved performance [11]. The arc-discharge synthesis method is the most established route for producing highly crystalline CNOs [12]. In this method, a high-current electric arc is generated between graphite electrodes in an inert atmosphere or a liquid medium, leading to the sublimation of carbon and its rapid condensation into onion-like structures [13]. CNOs synthesized via the arc-discharge technique typically exhibit excellent graphitization, narrow interlayer spacing, and low defect density, making them attractive for applications that require high electrical conductivity and thermal stability [14]. However, the process often yields a mixture of carbon nanostructures, including nanotubes and amorphous carbon, necessitating extensive purification. Recent improvements, such as liquid-phase arc-discharge and catalyst-assisted electrodes, have enhanced yield control and reduced structural heterogeneity [13]. Nevertheless, high energy consumption and limited tunability are achieved compared to CVD and remain as challenging for industrial-scale deployment. The polydisperse CNOs produced possess fullerene-like cores, making them suitable for high-power applications [9]. Underwater arc techniques also yield pure, water-dispersible CNOs that exhibit low toxicity and fluorescence, serving as functional agents in bioimaging and drug delivery [15,16]. Additionally, they exhibit electrocatalytic activity, boosting the performance of Oxygen Reduction Reactions (ORRs) in fuel cells (onset potential ~0.9 V) and acting effectively as cathodes in lithium–sulfur batteries, effectively trapping polysulfides, achieving an energy density of up to 800 Wh/kg [17]. Pyrolysis-based synthesis offers a simpler, cost-effective alternative, relying on the thermal decomposition of carbon-rich precursors, such as polymers, hydrocarbons, biomass derivatives, or metal–organic frameworks, under inert atmospheres [18]. Depending on the precursor and heating protocol, CNOs can form through carbonization followed by self-organization into concentric shells. Recent work highlights the use of polymeric resins and organic salts to produce metal-free or metal-templated CNOs at temperatures ranging from 700 to 1100 °C [19]. Although pyrolysis generally results in broader size distributions and lower crystallinity than arc-discharge and CVD, it offers significant advantages in precursor flexibility, lower equipment costs, and sustainable production. Overall, each synthesis method offers distinct advantages and disadvantages: CVD excels in structural control and scalability, arc discharge provides superior crystallinity, and pyrolysis enables economical, versatile production. Continued optimization of these techniques is essential to tailor CNO properties for next-generation energy storage and functional nanomaterial applications. Among the various synthesis routes, flame synthesis

has emerged as a promising technique for producing high-purity CNOs in a rapid, continuous, and scalable manner, making it well-suited for energy-storage applications [20]. Flame synthesis relies on the complete combustion of hydrocarbon fuels, such as acetylene, ethylene, or propane, in an oxygen-deficient environment. This process leads to the formation of carbon vapor species that nucleate and reorganize into onion-like graphitic shells [21]. Extremely high-temperature flame environments (typically 1200–2000 °C) promote rapid carbonization and graphitization, enabling the direct formation of well-ordered multilayer CNOs without the need for metal catalysts. This catalyst-free nature is a key advantage for electrochemical applications, as it eliminates the need for post-treatment such as acid washing and prevents metal contamination that can degrade cycling stability and electrolyte compatibility in device applications [1,9]. The spherical morphology and turbostratic shell arrangement provide short ion-diffusion pathways and an abundant electrochemically accessible surface site, which are crucial for forming efficient electric double layers in supercapacitor electrodes [22]. Furthermore, the synthesis process allows partial control over shell number, defect density, and surface functionalization by adjusting flame temperature, fuel-to-oxygen ratio, and residence time [23]. Combustion techniques use different oils and fats, which are cost-effective and readily available. Among such sources of carbon, ghee is an anhydrous dairy-based fat produced by heating butter above 100 °C to remove moisture, followed by filtration of the residual solids. It provides a clean, non-toxic flame and emits a pleasant aroma. Chemically, ghee consists mainly of triglycerides, free fatty acids, phospholipids, sterols, fat-soluble vitamins (A, D, E, and K), tocopherols, carbonyl compounds, and hydrocarbons [24]. Previous studies have reported global preparation methods for cow and buffalo ghee, as well as their fatty acid compositions, which are dominated by saturated and unsaturated species [25]. It is reported that the low moisture content and high hydrocarbon fraction in ghee favor the formation of high-quality soot during combustion. As ghee is a non-hydrogenated, animal-derived saturated fat, it serves as a unique carbon precursor; however, its systematic exploration for carbon nanoparticle synthesis and energy-storage applications remains largely unexplored. Despite its long-standing use in food and its cultural impact in India, ghee remains largely unexplored as a feedstock for the synthesis of carbon nanomaterials. In this study, we utilize ghee to produce carbon nanoparticles and assess their performance in energy storage, transforming a traditional material into a functional carbon source. A brief summary of synthesis techniques, precursor sources, and potential applications of carbon nano-onions is presented in Table 1.

Table 1. Recent research on the CNO synthesis method, source, and application to support the literature review.

Source	Synthesis Technique	Application	Reference
Ghee	Flame	UV photodetectors	[26]
Cooking oil	Flame	Rhodamine B degradation	[27]
Sesame oil	Flame	Energy storage	[28]
Flaxseed oil	Wick pyrolysis	Green catalytic synthesis	[29]
Coconut oil	Wick pyrolysis	Zinc–air batteries	[30]
Xylene	Flame spray pyrolysis	Gas sensors	[31]
Clarified butter	Flame synthesis	Flexible Printed Electronics	[32]
Coal samples	Ultrasonic-assisted wet-chemical oxidation method	Bioimaging, optoelectronic, and sensing applications	[33]

Table 1. *Cont.*

Source	Synthesis Technique	Application	Reference
Phosphoric acid and chitosan gel	Hydrothermal	Sensor for biomedical applications	[34]
Phosphoric acid and acetic acid	Hydrothermal	Colorimetric sensor	[35]
Jute stick pieces	Pyrolysis method	Hybrid Lithium-Ion Capacitor	[36]
Liquid paraffin	Wick and oil flame pyrolysis and hydrothermal	Removal of industrial dye from wastewater	[37]
Wax Candle	Flame	Removal of pollutants from water.	[38]
Glucose, Ammonium Chloride, and Thiourea	Pyrolysis method	Supercapacitors	[39]
Graphite rod	Submerged arc discharge in water	Neutral red dye adsorption	[40]

1.1. Novelty and Significance of the Work

Unlike conventional CNO synthesis techniques such as arc discharge, chemical vapor deposition (CVD), laser ablation, or high-temperature annealing, the proposed method operates under atmospheric-pressure conditions without requiring sophisticated vacuum systems, inert gas environments, or external catalytic reactors [41]. In the wick-pyrolysis process, the precursor is continuously supplied through capillary-driven wick transport, enabling controlled localized thermal decomposition and stable carbon generation near the flame zone. This creates a unique oxygen-limited pyrolytic environment favorable for graphitic shell formation while simultaneously minimizing complete combustion of the hydrocarbon precursor. Compared with conventional flame synthesis routes, the wick-pyrolysis approach offers improved precursor delivery control, localized carbonization conditions, energy efficiency, operational simplicity, and potential scalability for carbon nanomaterial production, as further shown in Table 2. Therefore, this work introduces wick pyrolysis as an alternative and sustainable route for the controlled synthesis of graphitic carbon nano-onions for energy-storage applications [42].

Copper was specifically selected because of its low carbon solubility and weak carbide-forming tendency, which promote surface-mediated carbon growth rather than bulk carbon diffusion-precipitation mechanisms typically observed with Fe, Ni, or Co catalysts [43]. This enables the formation of highly curved concentric graphitic shells characteristic of carbon nano-onions (CNOs). For instance, the silver substrate results in the formation of amorphous carbon [44].

Table 2. Comparison of different aspects of dairy-based or lipid-based precursors for the synthesis of carbon nanoparticles.

