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Lithium-Ion Battery Anodes Based on Graphite: Performance Enhancement Study Using Carbon Black, Carbon Nanotubes, and Other Additives

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ABSTRACT

This research aims to explore the potential of carbon soot, a byproduct of diesel fuel combustion, as a sustainable and cost-effective anode material for lithium-ion batteries. While graphite remains a standard, its limitations in terms of capacity and rate capability drive the pursuit of alternative materials. Initial experiments with carbon soot revealed subpar electrochemical performance compared to pristine graphite. This prompted the investigation of nanomaterial integration to enhance the electrical conductivity and charge storage capacity of the anode. Carbon black (CB) and carbon nanotubes (CNTs) were selected for their exceptional electronic properties and ability to create efficient charge transport networks. A series of anode materials were fabricated using various combinations of graphite, carbon soot, CB, and CNTs. Electrochemical characterization, including cyclic voltammetry and electrochemical impedance spectroscopy, was employed to assess the performance of these materials. The results demonstrated a significant improvement in the specific capacity, rate capability, and cycle life of the anodes incorporating nanomaterials. The synergistic effect of CB and CNTs in enhancing the electronic conductivity and providing additional active sites for lithium storage was particularly evident. This study highlights the potential of carbon soot as a viable anode material for lithium-ion batteries, provided it is subjected to appropriate treatments and combined with suitable nanomaterials. Further research is warranted to optimize the synthesis and processing of carbon soot-based anodes, as well as to explore the integration of other promising nanomaterials and innovative electrode architectures.

1. INTRODUCTION

The most widely used power generating and storage technology now is the lithium-ion battery (LIB), which is superior in terms of cost, safety, energy density, and power density. The most widely used power generating and storage technology now is the lithium-ion battery (LIB), which is superior in terms of cost, safety, energy density, and power density.

LIBs are thought to be a clean energy source since they store electricity in chemicals and use electrochemical reactions to transform chemical energy into electricity [1-3].

Because of their high energy and power densities, lithium-ion batteries (LIBs) can power electric cars as well as portable electronics like laptops, tablets, and phones. This effectively lowers the amount of fossil fuels used and greenhouse gas emissions [4, 5]. Moreover, LIBs' high-energy conversion rate makes it possible for them to be used in electrical grid applications, which facilitates the effective storage of energy obtained from renewable sources including geothermal, solar, and wind energy [6]. It is anticipated that LIBs will always be a part of our daily lives.

Lithium-ion batteries (LIBs) have revolutionized energy storage, powering devices from smartphones to electric vehicles. Graphite, a traditional anode material, offers stability and low cost but limits energy density. To address this, researchers explore alternative materials like silicon, tin, and metal oxides. Silicon, for instance, boasts ten times the theoretical capacity of graphite, but its significant volume expansion during cycling poses challenges. Tin-based anodes also offer high capacity but suffer from similar volume changes. Metal oxides, while stable, face limitations in electronic conductivity and ion diffusion. Carbon-based nanomaterials like CNTs and graphene, with their excellent conductivity and large surface areas, emerge as promising candidates. The future of LIBs lies in developing advanced anodes that balance high energy density, fast charging, long cycle life, and safety. By addressing the challenges of existing materials and exploring innovative approaches, researchers aim to push the boundaries of battery technology and enable the widespread adoption of electric vehicles and renewable energy storage systems [7, 8].

A typical LIB is an anode/separator/electrolyte/two current collectors composite structure. Examples of cathode materials are $LiCoO_2$ (LCO), $LiMn_2O_4$ (LMO), $LiFePO_4$ (LFP), and $LiNiMnCoO_2$ (NMC) Graphite is often used today as an anode material; more recently graphite enriched with SiO_x is used [9].

Li-ions that have been stored at the anode migrate to the cathode during discharging, producing electrons and creating a current flow. When charging the battery, the procedure is reversed. The conductive pathway for the transport of Li-ions in electrolytes is made up of lithium salts, such as LiPF6, LiBF4, and LiClO4, in various organic solvents, such as propylene carbonate (PC), ethylene carbonate (EC), ethyl methyl carbonate (EMC), dimethyl carbonate (DMC), and diethyl carbonate (DEC) [10].

