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Synthesis of Graphene-CeO_2 Nanocomposite with Enhanced Electrochemical Properties

Anil Kumar[1,*], Swati[2], Kusum[3]

Abstract

For the fabrication of graphene–cerium oxide (CeO_2G) composites, 99 mg of cerium oxide (CeO_2, 99.9%) was uniformly dispersed in 100 mL of deionized water via ultra-sonication (40 kHz) to achieve a stable suspension, followed by the incorporation of 1 mg of synthesized graphene. The resulting mixture was maintained at ambient temperature (25 ± 2 °C) under continuous magnetic stirring at 500 rpm for 2 h to promote effective interfacial interaction between CeO_2 nanoparticles and graphene sheets. The final product, denoted as CeO_2G1, was collected and dried under vacuum at 60 °C for 24 h. Using the same procedure, additional CeO_2G composites containing 3 mg and 5 mg of graphene with 97 mg and 95 mg of CeO_2 were synthesized and labeled as CeO_2G3 and CeO_2G5, respectively. The as-prepared composites were systematically characterized to investigate the influence of graphene content on their structural, morphological, and electrochemical properties. Structural analysis confirmed the successful incorporation of graphene without disturbing the crystalline phase of CeO_2, while morphological studies revealed improved dispersion of CeO_2 nanoparticles on graphene sheets with increasing graphene concentration. The presence of graphene significantly enhanced the electrical conductivity and interfacial charge transport within the composites. Electrochemical investigations demonstrated that graphene incorporation led to improved specific capacitance, reduced charge-transfer resistance, and enhanced cycling stability compared to pristine CeO_2. Among the synthesized materials, CeO_2G5 exhibited superior electrochemical performance due to the synergistic interaction between highly conductive graphene networks and redox-active CeO_2. These findings highlight the potential of graphene–CeO_2 composites as promising electrode materials for advanced energy storage and electrochemical applications.

Keywords: Graphene, CeO_2, Nanocomposites, XRD, FTIR analysis, Photo catalysis, Laser Raman Spectral Analysis.

INTRODUCTION

The industrial revolution has generated immense demand in the energy sector. However, the inherent energy resources such as coal, radioactive minerals, and petroleum for traditional energy generation are gradually diminishing and will eventually be entirely depleted. Thorough investigation into materials that can address the issue of energy requirements is underway, leading to innovative approaches for energy generation such as silicon solar panels, thermoelectric systems, photovoltaic devices, and dye-sensitized solar technologies [1]. Nevertheless, the accumulation of energy is essential in the future for optimal use of generated energy [2]. Thorough explorations are underway to pinpoint the electrode substances that possess enhanced storage potential and superior efficiency [3]. The nanoscale metal oxides and their composites

***Author for Correspondence**
Anil Kumar

[1]Associate Professor, Department of Applied Science, Bharati Vidyapeeth's College of Engineering, New Delhi, India.
[2]Assistant Professor, Department of Chemistry, Baba Mast Nath University, Asthal Bohar, Rohtak, Haryana, India
[3]Research Scholar, Department of Chemistry, Baba Mast Nath University, Asthal Bohar, Rohtak, Haryana, India

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have been documented to exhibit remarkable electronic characteristics alongside outstanding recyclability, attributed to their increased surface area [41]. Reports indicate that transition metal oxides possess the capability to surpass the constraints of low energy density in electrochemical capacitors [42]. Following the triumphant creation of graphene [43, 44], scientists delve into the exploration of graphene-infused composites alongside various nanomaterials, including precious metals, intricate oxides, and metallic oxides [4, 45-48].

Graphene-metal oxide composites have captured interest as a promising substitute to enhance the effectiveness of diverse catalytic and storage reactions in energy conversion applications [1, 5]. Recent advancements in composite science have emphasized the critical role of optimizing fabrication parameters and utilizing reinforcing fillers to enhance material properties [6]. For instance, Karuppiah *et al*. [43] demonstrated the importance of multi-objective optimization in fabrication parameters to achieve superior composite performance. Similarly, the integration of various fibers and fillers has been shown to significantly enhance mechanical and structural integrity in reinforced composites [7, 44, 46]. Lessons learned from the modification of natural fibers, such as alkali treatments to improve surface properties [48], and the analysis of processing parameters like drilling [8, 45], provide valuable insights into interface engineering. Furthermore, the role of specific fillers in enhancing the properties of hybrid polymer composites [47] parallels the strategy employed here: using graphene as a functional filler to augment the electrochemical properties of the cerium oxide matrix [9].