Reference	Primary Method	Collector Material & Distance	Special Process Features	Atmosphere	Product Type	Post-Treatment/ Purification	Capacitance (F/g)
[26]	Lamp combustion, wind-free, soot on Cu plate.	Copper plate, 2.5 cm above flame.	Post-anneal at 300 °C under Ar; nitric acid reflux for functionalization.	Ambient air combustion, then Ar during annealing.	CNOs with surface oxygen groups after acid treatment	Ethanol wash by centrifuge, anneal the sample, nitric acid reflux to filtration after the water/IPA wash and sample was vacuum dried.	-
[27]	Flame pyrolysis with cotton wick, soot on glass beaker.	Inverted glass beaker, fixed distance from wick.	Anneal at 500 °C, 2 h, controlled heating rate for CNO formation.	Ambient air combustion, furnace anneal likely under controlled atmosphere.	CNOs used as catalyst	Annealing (500 °C) after soot collection.	-
[28]	Flame pyrolysis with acoustic modulation option.	Nickel plate, 0.5 mm above flame.	Silver lamp + Ni collector (catalytic); optional 432 Hz acoustic modulation.	Ambient air combustion with acoustic excitation.	Soot/CNO-type carbon with possible catalytic influence of Ag/Ni and sound.	Collection at 30 min intervals; focus on modulated vs. unmodulated samples (no detailed chemical post-treatment described in snippet).	158
[29]	Wick pyrolysis in oil lamp, soot on glass beaker.	Inverted borosilicate glass beaker, directly above flame.	Emphasis on economical/energy-efficient wick pyrolysis; multiple solvent washes (hexane, acetonitrile)	Ambient air combustion.	-	Multiple washes with hexane and acetonitrile to remove unburnt residues.	-
[30]	CVD in inert environment.	CVD reactor surfaces (not lamp-based).	Uses oil as C source for MWCNTs (not CNOs) by CVD.	Inert gas (CVD).	MWCNTs from coconut oil.	-	-
[32]	Simple flame synthesis with lamp and cotton wick, soot on Cu foil.	Copper foil, 2 cm above flame.	Very simple, cost-effective flame route; mainly minimal workup for TEM and ink preparation.	Ambient air combustion.	CNOs and corresponding ink for electrochemical use	Ethanol dispersion, centrifuge; sonication to make stable ink	-

Table 2. Cont.

Reference	Primary Method	Collector Material & Distance	Special Process Features	Atmosphere	Product Type	Post-Treatment/ Purification	Capacitance (F/g)
[45]	Traditional wick-assisted pyrolytic flame synthesis using waste frying oil	Inverted glass beaker placed just above the flame to collect soot	Cotton wick-assisted continuous precursor feeding; catalyst-free synthesis; localized flame pyrolysis; sustainable waste-oil precursor	Stable air atmosphere	Carbon nano-onions (CNOs) and activated CNOs (a-CNOs) with graphitic multishell structure	Calcination at 900 °C for 1 h to remove amorphous carbon/unburnt oil and improve graphitization; further KOH activation (1:8 ratio) at 800 °C for 1 h under inert atmosphere; washed with 0.1 M acetic acid and DI water until neutral pH	Activated CNOs ~ 71; un activated CNOs ~30

1.2. Highlighting Key Potential Applications

This section highlights the potential applications of the synthesized carbon nanomaterials. Owing to their high surface area, graphitic structure, electrical conductivity, tunable surface chemistry, and porous morphology [46], the synthesized CNOs can be promising candidates for several advanced applications including supercapacitors, lithium/sodium-ion batteries [47], electrocatalysis, sensors, electromagnetic interference (EMI) shielding [48], adsorption, and conductive composite materials.

1.3. Short Review of Purification Processes of Carbon Nano-Onions

The purification of carbon nano-onions (CNOs) is a crucial step in their synthesis, aimed at enhancing their structural integrity and overall purity for various applications. Several conventional methods are employed in the purification process.

Figure 1 summarizes various purification processes of carbon particles. The acid treatment often uses strong acids, such as nitric acid, to dissolve metal catalysts and other unwanted inorganic materials. The acid refluxing technique enables the effective removal of these contaminants, although it may also alter the surface properties of CNOs [49]. High-temperature calcination involves heating the CNOs to high temperatures to eliminate amorphous carbon and other residues. The thermal treatment helps maintain the structural integrity of the CNOs, but precise temperature control is necessary to prevent structural damage. Filtration and ultracentrifugation are mechanical methods used to separate CNOs from larger impurities. Filtration can effectively remove particulates, while ultracentrifugation can be used to segregate particles based on size and density [50]. The use of organic solvents is another practical approach; here, the choice of solvent is crucial to ensure that CNOs retain their unique properties after purification [51].

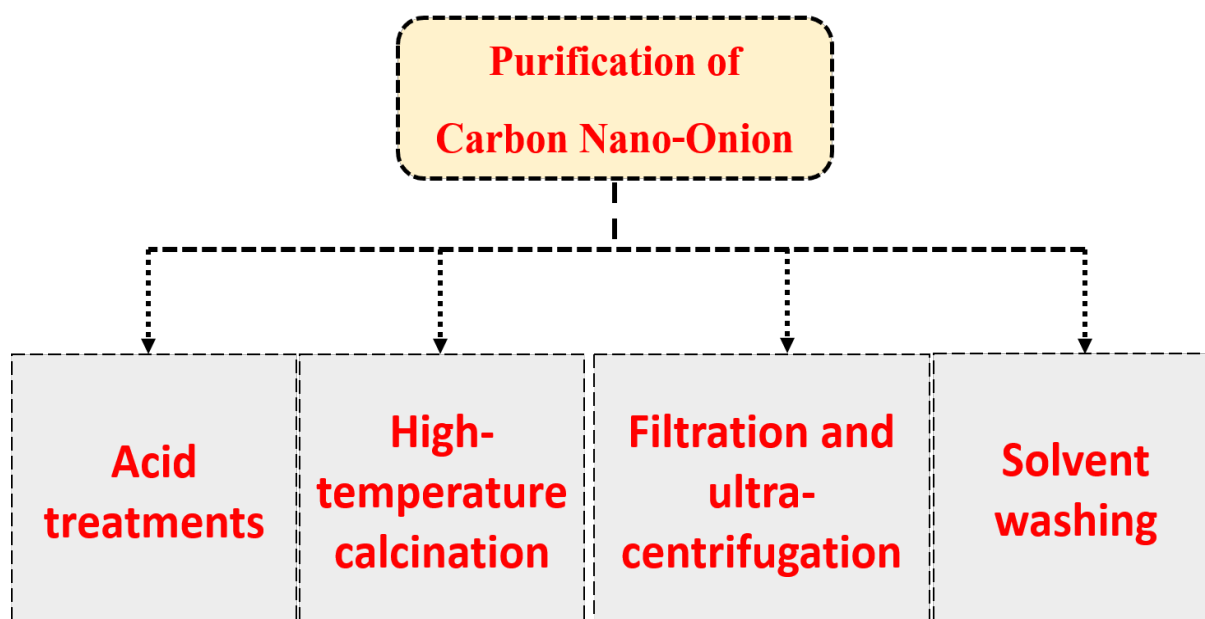


Figure 1. Purification techniques for carbon nanoparticles.

The solvent-assisted purification method uses mild solvents to remove impurities. This approach is characterized by low toxicity, rapid processing, and compatibility with other processing techniques. Among various purification methods, acetone purification stands out as a preferred choice as it helps retain the intrinsic structure and physicochemical properties of carbon nanomaterials (CNOs) [52]. It aims to preserve the unique properties of nanomaterials while ensuring high-quality outputs. Further, cost-effectiveness is an essential feature for large-scale production, as it is readily available and relatively inexpen-

sive compared to specialized solvents or complex acid treatments [4]. This approach has gained significant attention because it yields shorter processing times than more complicated methods, making it a safer choice for laboratory personnel and suitable for potential biomedical applications where toxicity is a concern [53].