To avoid direct contact between the cathode and anode, separators—typically microporous layers made of non-woven fabric mats or polymeric membranes—are positioned between them [11]. Electrical current produced at the electrodes is collected by current collectors, which are bridge components that link to external circuits. Al and Cu foils are used as commercial current collectors for cathodes and anodes, respectively [12].

Several investigations have shown that the carbon layer including amorphous carbon, graphene, graphene oxide, carbon nano tub, and carbon black improves the electronic conductivity and hinders side reaction because of its barrier ability against electrolytes [13-16]. In general, the thin carbon layer is characterized by high electronic conductivity as well as protection of the structure from deformation due to the elution of transition metal ions. On this basis, carbon layer and CNTs are found to be the best fit for the improvement of electrochemical Anod electrode performances of Lithium-ion batteries [17]. They offer electronic conductivity and inhibit side reactions by acting as a good physical/chemical barrier to the electrolyte [13-16]. In general, a thin carbon layer not only offers excellent electrical conductivity but also protects against structural deformation caused by transition metal ion elution. Based on them, the carbon layer and CNTs are excellent candidates to enhance the electrochemical Anod electrode performances of Lithium-ion batteries [17]. Some of the common techniques to deposit carbon layer (CB or CNTs) includes mechanical alloying, chemical vapor deposition (CVD), pyrolysis of adsorbed organic species as well as thermal cracking [18-20]. But all these methods are only applicable for a laboratory scale because they are time-consuming and organizing require complex systems and expensive apparatus.

The graphite-like crystallites in CB have random orientation and the onset of the graphite basal plane spacing is random. A suitable concentration of CB in the anode can enhance the number of active sites and facilitate Li-ion penetrated into graphite from multi-directions. The efficiency is the same as that of a funnel. From an energy consideration it may be said that the intercalation of CB significantly lower the energy for the transport of Li into the graphite lattice [21].

Specifically, combustion soot is a much more heterogeneous material which in most cases contains a large (>50% w/w) organic carbon component and exhibit higher ash and extractable organic matter concentrations than carbon black. The characteristics as a chemical compound and a physical one depend greatly on its source. While some soot types may appear in certain morphologies including diesel exhaust particulate (DEP) that groups acini form carbon black aggregates, other soots may drastically differ in their morphologies [22].

CB has a unique particle shape, which is formed in its acini-form (grape-like) aggregates located in large-sized agglomerates of highly fused spherical primary particles [23].

Carbon black is often used as a conductive additive in lithium-ion battery anodes, enhancing conductivity and improving overall performance. In lithium-ion batteries, carbon black is mixed with active materials to ensure efficient electron transport, contributing to better charge and discharge rates [24, 25]. The use of diesel generators raises environmental concerns, especially in terms of emissions. Transitioning to cleaner energy sources for production could improve the sustainability of carbon black manufacturing and, by extension, lithium-ion battery production when the conversion process to convert carbon soot emissions into carbon black is suitable for batteries application [26, 27].

In the present study, first, we used carbon black (soot emissions) as a carbon source to anode graphite base LIB through a mixing-compression method to get coin belt samples [28-30]. Second, we investigate the effect of carbon black and carbon Nano tubs (CNTs) with graphite (commercial powders) in weight ratios using mixing-coating--compressing method to get belts technique then fabricate a half-cell of LIB to investigate some electrical properties to evaluate half-cell performances due to prepared anode electrodes [30, 31].

Compared to graphite-carbon anodes, silicon-based anodes have a substantially greater theoretical specific capacity, which could result in a higher energy density in lithium-ion batteries. Longer battery life and quicker charge times may result from this. However, during lithiation, silicon anodes experience high volumetric expansion, which results in mechanical stress and quick capacity deterioration. On the other hand, because of their well-established production procedures and reduced volumetric expansion, graphite-carbon anodes have exceptional cycle life and stability. Despite having a lower theoretical capacity than silicon, they are currently the industry standard for lithium-ion batteries due to their overall performance and commercial maturity [32, 33].