Nevertheless, the infrequently explored rare earth oxide graphene composites have shown limited examination regarding their electrochemical characteristics [10]. The combination of graphene and CeO_2 offers a unique synergistic advantage: CeO_2 acts as a spacer to prevent the restacking of graphene sheets, thereby maintaining a high surface area, while graphene provides a highly conductive network that overcomes the poor electrical conductivity of pure CeO_2 [11]. This synergy facilitates faster electron transport and enhances the utilization of CeO_2 for redox reactions. The synthesis and characterization of CeO_2-graphene composites have been documented [12, 37]. The electrochemical capabilities of cerium oxide-graphene nanocomposites were explored as a potential anode material for lithium-ion batteries, revealing that the inclusion of graphene nanosheets in the nanocomposites improved electronic conductivity and inhibited the agglomeration of CeO_2 nanoparticles [13, 38]. Nonetheless, the necessary amount of graphene in the composite to boost the electrochemical performance is crucial for developing an effective composite, which has yet to be documented within the boundaries of our literature review [14].

In this regard, to ascertain the necessary amount of graphene for improving the electrochemical performance, various weight percentages (1, 3, and 5%) of synthesized graphene were combined with cerium oxide, and the nanocomposites were created using a direct solution mixing technique [14]. This chapter delves into the characterization of these nanocomposites through a multitude of techniques; the electrochemical performance was scrutinized via cyclic voltammetric analysis [15].

SYNTHESIS OF GRAPHENE-CEO_2 COMPOSITES

For the fabrication of graphene-cerium oxide (CeO_2G) composites, 99 mg of cerium oxide (CeO_2) (99.9%) was uniformly dispersed in 100 mL of deionized water. The dispersion was subjected to ultrasonication at 40 kHz frequency for 30 minutes to ensure the breakdown of agglomerates and formation of a stable suspension. This was followed by the incorporation of 1 mg of synthesized graphene into the mixture.

Subsequently, the mixture was maintained at ambient temperature (25 ± 2 °C) while being continuously agitated using a magnetic stirrer at 500 rpm for a duration of 2 hours. This rigorously controlled mixing time was essential to promote the adsorption of CeO_2 nanoparticles onto the graphene surface via van der Waals forces and electrostatic interactions. The ultimate product CeO_2G1 was gathered and dehydrated in a vacuum at 60°C for a duration of 24 hours to ensure complete moisture

removal. Following the identical method and process parameters, CeO_2G composites incorporating 3 and 5 mg of graphene alongside 97 and 95 mg of CeO_2 were also synthesized and designated as CeO_2G3 and CeO_2G5 correspondingly. The impact of graphene on the structural and electrochemical characteristics of the composites has been examined.

RESULTS AND DISCUSSION

Power X-ray Diffraction Studies

The produced graphene, untainted cerium oxide, and CeO_2G nanocomposites underwent powder X-ray diffraction examinations, and the captured patterns are illustrated in Figure1. The diffraction pattern observed in graphene showcases merely two distinct peaks, which align with the (002) and (100) diffraction planes at two theta angles of 23.44 and 42.92 degrees, correspondingly [16]. The diffraction patterns of cerium oxide and the CeO_2G composites exhibit numerous prominent peaks, affirming their excellent crystalline characteristics. The diffraction patterns were analyzed in relation to the reference JCPDS#65-5923 for cerium oxide, and the peaks were systematically indexed [17]. Analyzing the patterns of CeO_2G composites reveals that the impact of graphene is minimal due to its limited quantity. Upon meticulous scrutiny of the amplified peaks at different two theta values, it becomes evident that the incorporation of graphene has noticeably altered the peaks, shifting them towards lower diffraction angles due to lattice strain, while also diminishing their intensity, as illustrated in Figure 2.