The primary objective of the high-temperature calcination process is to enhance graphitization, which significantly improves electrical conductivity. This process reorganizes carbon nano-onions by transforming their carbon atoms from disordered, amorphous structures into highly ordered, graphitized forms, particularly under elevated temperatures and in the presence of catalysts. The structural evolution of carbon–nitrogen–oxygen (CNO) compounds occurs through two main mechanisms, leading to the formation of graphitic carbon within the samples. Initially, amorphous carbon spheres present a smooth surface and distinct microporosity. Previous studies have shown that temperature and catalyst pathways can retain oxygen and hydrogen functional groups, enhancing polarity while reducing conductivity [54,55]. This process facilitates elemental carbonization and fosters a gradual transition towards sp^2 graphitic domains. Under optimal conditions, the process drives graphitization, promoting a shift from disordered amorphous carbon to well-ordered sp^2 -bonded graphitic domains, suitable for various applications. Notably, amorphous carbon undergoes a transition from sp^3 to sp^2 hybridization at temperatures between 900 and 1100 °C, with variations in temperature affecting oxygen content and surface area. The introduction of metal catalysts further promotes effective graphitization, leading to the formation of mesoporous structures and increased carbon content while concurrently reducing oxygen and hydrogen levels as temperatures rise [1,56–58].

This article mainly focused on the insightful physicochemical characteristics of pristine CNO obtained from the combustion of ghee, coded as carbon nanoparticle (CNP), acetone-assisted purified carbon nanoparticle (PCNP), and pyrolyzed carbon nanoparticle (Pyro-CNP), and their applications in electric double-layer capacitors (EDLCs).

2. Experimental Section

2.1. Materials

The dairy product, ghee, was procured from Co-lite, a local manufacturer associated with the cooperative society in the Union Territory of Puducherry, Kurumampet, India, known for its commitment to quality and sustainability. High-purity copper sheet (Sigma-Aldrich, Bangalore, India) was cut into a square shape with 50 mm sides and used as a substrate for carbon particle deposition. A handmade cotton wick, skillfully crafted by a self-employed local artisan, was selected for its burning efficiency. The pyrolysis process was carried out inside a specially designed glass chamber that supports a substrate holder and sustains the flame, protected from the external wind. The substrate holder can be adjusted to different heights to optimize particle deposition yield. Further details about the design of the wick-pyrolysis technique are explained in the previous report [44].

2.2. Synthesis of CNO Particles and Purification

The as-prepared CNO particles synthesized through the wick-pyrolysis technique are deposited on a copper substrate kept at a distance of 5 cm from the tip of the flame for optimal yield and are termed as CNP. The CNO yield from 20 mL of the ghee precursor is 166 mg, calculated using the following formula.

$$\text{Carbon Yield(\%)} = \frac{\text{Amount of Carbon(g)}}{\text{Amount of Precursor(g)}} \times 100$$

Then, this pristine carbon is processed via acetone-assisted purification and pyrolysis under a nitrogen atmosphere, as depicted in Figure 2.

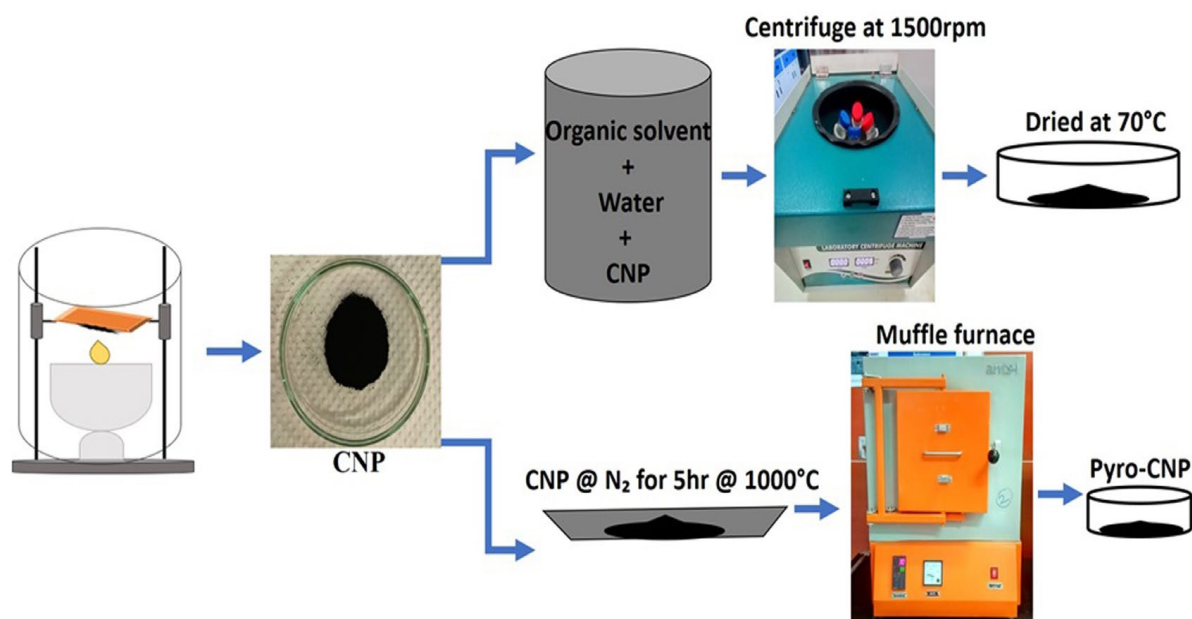


Figure 2. Schematic diagram of synthesizing, purification, and pyrolysis of CNO.

In a standard purification batch, 20 mg of the carbon nanoparticles were initially dispersed in a solvent mixture consisting of acetone and distilled water in a 1:3 ratio. This emulsion was then subjected to ultrasonication for 20 min at ambient temperature to facilitate uniform particle dispersion and the removal of contaminants. The solution was then centrifuged at 1500 rpm, allowing the purified carbon nanoparticles to settle to the bottom of the tube. The supernatant was carefully discarded, and the remaining nanoparticles were dried at 100 °C for 12 h. This drying process helped to remove any residual solvent and moisture, yielding purified nanoparticles. Alternatively, a separate batch of carbon nanoparticles was placed in an alumina crucible and heat-treated at 1000 °C for 5 h under a nitrogen atmosphere to preserve the desired properties of the carbon nanoparticles without oxidation. This comprehensive approach ensures the production of high-quality carbon nanoparticles suitable for various applications.

2.3. Characterization Techniques

The X-ray diffraction (XRD) pattern of the carbon samples was obtained using Cu-K α radiation (Empyrean 3rd Gen, Malvern PANalytical, 7602 EA Almelo, The Netherlands) in the 2θ range with a step size of 0.01 ° and a scanning rate of 1 °/min. The IR spectra were obtained using a Fourier-transform infrared spectrometer (FTIR) FT/IR-4700 type A (JASCO, Tokyo, Japan) in the wavenumber range of 400–4000 cm^{−1} by incorporating the sample into KBr. The Raman spectra were recorded using a Renishaw Raman spectrometer in the range 500–3000 cm^{−1}. The surface morphology of the carbon nanoparticles was obtained using a Carl Zeiss (GEMINI 300), (Oberkochen, Germany) scanning electron microscope. The CHN (carbon, hydrogen, and nitrogen) analysis of the sample was performed using the Elementar (Vario Micro Cube) instrument, (Langensfeld, Germany). The High-Resolution Transmission Electron Microscopy (HR-TEM) images and SAED patterns were obtained using the JEOL JEM 2100 instrument, (Tokyo, Japan). The XPS machine ESCA + Omicron nanotechnology (Oxford Instruments, Wiesbaden, Germany) was used to determine the elemental composition of the carbon nanoparticles. The surface area of the carbon nanoparticles was measured through 7-point Liquid N₂ adsorption and desorption isotherms obtained at a degassing temperature of 250 °C, using the Brunauer–Emmett–Teller (BET) Autosorb iQ instrument, (Boynton Beach, FL, USA). The contact angles

of substrates and carbon nanoparticles are measured using an Acam-MSI instrument, (Seoul, South Korea) with the tangent-fitting method.

2.4. Fabrication of EDLC

The electrochemical performance of the carbon nanoparticle (CNP), polycarbonate nanoparticle (PCNP), and pyrolyzed carbon nanoparticle (Pyro-CNP) samples was systematically evaluated using nickel foam as the current collector. The nickel foam, with a precise 10 mm diameter, underwent a thorough washing and drying process to eliminate any residual moisture that could affect performance. To create a conductive and stable electrode, the foam was meticulously coated with a specially prepared slurry. This slurry comprised 70% synthesized and functionalized carbon nanoparticles, 20% polyvinylidene fluoride (PVDF), a binder that maintains electrode structural integrity, and 10% Sp Carbon, which enhances electrical conductivity. The mixture was dried at 70 °C to ensure the complete evaporation of any solvents and to facilitate a secure bond between the components. For electrochemical testing, a 2 M potassium hydroxide (KOH) solution was used as the electrolyte in a two-electrode configuration. This setup enables a comprehensive assessment of electrochemical characteristics, including capacitance and charge–discharge behavior, which are critical for optimizing the performance of electrode materials in energy storage applications.