The influence of the dispersion process of additives Carbon (soot) and (CB-CNTs) -Graphite and preparation method on the properties of lithium-ion battery anode is investigated. First, prepare the graphite -soot carbon as belt or disk half cell model and secondly as thin film from graphite-CB-CNTs - binder composite coated on copper foil as half-cell anode model.

2. EXPERIMENTAL PART

2.1 Materials and chemicals

In this study, carbon black (soot) used as product from deiseal generator type Perkins used in Baghdad (MTU) site. Table 1 and Figure 1 show a photo and technical specification of the deiseal generator. Table 2 and Figure 2 show the properties of diesel fuel used for the generator and chemical composition of carbon soot product using X ray florescence technique.

Figure 1. Perkins deiseal generator

Table 1. Diesel fuel properties tests results

	Test(Unit)	Value
1	Flash point (°C)	54
2	Cloud point (°C)	40
3	Density (g/L)	850
4	Pour point (°C)	27
5	Viscosity cst at 40 (°C)	2.92
6	Carbon Residue (%)	0.0002
7	Sulphated ash(%)	60.5
8	Water ratio%	0.0

Table 2. Engin technical datasheet

	Part- Property	Data
1	Engine Make	Perkins
2	Engine Model	500KVA-50HZ
3	Governing Type	Electronic
4	Number of Cylinder	6
5	Cylinder Arrangement	Vertical in line
6	Bores and Stroke mm	137×171
7	Displacement - Cubic Capacity litres	15.2
	Induction System	Turbocharged and air-air charge cooled
8	Cycle	4 stroke
9	Combustion System	Direct Injection
10	Compression ratio	16:1
11	Rotation	Anti-clockwise, viewed on flywheel
12	Cooling System	Water-cooled
13	Frequency and Engine Speed	50 Hz, 1500 rpm
14	Total Lubrication System Capacity litres	62
15	Exhaust Temperature °C	500°
16	Total Lubrication system litres	62
17	Total coolant capacity litres	58

Commercial graphite and nanocarbon powder type (USA, MERCK) with purity 99.99% was selected as the anode battery conductive matrix with graphite for anode of Li-ion half cell model. Carbon nanotube (CNTs) powder specifications are highlighted below in Table 3. The unique properties of Graphite include: excellent modality of properties such as hardness, chemical insensibility and high heat distortion temperature and thermal stability. Also, commercial materials used in this study, lithium cobalt oxide and lithium carbonate type (USA, MERCK) powder with purity 99.99% as a source of Li ion and electrolyte solution .

X -ray fluorescence used to determine chemical composition of black powder soot as in Figure 2 which produced from gases emissions from diesel generator and accumulated in chimney tube. Deiseal fuel chemical characterization also obtained carbon ash ratio in percentage ratio. Table 3 shows the chemical and physical properties of CNTs powder such as diameter of carbon nano tubes in nano scale (60-80) used an additives to graphite base material of the anode electrode.

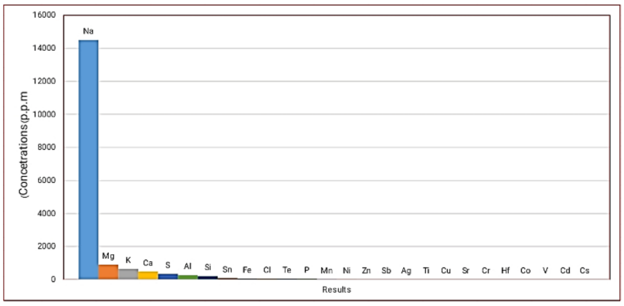

Figure 2. Carbon (soot) analysis using XRF technique

Table 3. Carbon nanotubes powder properties

Property	Unit	Value
Tube diameter (d)	Nm	60---80
Tube Length	µ m	10---15
BET Nitrogen surface	m^2 g	55---75
Adsorption Value	ml ⁄ 100g	500---550
Density	Kg ⁄ m^{li}	120
Volume Resistivity	Ohm,cm	2—5 × 10^{-4}
Moisture	%	0.1---0.3
Ash Content	%	0.3 max
Ni	%	0
PH		8—9
Appearance		Black Powder