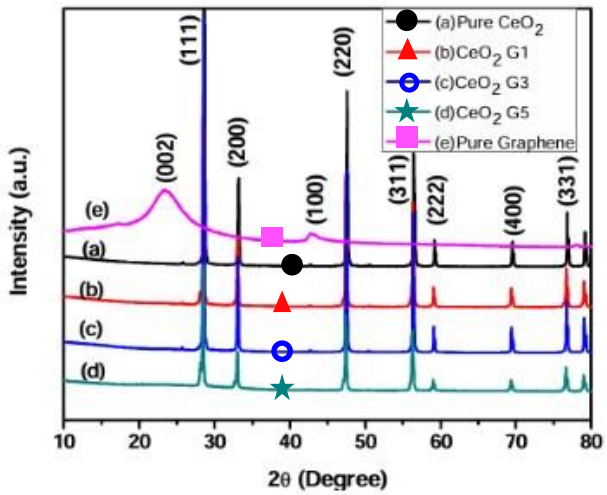

Figure 1. Powder X-ray diffraction pattern of graphene, pure CeO_2 and CeO_2G composites

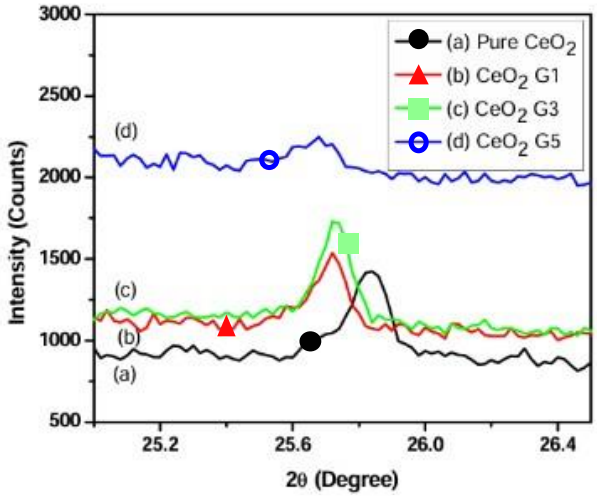

Figure 2 Peak shift observed in the powder X-ray diffraction patterns of pure CeO_2 and CeO_2G composites

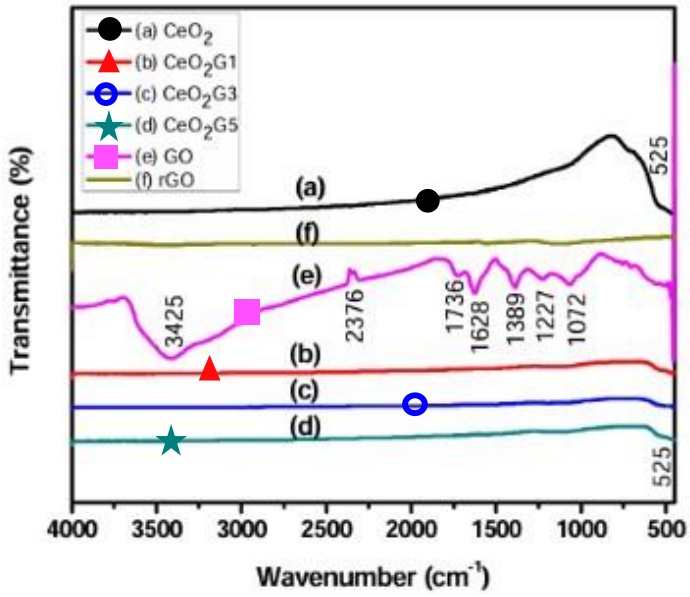

Figure 3: FTIR spectra of (a) CeO_2, (b) CeO_2G1, (c) CeO_2G3, (d) CeO_2G5 (e) graphene oxide and (f) reduced graphene oxide

FTIR Spectral Analysis"

FTIR spectral analyses have revealed the existence of functional groups in CeO_2, graphene oxide, reduced graphene oxide (rGO), and CeO_2G composites. The spectra have been documented within the wavenumber spectrum spanning from 400 to 4000 cm^{-1}, as illustrated in Figure 3. The prominent peak observed at 553 cm^{-1} arises from the stretching oscillations of Ce-O, as illustrated in Figure 3 (a) [18]. No additional notable peaks are detected in the spectrum, confirming that the ceria remains devoid of absorbed moisture and carbon dioxide found in the environment. Analysis of the GO spectrum reveals that the peaks near 1700 cm^{-1} correspond to the stretching vibrations of C=O. The extensive band noted at 1072 cm^{-1} is linked to C-O stretching oscillations [19]. The stretching oscillations attributed to C=C manifested at 1628 cm^{-1}. The FTIR spectrum of the graphene derived from graphene oxide confirmed its successful reduction, as it displayed an absence of peaks [20]. Nevertheless, the spectral analysis of all composites reveals that the peak observed at 525 cm^{-1} corresponds to the stretching vibrations of Ce-O, and this absorption diminishes in the CeO_2G5 sample. Crucially, the residual functional groups (C=O and C-O) on the graphene surface, though diminished, play a vital role in electrode stability [21]. They serve as anchoring sites for the CeO_2 nanoparticles, preventing phase separation during the charge-discharge cycles, and facilitate the wettability of the electrode by the electrolyte, thereby enhancing ion diffusion kinetics [22].