3. Results and Discussion

3.1. Structural and Functional Group Studies

The XRD diffraction pattern of CNP, PCNP, and Pyro-CNP samples is shown in Figure 3. The two prominent peaks, observed at 24° and 42°, are attributed to the (002) and (100) crystallographic planes. The broad peak corresponding to the (002) plane indicates the formation of CNO with poor graphitization and an amorphous-rich phase, as further confirmed by HR-TEM images. The Pyro-CNP sample shows an increase in the graphitic content in the CNO, as indicated by high-intense sharp peaks.

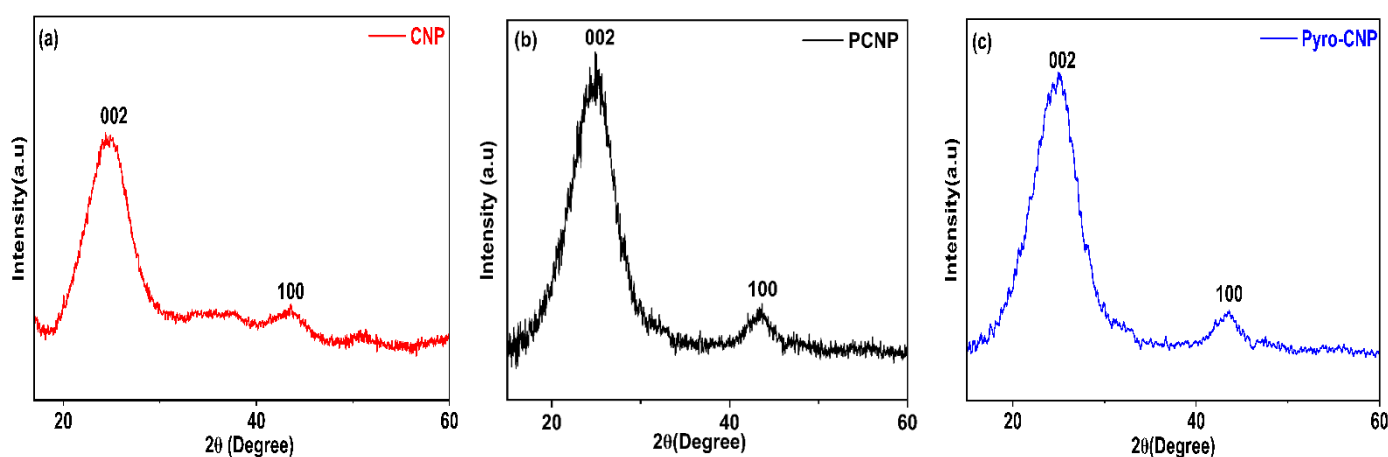


Figure 3. (a) X-ray diffraction pattern for synthesized CNO, (b) XRD pattern for purified CNO sample, (c) XRD pattern for pyrolyzed CNO sample.

3.2. FTIR and RAMAN Spectroscopy

FTIR spectra of as-prepared carbon nanoparticles, purified and pyrolysis samples are presented in Figure 4a–c. The IR-active peaks corresponding to O–H stretching were observed at 3444 cm^{-1} , 3400 cm^{-1} , and 3478 cm^{-1} , with varying broadness and sharpness. The strong bands corresponding to C=C stretching were observed at 1620 cm^{-1} , 1636 cm^{-1} , and 1632 cm^{-1} , attributed to an aromatic or alkene structure, indicating par-

tial graphitization of CNO. The C-H stretching vibrations observed at 2920 cm^{-1} and 2929 cm^{-1} were attributed to CNP and Pyro-CNP samples, respectively. On the other hand, the absence of this band for the PCNP sample is attributed to changes in the functional group due to the influence of acetone used during the washing process, which affected C-H and CO-H bonds. Additionally, CO-H stretching peaks are observed at 1384 cm^{-1} for the CNP sample and 1388 cm^{-1} for the Pyro-CNP sample. Thus, it is evident that the solvent-based purification process has altered the surface characteristics, leading to variation in the IR SPECTRA of the sample.

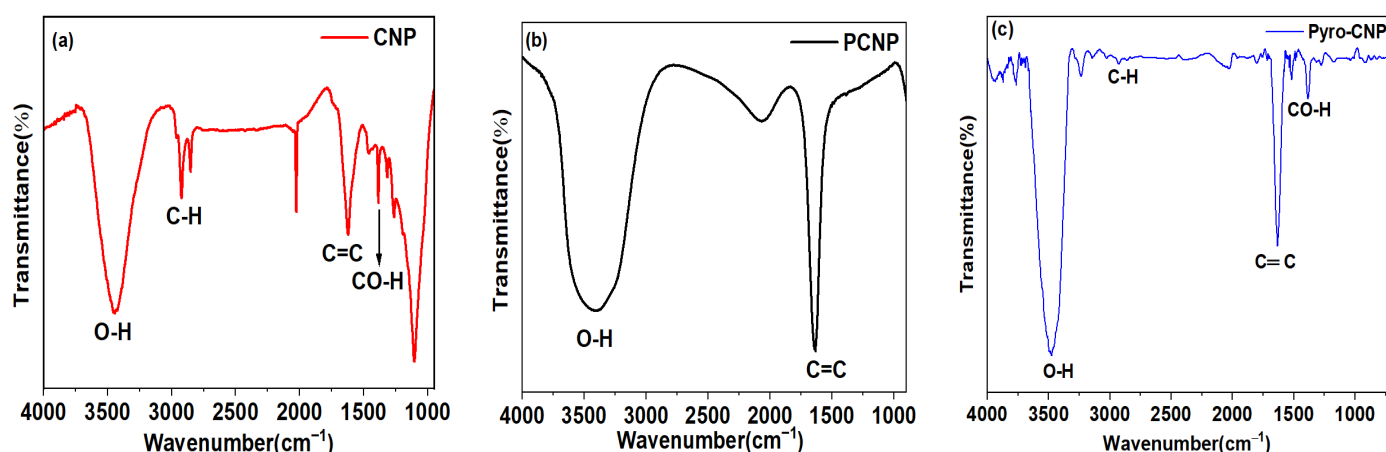


Figure 4. (a) FTIR Spectra for synthesized CNO, (b) FTIR graph for purified CNO sample, (c) FTIR graph for pyrolyzed CNO sample.

The Raman-active modes of the samples, CNP, purified and pyrolyzed samples, are illustrated in Figure 5. The prominent D band found in the range of 1343 cm^{-1} to 1349 cm^{-1} indicates the presence of disorder in sp^2 -hybridized carbon spheres. Additionally, the peaks observed at 1586 cm^{-1} , 1590 cm^{-1} , and 1591 cm^{-1} confirm the G band associated with sp^2 -hybridized carbon [59]. The broadness and sharpness of the D and G bands suggest a transition from the amorphous nature of the carbon particles toward enhanced graphitization. The intensity ratios of the D and G bands (I_D/I_G) for the samples CNP, PCNP, and Pyro-CNP are estimated to be 0.68, 0.89, and 0.95, respectively. This I_D/I_G ratio implies progressive graphitization of the defective, amorphous carbon forms into an ordered framework. The Raman-active modes corresponding to the 2D band are visible and distinct from the G band, appearing in the range $2640\text{--}2680\text{ cm}^{-1}$.