2.2 Material test characteristics

Crystallinity and phase diagram with specific surface area where established to graphite and carbon black powder using x-ray diffraction method as shown in Figure 3. X-ray diffraction (XRD) analysis using monochromatic Cu Kα radiation and Bruker D8 Advance geometry was performed to investigate the crystalline composition of the thin films. The average size of the thin film crystallites was determined using single and multiple line analyses from the peak broadening. In the field of nanocomposites and energy storage, significant progress has been made for large-scale applications.

Figure 3 shows the graphite and carbon black exhibit different properties according to X-ray diffraction (XRD) study. With noticeable peaks at particular angles that correspond to the Miller indices (002) at roughly 2θ = 26.5°, (004) at roughly 2θ = 54°, and (006) around 2θ = 78°, graphite has a clearly defined crystalline structure. The ordered layers of the basal planes of graphite are indicated by these peaks. Carbon black, on the other hand, is less crystalline and shows broad peaks, particularly around 2θ = 25°, which reflect tiny crystallites that resemble graphite. Because of the loose packing of its tiny grains, carbon black's XRD pattern also exhibits small-angle scattering, indicating heterogeneity. Other peaks, such as (004) and (006), are less evident than the (002) peak for graphite-like crystallites, indicating the disordered structure of carbon black. Overall, the XRD analysis underscores the more ordered crystalline structure of graphite compared to the disordered, small-crystallite structure of carbon black.

A smaller FWHM indicates fewer defects and greater crystallinity. Graphite High crystallinity makes it suitable for applications requiring good electrical conductivity and thermal properties, like batteries and lubricants while carbon black displays broader and less defined peaks, suggesting an amorphous or partially graphitic structure. Peaks may appear around 22° and 43°, but they are generally much broader compared to those of Graphite. Carbon Black show lower peak intensities overall, with significant background noise due to the amorphous content. The broad peak shapes indicate a distribution of particle sizes and orientations. Carbon Black generally exhibits smaller crystallite sizes, often in the range of tens of nanometres. The broader peaks indicate more defects and lower crystallinity contributes to different applications, such as reinforcing agents in rubber and plastics, where high surface area and adsorption properties are desirable.

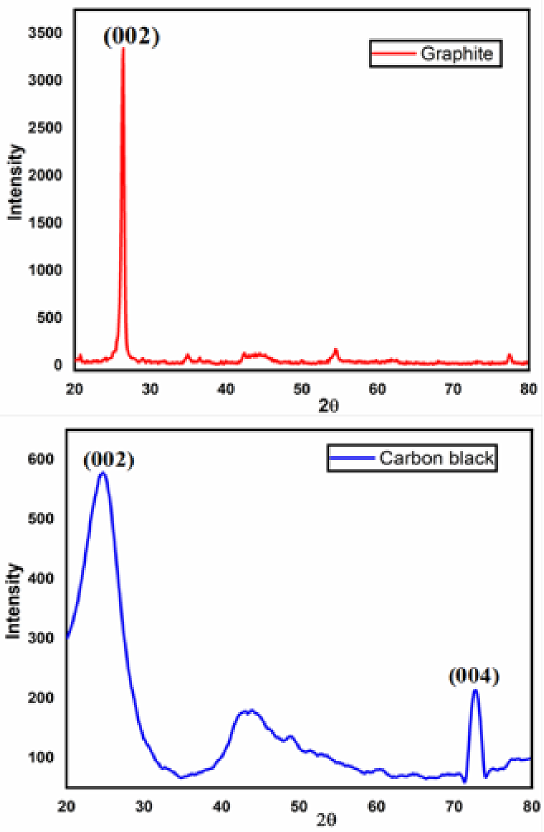

Figure 3. Carbon black and graphite analysis using XRD technique

The surface morphology of the materials was examined using scanning electron microscopy (SEM) type INSPECT-S50 with a RAITH-e-LiNE instrument at 10 kV voltage and distances of 5.6 mm and 10.6 mm, used to determine particle size and surface shape to graphite and Carbon black powders as in Figures 4 and 5.