Laser Raman Spectral Analysis

Laser Raman spectra were captured within the range of 800 to 2000 cm^{-1} for the CeO_2, graphene, CeO_2G1, and CeO_2G5 composites. The pure graphene spectrum displays two distinct bands, known as the D and G bands, located at 1343 and 1577 cm^{-1}, respectively, as illustrated in Figure 4. The intensity ratio of the D and G bands, ID/IG for graphene, exceeds 1 (1.11), indicating that the graphene derived from GO possesses an ordered structure with commendable quality and confirms the presence of sp2 bonded carbon [23]. The CeO_2G5 composite exhibits an extra pronounced peak at 1161 cm^{-1} attributed to cerium oxide, alongside the D and G bands characteristic of graphene. Figure 5 presents a comparison of the Raman spectra for CeO_2G1 and unadulterated CeO_2, revealing that the spectrum of pure CeO_2 exhibits a pronounced peak at 1163 cm^{-1} [24]. Extremely faint signals were detected for the D and G bands of graphene within the CeO_2G1 composite, attributed to the inadequate development of the composite. The alterations in the band, heightened intensity, and supplementary peaks serve as proof for the creation of the CeO_2G5 composite.

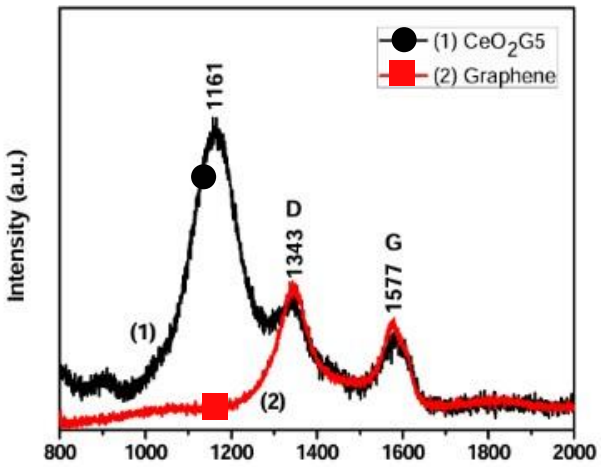

Figure 4. Comparison of Laser Raman spectra of CeO_2G5 and graphene

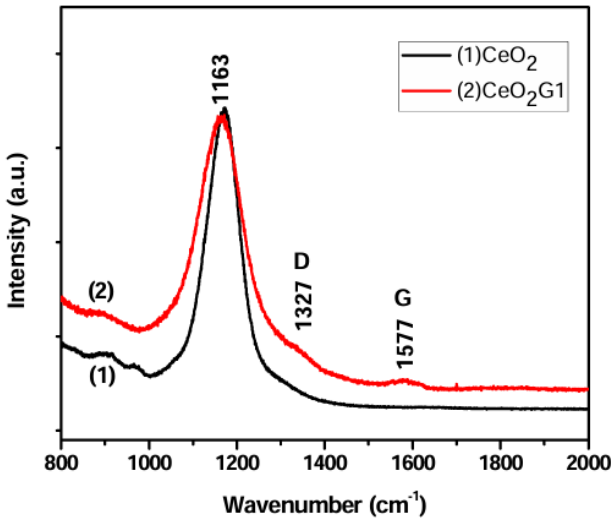

Figure 5. Comparison of Laser Raman spectra of CeO_2G1 and pure CeO_2"

Morphology Studies

The structure of the acquired composites was examined using a Field Emission Scanning Electron Microscope (FE-SEM), and the resulting images are presented in Figure 6 (a-d) [25]. The structure of CeO_2G1 reveals that the graphene layers are haphazardly arranged within the CeO_2 matrix, merely inserted between the graphene sheets without creating a composite, as illustrated in Figure 4.6a [26]. Images captured at elevated magnification (Figure 6b) demonstrate that the crumpled graphene exhibits a thickness on the scale of several nanometres [27]. The structure of the CeO_2G5 composite is illustrated in Figure 6c. The rise in the weight percentage of graphene has led to the clustering of CeO_2 alongside graphene, resulting in a distorted, flake-like configuration of the graphene structure, as illustrated in the enlarged image (Figure 6d).