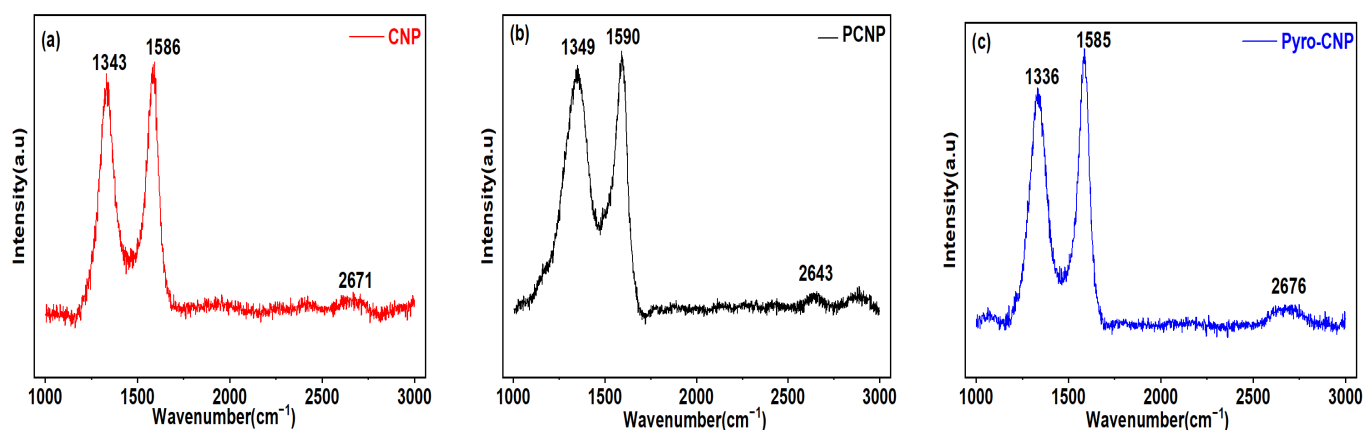


Figure 5. (a) Raman spectra for synthesized CNP, (b) Raman graph for purified CNP sample, (c) Raman graph for pyrolyzed CNP sample.

3.3. Surface Morphology Studies

The FE-SEM micrographs of the carbon particles deposited on the substrate are presented in Figure 6a. The surface micrographs show a uniform distribution of spherical particles with diameters ranging from 30 to 50 nm. Meanwhile, the purified carbon sample particles ranged from 40 to 80 nm, and the Pyro-CNP sample had agglomerated more, as illustrated by a particle size range of 50 to 250 nm in Figure 6c. The increase in particle size may be due to the purification technique and annealing temperatures, whereas the pyrolysis process agglomerated at 1000 °C, resulting in a significant increase in particle size and a lack of uniform distribution.

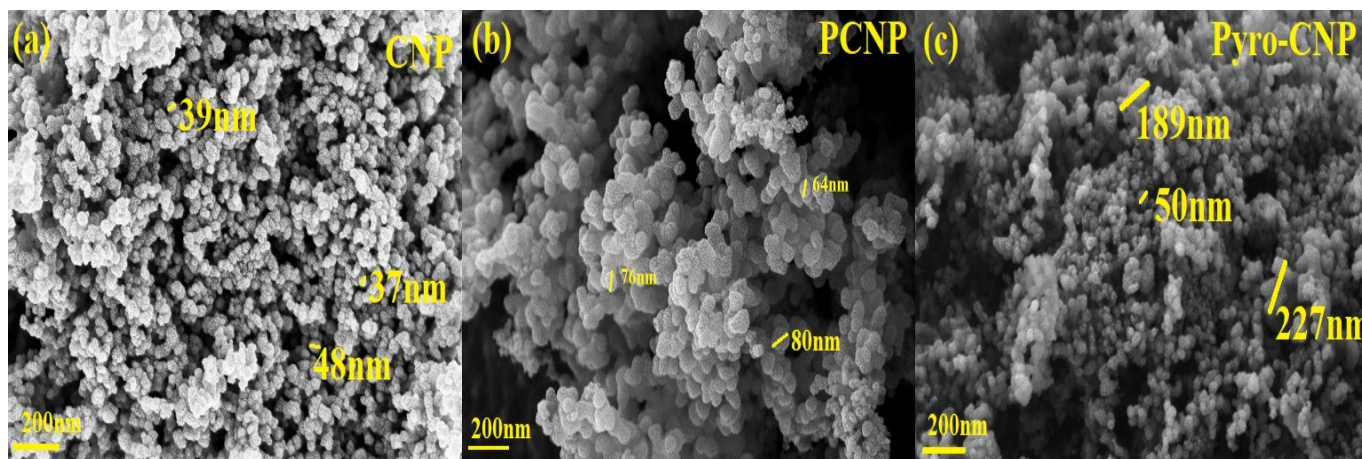


Figure 6. (a–c) shows FE-SEM images of the carbon nano-onion, (a) CNP, (b) purified CNO, and (c) pyrolyzed CNO.

3.4. Compositional Analysis

The EDS data obtained for the CNP, PCNP, and Pyro-CNP samples are presented in Table 3. The presence of 99.1% carbon in Pyro-CNP is attributed to a carbon-rich graphitic content, with a decrease in the oxygen element. On the other hand, the presence of 14.90% oxygen observed in the EDS data corresponding to PCNP suggests the possibility of oxygen bonding to the surface of CNO due to the use of acetone. The CHN analysis data presented in Table 4 indicate the absence of Nitrogen in all the samples. It is evident from both EDS and CHN analysis that the carbon percentage increases with pristine CNO, Pyro-CNP, and PCNP. The low purity of PCNP is attributed to the presence of hydrogen and oxygen on surface groups. On the other hand, although Pyro-CNP exhibits high carbon purity, trace elements and hydrogen remain on the particle surface. Comparison of EDS and CHN data indicates Pyro-CNP exhibits a carbon-rich phase among the two variants.

Table 3. EDS analysis data of carbon nano-onions.

Sample Name	C%	O%
CNP	97.08	2.92
PCNP	85.10	14.90
Pyro-CNP	99.1	0.9

Table 4. Elemental analysis data of clarified butter-carbon nano-onions.

Sample Code	C%	H%	N%
CNP	98.42	0.62	0
PCNP	87.74	1.39	0
Pyro-CNP	99.43	0.97	0

3.5. XPS Analysis

X-ray photoelectron spectroscopy (XPS) micrographs and binding energy data for the CNP variants shown in Figure 7a,c have revealed C1s, O1s, and survey peaks associated with the CNP and Pyro-CNP samples. The specific binding energy values for C1s in CNP and Pyro-CNP were recorded at 282.9 eV and 283.3 eV, respectively. Furthermore, a distinct O1s peak was observed at 532 eV in both samples. The binding energy values corresponding to the C1s data have revealed the presence of surface functional groups at 284.3 eV and 284.0 eV, associated with C–O, as well as at 286.8 eV and 286.2 eV, associated with C=O. Additionally, the O1s spectrum shows a characteristic peak at 532 eV, accompanied by three peaks at 531.45 eV, 532.64 eV, and 533.14 eV for the CNP sample, and at 531.7 eV, 532.72 eV, and 533.73 eV for the Pyro-CNP sample. In both cases, these peaks correspond to the C–O, C–OH, and C=O functional groups, respectively. In the case of PCNP, C–O at 284.7 eV for C1s and C=O at 533.3 eV for O1s are observed alone.

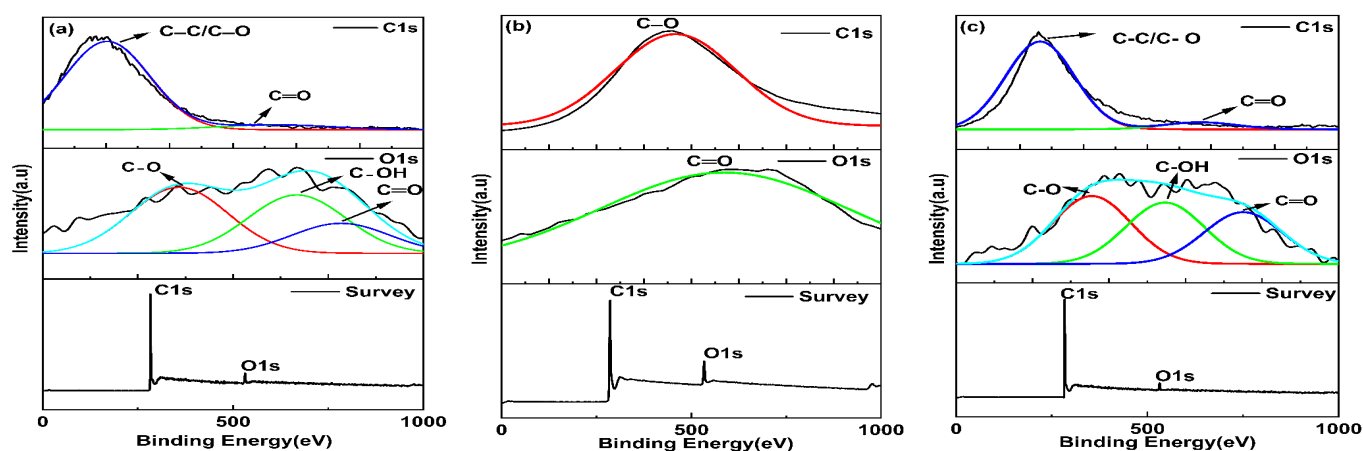


Figure 7. (a–c) XPS spectra for (a) CNP sample, (b) purified CNP, and (c) Pyro-CNP sample.