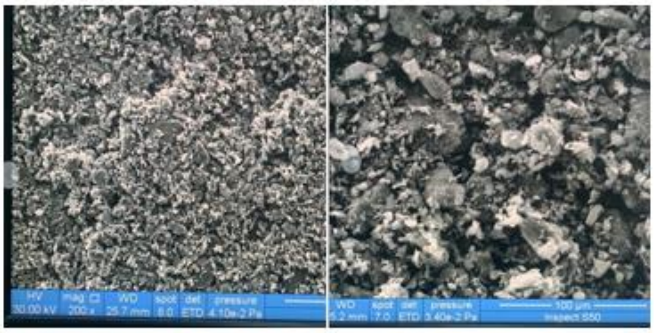

Figure 4. Graphite powder SEM TEST

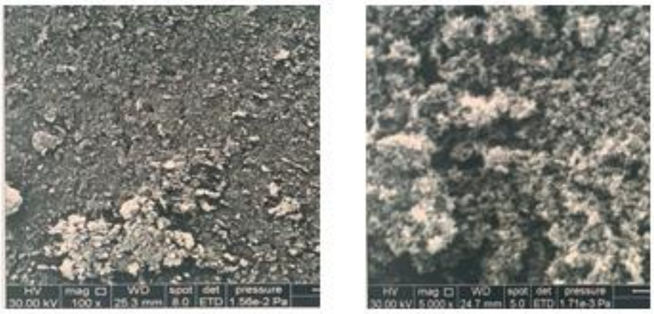

Figure 5. Carbon black powder SEM test

SEM analysis effectively highlights the distinct morphological and structural differences between graphite and carbon black. Graphite's semi layered appearance with the irregular, rough, and aggregated nature of carbon black, influencing their respective applications and functionalities. Graphite typically exhibits a layered structure, flat, and flaky particles. The flakes can vary in size, assume showing a hexagonal shape, and may appear smooth and shiny due to their layered arrangement. Carbon Black generally shows a more irregular morphology with a high surface area. Particles are usually spherical or aggregate into chain-like structures, resulting in a more complex and rough surface. Graphite particles can range from micrometres to larger flakes, with a relatively uniform size distribution. The size can vary depending on the grade of graphite, Graphite generally has lower porosity compared to carbon black, which can lead to denser packing in applications. Carbon Black particles are typically much smaller (nanometres to a few micrometres) and often exhibit a broad size distribution. The formation of aggregates and agglomerates can be observed, making the particle size less uniform. Carbon Black displays a rough surface texture with porosity, which enhances its adsorption capacity and makes it useful in various applications like reinforcement. Carbon Black irregular morphology and high surface area make it suitable for reinforcing agents in inks, and coatings, where mechanical strength and durability are crucial.

3. EXPERIMENTAL PROCEDURES

3.1 Anode graphite-carbon soot composite belt half cell model

The anode electrodes for Li-ion batteries were prepared using a graphite base combined with carbon soot powder derived from diesel generators. These electrodes were manufactured in the materials laboratory at MTU University, Iraq. To create the graphite/carbon soot-binder composite, small weight ratios as specified in Table 4 were measured using an OHAUS balance (China). The powder samples were then poured into a steel mold with a diameter of 20 mm, as depicted in Figure 6. The composite disks were formed by compressing the mixture with a hydraulic press (China)., applying a pressure between 10 and 50 tons. Following compression, samples A1-A4 underwent heat treatment at 60°C for about 1 hour in a KOTTERMANN oven (2715 - China) to eliminate any moisture or residual oils. The resulting belt samples (A1-A2), shown in Figure 7, were then subjected to electrical testing. These tests included measurements of electrical resistance and capacitance under AC voltage across a frequency range of 10-100 kHz, using an LCR meter (6500p

- Italy) and HC-3500 T (Italy). Testing was conducted under vacuum conditions in the laboratory at Al Nehrain University, as illustrated in Figure 8. This methodical approach ensures precise preparation and characterization of the graphite-carbon soot composite anode materials, aiming to enhance their performance in lithium-ion batteries.