High-resolution transmission electron microscopy images of CeO_2G5 at various magnifications are presented in Figure 7 a-d. The CeO_2 particles, varying in size from 100 to 250 nm, were noted to be dispersed across the surface of the graphene sheets, as indicated in Figure 7a. The image with significant magnification suggests that the dimensions of the particles range from approximately 10 to 50 nm at various depths of graphene layers, as illustrated in Figure 7b. Illustration Figure 7c depicts the crumpled and contorted graphene layers at a 50 nm dimension, while the highlighted area is presented in Illustration Figure 7d at a 5 nm dimension [28]. Images captured at elevated magnification within the

5-10 nm range of highlighted areas in Figure 4.7b are presented in Figure 4.7e-g. The rectangular indication displays the dense cluster of graphene sheets that lack any lattice fringe pattern, as illustrated in Figure 7e. The ongoing circular area displays vibrant lattice fringes, as illustrated in Figure 7f, confirming the existence of CeO_2 directly beneath the graphene layer [29]. The dotted circular area exhibits distinctly visible luminous fringes (Figure 7g), confirming that CeO_2 is positioned atop the graphene layer. These visuals significantly aided in the creation of a composite [30]. The graphene configuration comprises 15 layers within 5 nm, as illustrated in Figure 7g, which validates that graphene has been successfully diminished from graphene oxide using this approach [31].