The carbon composition in the CNP sample is 94.32%, in the PCNP sample 90.14%, and in the Pyro-CNP sample 95.52%. Despite the similar functional groups in the pristine and Pyro-CNP samples, the latter shows a carbon-rich composition, as indicated by compositional analysis.

Although XPS analysis indicates that oxygen species remain after pyrolysis, this does not contradict the observed increase in hydrophobicity. XPS is an extremely surface-sensitive technique and can detect even small amounts of residual oxygen functionalities. However, after pyrolysis, the concentration and chemical nature of these oxygen groups are altered significantly. The remaining oxygen species associated with thermally stable groups (such as carbonyl or ether functionalities) do not contribute to hydrophilicity. In addition, pyrolysis promotes the formation of graphitic sp^2 carbon domains, which possess intrinsically lower surface energy and greater hydrophobicity. Increased graphitization, reduced defect-associated polar sites, and the development of surface roughness together contribute to enhanced hydrophobic behavior despite the presence of residual oxygen detected by XPS.

3.6. HR-TEM Analysis

The HR-transmission electron micrographs of the pristine and processed CNOs are presented in Figure 8a–d. The micrograph images indicate that the quasi-spherical particles have a size of about 30–40 nm. The onion-ring type morphology observed for CNP has exhibited an interlayer spacing of 0.37 nm, compared to 0.40 nm for PCNP. However, the Pyro-CNO micrographs show a higher degree of aggregation. The selected area electron diffraction (SAED) pattern shows the presence of the crystallographic planes (002) and (100).

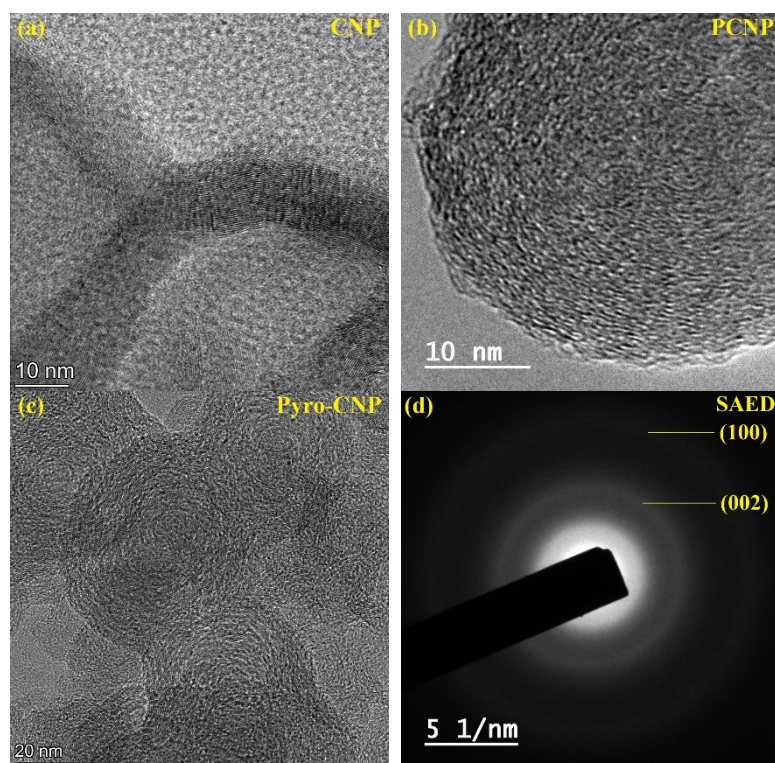


Figure 8. (a) HR-TEM images of carbon nanoparticle (OL) from copper substrate, (b) purified CNP, (c) pyrolyzed CNP, and (d) indexed SAED pattern for CNP.

3.7. Physical Properties of Nanocarbon Structures

The Brunauer–Emmett–Teller (BET) adsorption and desorption isotherms for the CNP, PCNP, and Pyro-CNP samples are shown in Figure 9. The surface area of the CNP, PCNP, and Pyro-CNP samples was estimated to be $79.795 \text{ m}^2/\text{g}$, $152.52 \text{ m}^2/\text{g}$, and $261.78 \text{ m}^2/\text{g}$, respectively, as indicated in Table 5. The isotherms exhibit a type-IV pattern at higher relative pressure, indicating the presence of mesopores. Thus, it is evident that the surface area of purified carbon samples has increased compared to that of the CNP sample, as shown in Figure 8, due to acetone chemisorption and annealing at higher temperatures [20]. Unlike the trend toward increased surface area in CNP, PCNP, and Pyro-CNP samples, a significant change in pore volume is observed in PCNP, which differs from comparable values for the other two variants. The pyrolysis process has led to increased graphitization and a gradual increase in surface area. However, the lower pore volume corresponding to Pyro-CNP is attributed to the agglomeration of particles.

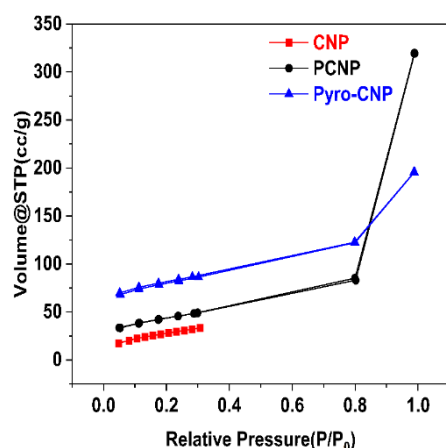


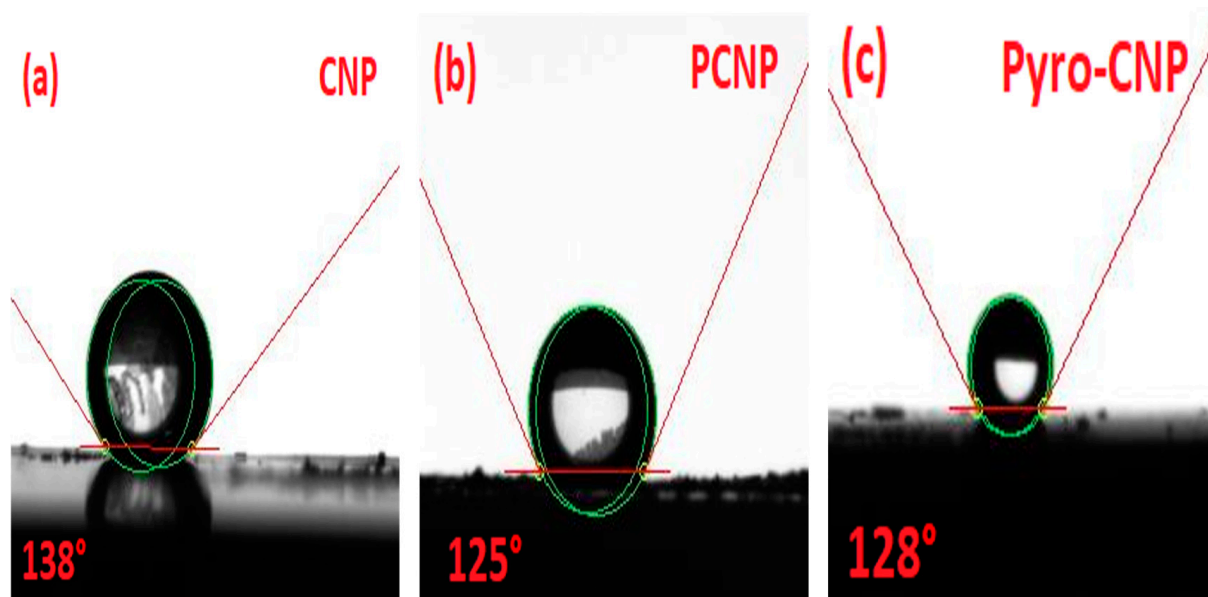
Figure 9. Brunauer–Emmett–Teller graph for carbon nanoparticles.