Table 4. Graphaite-carbon soot half cell perpetration ratios

	Code	Graphite (Gm)	Binder (Gm)	Carbon Soot (Gm)	Compression (Ton)	Total Weight (Gm)
1	A1	3	0.3	-	50	3.29
2	A2	3	0.3	0.1	50	3.36
3	A3	3	0.3	0.2	50	3.42
4	A4	3	0.3	0.5	50	3.73

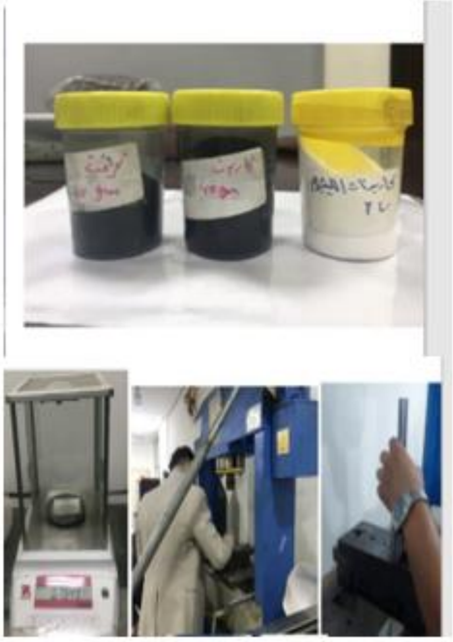

Figure 6. Image of chemical materials used for samples of graphite-carbon soot anode

Figure 7. Photes of graphite-carbon soot composite anode

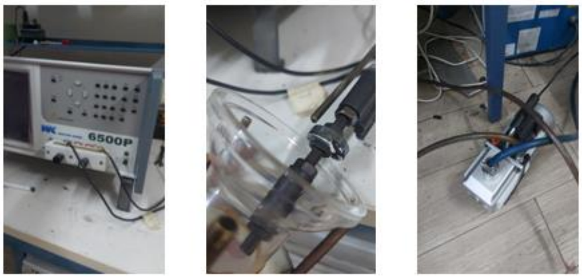

Figure 8. LCR electrical measurement system

3.2 Anode graphite-carbon black and CNTs composite thin film half cell model

Slurry mixture is prepared by mixing weight ratios of graphite (60-80%), commercial carbon black (5-7%), CNTs (5-10%) and binder CMC (5-10%). A thorough process was followed in order to create a half cell (anode electrode) containing graphite, carbon black, and carbon nanotube (CNTs) composite with binder CMC solvent thin films coated on copper foil as current collector. In order to create a uniform slurry, graphite, carbon black, and carbon nanotubes are combined with an appropriate weight ratio of CMC and distilled water followed mixing process for few minutes to get prober homogeneity slurry. Copper foil was cleaned with alcohol and distilled water then dried after surface treatment with polishing process to improve adhesion aim of the slurry with copper foil surface. To get a consistent and conductive layer, this mixture or slurry is subsequently applied on copper foil with doctor blade technique, dried in air oven for about 1 Hr at 50-60 °C. Dried samples applied to compression about (5-10) tons which effect greatly on coated film surface densities. The samples as in Figure 9 followed to drying process before electric tests to reduce any moisture effect on composite coated film surfaces. A lithium substance, such as lithium cobalt oxide ($LiCoO_2$), is deposited onto aluminium foil to create the cathode. After that, a separator (polymeric material thin foil) is placed between the anode and cathode to allow ion movement but avoid direct contact. To promote ion movement between the two, the electrolyte usually a lithium salt solution in an organic solvent is added. After that, a cell casing is placed over the entire assembly to create a sealed environment. A assemble Lithium battery (handmade) performance and longevity are studied by electrical tests in MTU lab to check out electrochemical properties as declared in Figure 9.