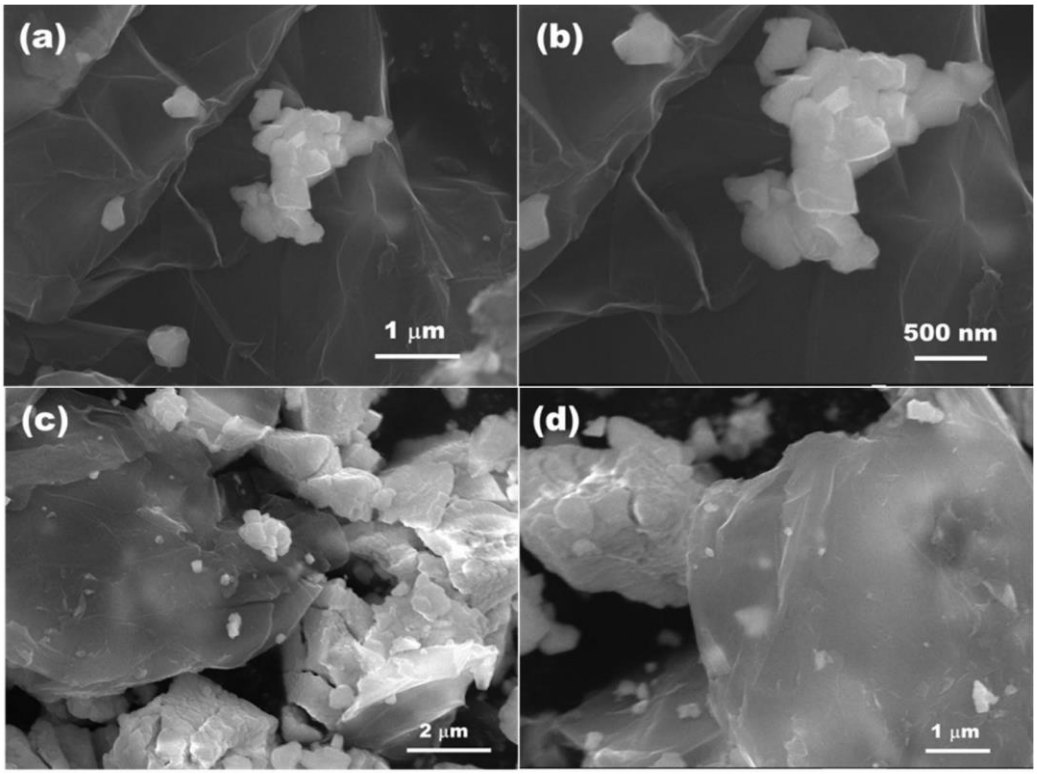

Figure 6. FE-SEM images of (a & b) CeO_2G1 and (c & d) CeO_2G5

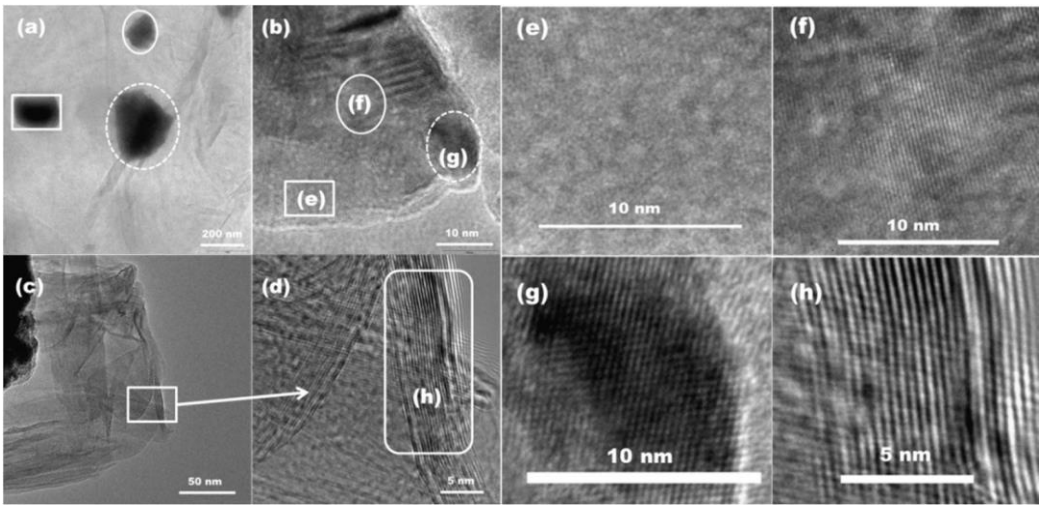

Figure 7. HR-TEM images of CeO_2G5 (a) nanocrystals of CeO_2 on the top surface of graphene sheets, (b) CeO_2 nanocrystals at different depth between the graphene sheets, (c) wrinkled graphene sheets with CeO_2 nanocrystals (d) High magnification image of folded graphene sheets and (e-h) magnified images of marked regions in (b and d)."

Table 1. Elemental composition of reduced graphene and CeO_2G composites

Sample	Element	Atomic %	Error
Graphene	C K	85.67	0.12
	O K	14.33	1.23
CeO_2	Ce L	30.30	1.77
	O K	69.70	0.24
CeO_2G1	Ce L	6.68	0.96
	C K	65.84	0.06
	O K	27.48	0.20
CeO_2G5	Ce L	4.37	1.19
	C K	72.89	0.07
	O K	22.74	0.28

Elemental Mapping Studies

The crafted composites underwent field emission electron probe microanalysis, and the elemental mapping was documented. Figure 8 illustrates the EPMA mapping alongside the associated energy dispersive spectrum for the CeO_2G5 composite [32]. The even distribution of carbon, oxygen, and cerium elements was noted in all composites. The energy dispersive spectra were additionally captured for every composite using this approach, revealing a consistent rise in carbon concentration from CeO_2G1 to CeO_2G5 [33]. The fundamental makeup of the composites and originating materials is presented in Table 1. The typical oxygen proportion was noted to be below 21 percent in both pure graphene and its composites, which guarantees the efficient minimization of graphene oxide [34].