Table 5. Comparison of the BET results for carbon particles and the purified sample.

Sample Name	Surface Area (m ² /g)	Pore Radius (Å)	Pore Volume (cc/g)
CNP	79.795	66.6	0.26
PCNP	152.52	64.7	0.49
Pyro-CNP	261.78	23.11	0.30

3.8. Contact Angle Measurement

The contact angle of a de-ionized water droplet on a thin layer of carbon deposition was observed at $t > 250$ ms with a volume of 10 μ L. The contact angle of a water droplet on the bare copper substrate is estimated to be less than 90° , indicating the substrate's hydrophilic nature. Figure 10a shows the contact angle of a water droplet on pristine CNP at 138° . Similarly, the PCNP and Pyro-CNP have exhibited contact angles of 125° and 128° , respectively, as shown in Figure 10b,c. These values reflect the hydrophobic nature of the CNP materials and the lower contact angles associated with the samples' high surface area.

**Figure 10.** (a–c) are the contact angle images of the water droplet on the CNP-coated substrates.

3.9. Electrochemical Studies

Electrochemical studies using two-electrode configurations were conducted using electrochemical impedance spectroscopy (EIS), cyclic voltammetry (CV), and galvanostatic charge–discharge (GCD) on all three samples.

Figure 11a–c show the Nyquist plot from the electrochemical impedance spectroscopy (EIS) data at the start and after 5000 cycles of galvanostatic charge–discharge (GCD). Fitting the Nyquist data to an equivalent circuit indicates an increase in interfacial resistance, which paradoxically enhances ion-transfer efficiency. The use of modified carbon materials improves interfacial contact, leading to greater capacitance and better performance during long-term cycling tests. The semicircle in the high-frequency region reflects charge-transfer resistance, while the linear trend at lower frequencies indicates diffusion-controlled electron and ion transport, as detailed in Table 6.

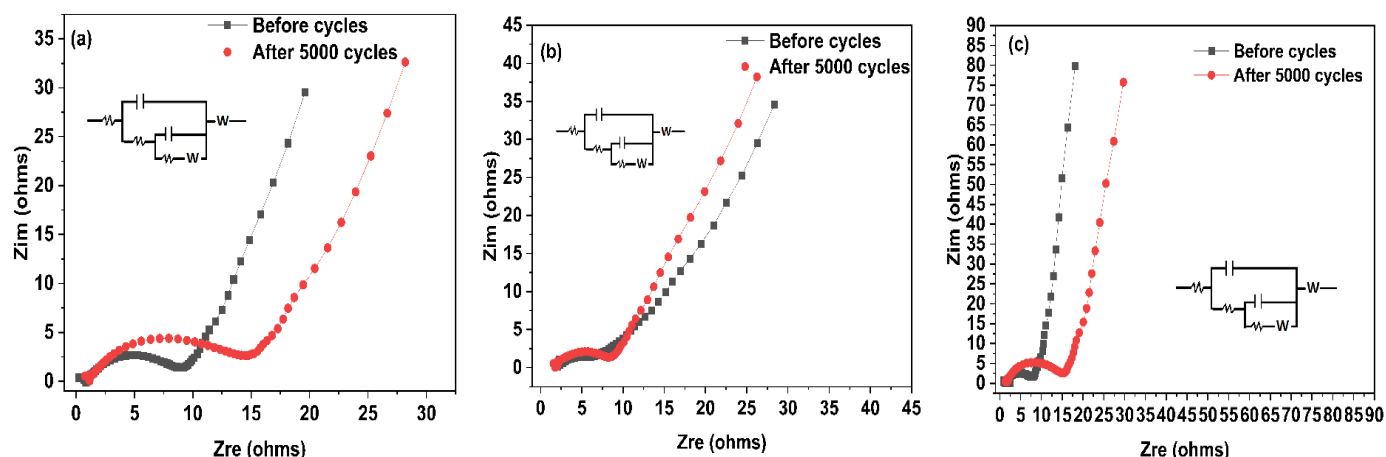


Figure 11. Electrochemical studies: (a) EIS plot of CNP, (b) EIS plot of PCNP, (c) EIS plot of Pyro-CNP.

Table 6. Characteristic values of supercapacitors.

Sample	Initial Interfacial Resistance (Ω)	After the 2000 Cycle, Interfacial Resistance (Ω)	Specific Capacitance (F/g)	Power Density (W/Kg)
CNP	9	14.6	83.26	661.1
PCNP	5.8	8.3	109.7	925.5
Pyro-CNP	7.7	15.2	75.2	2592

Cyclic voltammetry was performed over a voltage range of -0.5 to 0.5 V at scan rates from 1 to 100 mV, demonstrating symmetric supercapacitor behavior, as evidenced by the square-shaped CV curves across different areas. Figure 12 illustrates a broader range of areas at various sweep rates, showing near-rectangular cyclic voltammograms. The consistent curves at a higher scan rate of 100 mV highlight the excellent stability and storage mechanism of the electrode. As the scan rate increases, the reduction in specific capacitance is attributed to the limited time available for ions to navigate and occupy the available spaces within the active material. This results in restricted interactions occurring primarily on the outer surfaces. Consequently, at higher scan rates, some surface areas may become less effective for charge storage. The capacitance calculated for the electrostatic mechanism of the non-Faradaic process in symmetric electric double-layer capacitors (EDLCs) is displayed in Table 7.

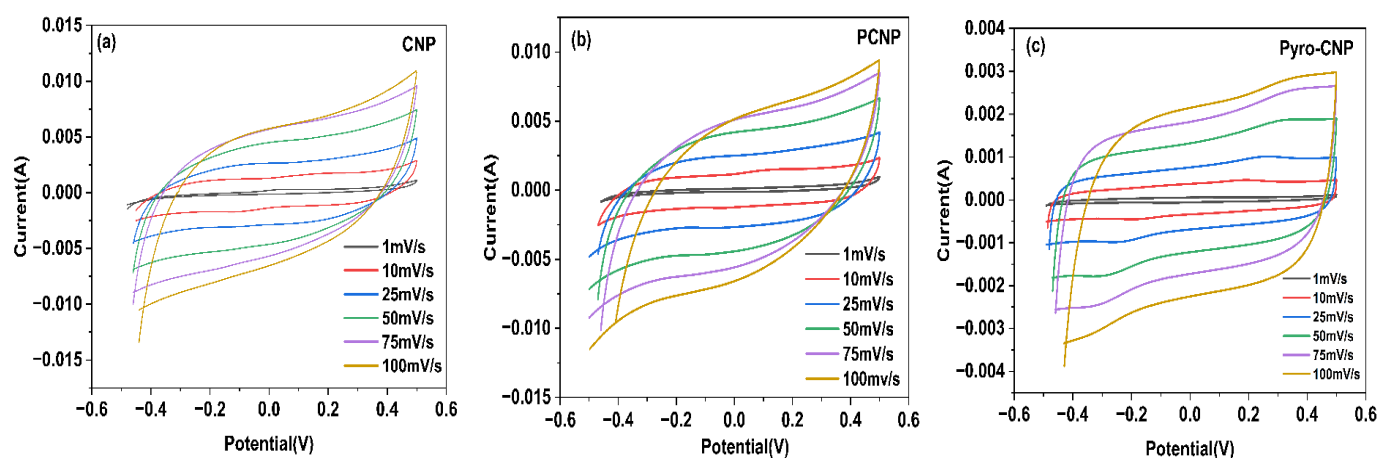
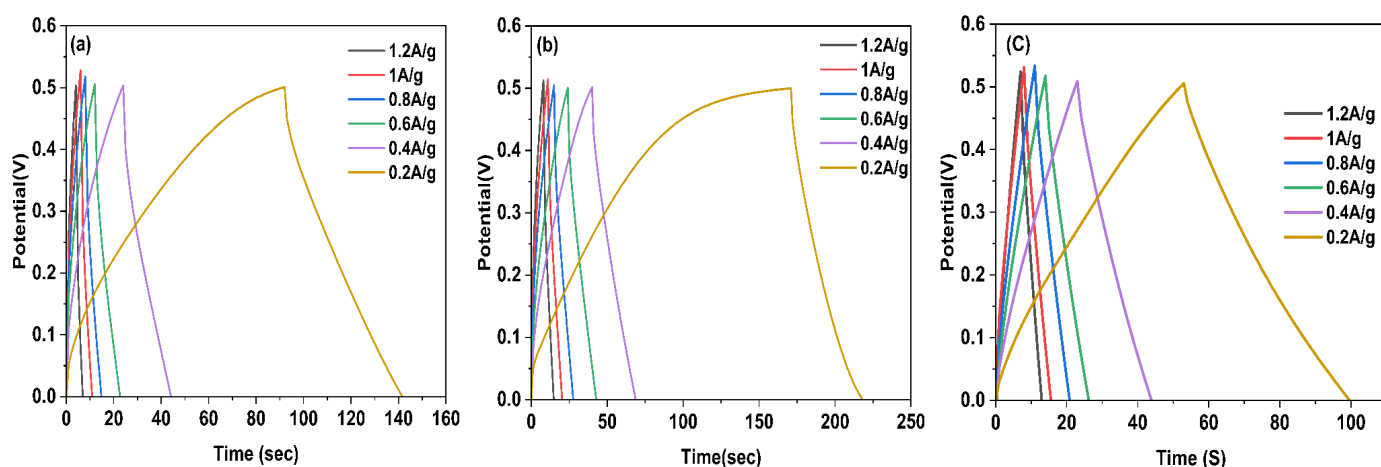