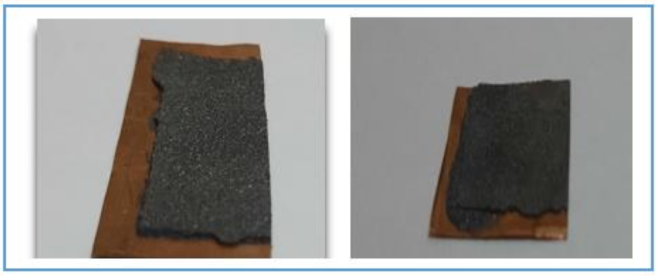

Figure 9. Photos of graphite-carbon black -CNTs composite anode after compression (1-10) ton

4. RESULTS AND DISCUSSION

The first experimental part results to demonstrate the different electrical characteristics of diesel generator soot (A4) and graphite (A1) as anode materials. Diesel soot, which has weak and unstable electrical qualities because of its irregular composition and impurities, was constantly outperformed by graphite, which is recognized for its good conductivity (Figure 10). This performance instability highlights how unsuitable soot for battery applications in the absence of intensive treatment. Since larger graphite ratios improve electrical pathways, increasing the graphite content greatly increased conductivity for the graphite and carbon soot anode samples (A1-A4). The results of studies of AC impedance over a frequency range of 10-15,000 Hz shed light on the efficiency of ion transport and the kinetics of charge transfer. Soot's broad variability in electrical resistance and capacitance at different frequencies further illustrated its lack of reliability compared to more refined carbon materials.

Secondly. We used graphite, carbon black (CB), and carbon nanotubes (CNTs) as additives to graphite base to create composite anode samples (A, B, C, and D) using coating technique to prepare anode electrode. The electrical characteristics of these composites significantly improved. Because of their exceptional conductivity and steady electrochemical performance, carbon black and carbon nanotubes (CNTs) were selected as conductive additions that greatly increase the anode's total conductivity. Carbon black and carbon nanotubes (CNTs) added to graphite improved the anode's electrical conductivity and capacitance, according to the electrochemical tests (Figures 11(A)-(D)). The development of a conductive network inside the anode, which promotes higher charge/discharge rates and more active sites for lithium-ion storage, is probably what caused this improvement. The combination of these materials also contributed to maintaining structural integrity during cycling, as CNTs support the mechanical stability of the anode.

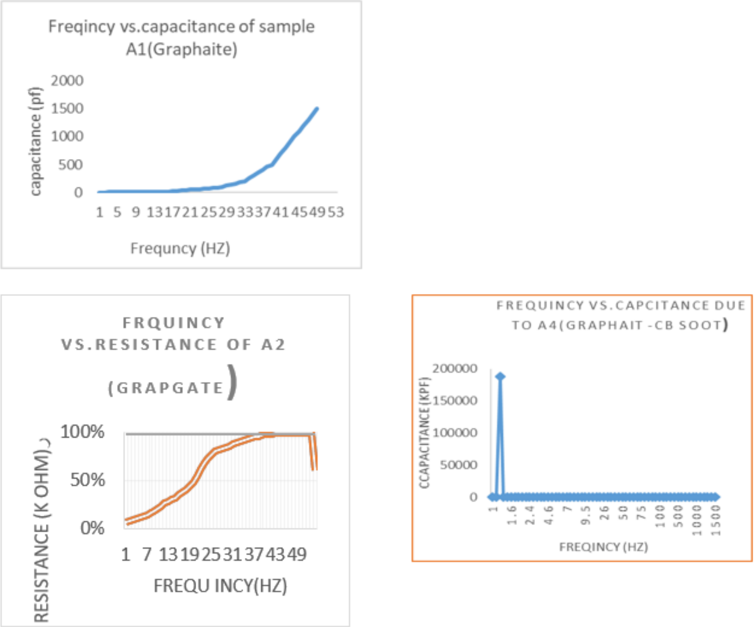

Figure 10. Line curve due to graphite a1 and graphite-carbon soot composite A4 anode resistance and capacitance characterization