Figure 12. CV for different scan rates: (a) CNP, (b) PCNP and (c) Pyro-CNP.

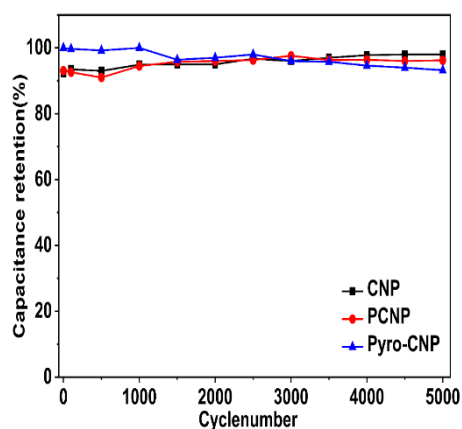
Table 7. The specific capacitance from cyclic voltammetry.

Sweep Rate of Cyclic Voltammetry	CNP (F/g)	PCNP (F/g)	Pyro-CNP (F/g)
100 mV	10.4	12.8	13.3
50 mV	15.0	19.3	17.5
10 mV	24.8	30.9	25.4
1 mV	24.7	33.6	34.4

The GCD profiles of EDLC utilizing CNP, PCNP, and Pyro-CNP electrodes corresponding to various densities between 0.2 and 1.2 A/g are depicted in Figure 13. The symmetric triangular profiles with a long discharging time indicate supercapacitive behavior. Table 6 presents the capacitance values of the EDLC at different current densities. The maximum values of specific capacitance of the EDLC observed at a current density of 1.2 A/g are estimated as 29.3 F/g, 67.27 F/g, and 57.7 F/g for CNP, PCNP, and Pyro-CNP, respectively.

**Figure 13.** GCD for different current densities: (a) CNP, (b) PCNP and (c) Pyro-CNP.

Capacitance retention is a crucial indicator of the cycle life and reliability of supercapacitors. A laboratory prototype typically achieves over 90% capacitance retention after extensive cycling, indicating advancements in material design and electrochemical engineering. The capacitance retention over 5000 cycles is shown in Figure 14. The CNP and PCNP supercapacitors exhibited a gradual increase in capacitance and maintained a strong retention of 96% from the 1000th cycle to the end of 5000 cycles. Thus, the CNP and PCNP electrodes demonstrated good cycling stability throughout the testing period.

**Figure 14.** Retention graph of CNPs.

On the other hand, the Pyro-CNP supercapacitors' initial retention declined to 94% over 5000 cycles. In summary, comparing the performance of EDLC using the same electrolytes and working potential has ensured consistency. The capacitance for the CNP and PCNP supercapacitors remained stable, exceeding 96%.

The Ragone plot illustrates the relationship between energy density and power density for the two electrode systems, providing insight into their energy-storage capability and rate performance. From Figure 15, the energy density decreases gradually with increasing power density, which is a typical characteristic of supercapacitor materials due to limited ion diffusion and reduced charge storage time at higher current densities. Among the three samples, Pyro-CNP exhibits the highest electrochemical performance, delivering a maximum energy density of approximately 33.84 Wh kg^{-1} at a power density of $\sim 2592 \text{ W kg}^{-1}$ and retaining $\sim 25.92 \text{ Wh kg}^{-1}$ even at a high-power density of $\sim 15,552 \text{ W kg}^{-1}$. This indicates superior charge storage capability and excellent rate handling performance, which can be attributed to the highly graphitized multishell structure, improved electrical conductivity, and efficient ion transport pathways provided by the hollow polyhedral carbon nano-onion architecture. PCNP demonstrates intermediate performance, showing higher energy density than CNP across the entire power density range. The improved behavior suggests enhanced electrochemical accessibility and better ion/electron transport kinetics compared to CNP. CNP shows the lowest energy density values, decreasing from approximately 9.37 Wh kg^{-1} to 3.30 Wh kg^{-1} with increasing power density. The comparatively lower performance may arise from reduced graphitic ordering, lower accessible surface area, or higher internal resistance.

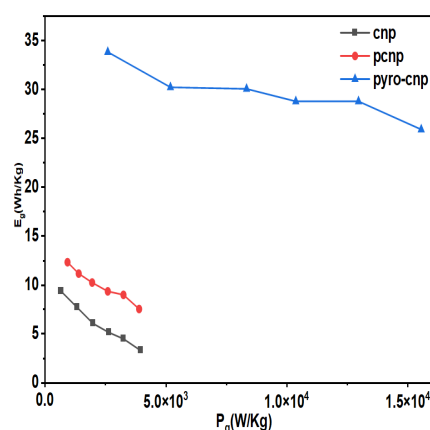


Figure 15. Ragone plot for CNPs.

Overall, the Ragone analysis confirms that Pyro-CNP possesses the best balance between energy density and power density, indicating its suitability for high-performance energy storage applications requiring both rapid charge–discharge capability and high energy retention.

4. Conclusions

Carbon nanoparticles synthesized via wick pyrolysis of ghee on a copper substrate have yielded hydrophobic carbon nano-onions (CNOs). The CNOs thus obtained were purified using acetone as the organic medium, pyrolyzed under a N_2 atmosphere at 1000°C , and their physicochemical characteristics were investigated. Post-treatment of pristine CNOs has resulted in a carbon-rich phase and significant increases in surface area and pore volume. The solvent treatment has played a substantial role in enhancing the graphitic nature of carbon, as indicated by XRD, TEM, and XPS studies. Although high-temperature pyrolysis favors the formation of carbon-rich CNOs relative to other pyrolysis conditions,

agglomeration results in a lower pore volume. These comparisons of physicochemical properties are demonstrated by the performance of EDLCs characterized by Electrochemical Impedance spectroscopy, Cyclic Voltammetry, and Galvanostatic charge-discharge profiles. The EDLC using CNOs treated with an organic solvent has exhibited a specific capacitance of 109 F/g over 5000 cycles. Thus, this study demonstrates the synthesis of CNOs at ambient conditions, promising for energy storage applications.

Author Contributions: A.S.: Conceptualization, Methodology, Investigation, Data curation, Writing—original draft; A.B.P.: Methodology, Investigation, Formal analysis, Validation; C.N.S.K.: Supervision, Project administration, Resources; A.B.: Supervision, Project administration, Resources, Conceptualization, Writing—reviewing and editing draft. All authors have read and agreed to the published version of the manuscript.

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