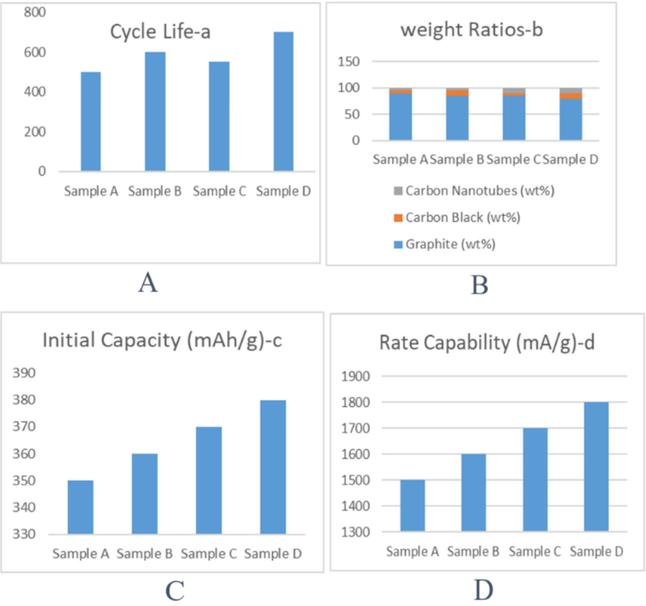

Figure 11. Electrochemical test due to (A-D) composite anode half cell model LIB

While Figure 10 shows the variations in the resistance and capacitance curves between graphite and the graphite-carbon soot composite demonstrate how the impurities and structural irregularities of carbon soot affect the anode material's electrical characteristics. This comparison emphasizes how carbon soot must be treated and purified in order to improve battery applications' performance.

Assessments of second set due to initial capacity, and cycle life revealed encouraging patterns. In particular, increased beginning capacities and improved cycle stability were the results of increasing the carbon black and CNTs content as in (Figure 11(A), 11(B), 11(C)). The adding carbon black to carbon nanotube ratios for best performance were also shown by the rate capability tests (Figure 11(D)). A promising strategy for high-performance lithium-ion batteries, the combination of graphite, carbon black, and carbon nanotubes (CNTs) in the anode material showed notable improvements in electrochemical performance overall.

The electrochemical performance of graphite-carbon anodes in lithium-ion batteries is greatly influenced by their microstructure. Lithium-ion diffusion kinetics and storage capacity are influenced by the degree of graphitization, crystallite size, and interlayer spacing as declared in second prepared set samples where addition of carbon black and CNTs improved the electrical performance. Rapid lithium-ion intercalation and de intercalation are made possible by highly graphitized materials with bigger crystallites and well-ordered layers, which boost rate capability and cycle life. On the other hand, increased lithium storage capacity is provided by disordered carbon architectures with smaller crystallites and wider interlayer spacing, but rate performance and cycle stability may be jeopardized. Furthermore, mechanical stability during cycling, ion transport, and electrolyte accessibility are all impacted by the carbon material's porosity and particle shape. Achieving high performance, long-lasting lithium-ion batteries requires optimizing the microstructure of graphite-carbon anodes.

5. CONCLUSIONS

The study emphasizes how crucial it is to choose the right materials for creating composites when creating graphite-based lithium battery anodes. The addition of carbon black and carbon nanotubes improves the anode's conductivity and electrical capacity, even though carbon soot powder is not a naturally usable material for graphite electrodes without undergoing severe thermal and chemical treatments. These enhancements are ascribed to the composite materials' advantageous chemical and physical characteristics as well as the efficient half-cell model preparation and creation techniques. The encouraging outcomes point to the possibility of continued development utilizing different nanomaterials and investigating additional features [34, 35]. Future studies would be concentrated on assessing the anode electrode's mechanical, thermal, and life cycle characteristics, paying special emphasis to the effects of pressing, coating, thickness and other properties of anode and cathode material [36, 37].

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