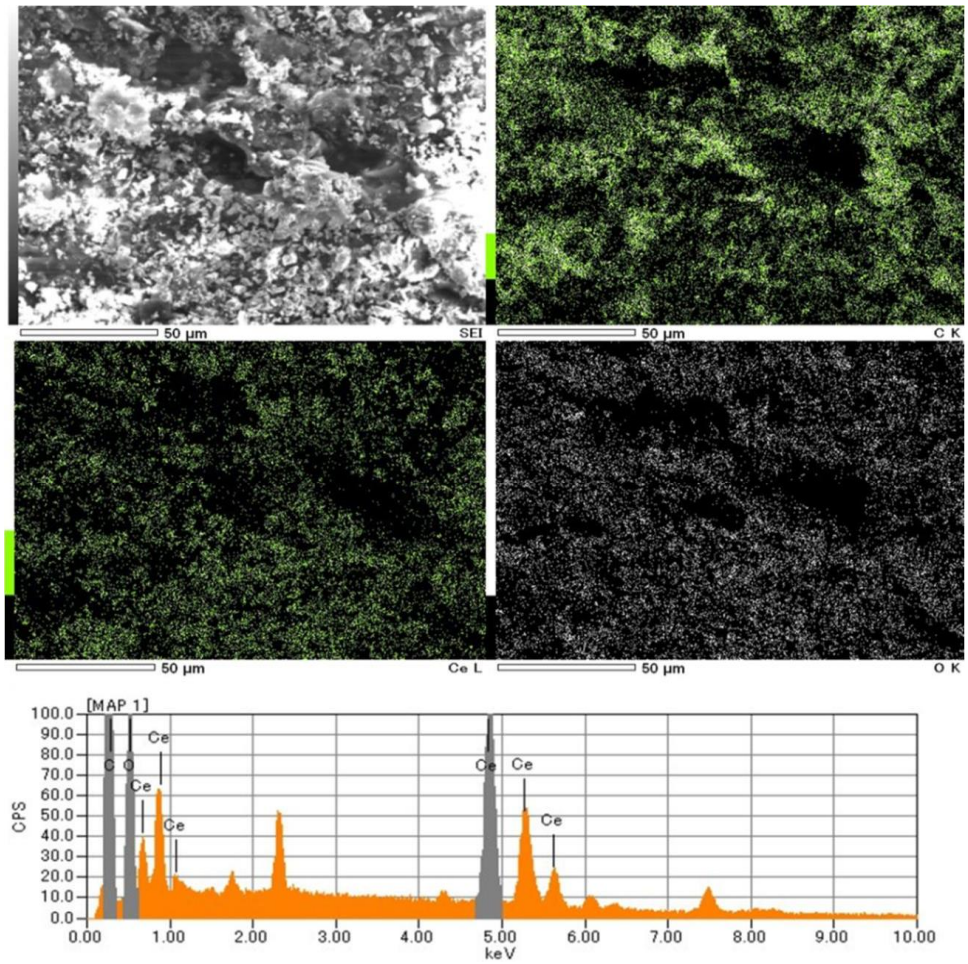

Figure 8. FE-EPMA images of CeO_2G5 and corresponding EDS spectrum

X-ray Photoelectron Spectral Studies (XPS)"

The XPS spectrum of the CeO_2G5 composite was obtained utilizing the ESCA 3400 SHIMADZU X-ray photoelectron spectrometer and examined through core level XPS spectra of different elements. The examination range of the CeO_2G5 composite is illustrated in Figure 9. The range of the survey encompasses the signals for O1s and C1s electrons [35]. Nonetheless, the remaining components are not distinctly detected in the survey spectrum. Consequently, the fundamental core level spectra were captured, and Figure 10 illustrates the 3d electrons core level spectrum of cerium within the energy range of 875-920 Ev [36]. The XPS spectrum comprises three unique signals that correspond to various ionic states of Ce^{3+} and Ce^{4+}. The distinctive XPS signals of Ce^{3+}, 3d5/2 and 3d3/2 electron states have been detected at 886 and 905 eV, correspondingly, with a binding energy difference of 9 eV. The supplementary satellite signal arises from the Ce^{4+}, 3d3/2 electron state at 919 eV [37]. The fundamental spectrum of the 4p electrons of cerium within the range of 200-240 eV is illustrated in Figure 11. The spectrum exhibits two distinct peaks associated with the 4p3/2 and 4p1/2 states, located at 210.3 eV and 227.6 eV, respectively. The disparity in binding energy between these two states is recorded at 17.3 eV, showcasing a commendable alignment with the established values [38].

The fundamental XPS spectrum for C 1s electrons is illustrated in Figure 12, featuring a significant signal at 284.3 eV along with a satellite peak at 289.4 eV. The first aspect relates to sp2 hybridized carbon (C-C), while the second pertains to carboxyl groups (C-C=O), suggesting that the carbon possesses a significant quantity of oxygen-rich functional entities [39]. Two distinct oxygen species can be identified through the deconvolution of the core level spectrum of O1s electrons within the range of 525 to 540 eV, as illustrated in Figure 13. The binding energies of 530.5 eV and 531.8 eV have been attributed to lattice oxygen and carboxyl groups, respectively. The presence of surface oxygen functionalities, as confirmed by the peak at 531.8 eV, is critical for the charge transfer mechanism. These functional groups contribute to pseudocapacitance through surface redox reactions and improve the interfacial contact between the electrode material and the electrolyte, reducing the interfacial resistance [40].

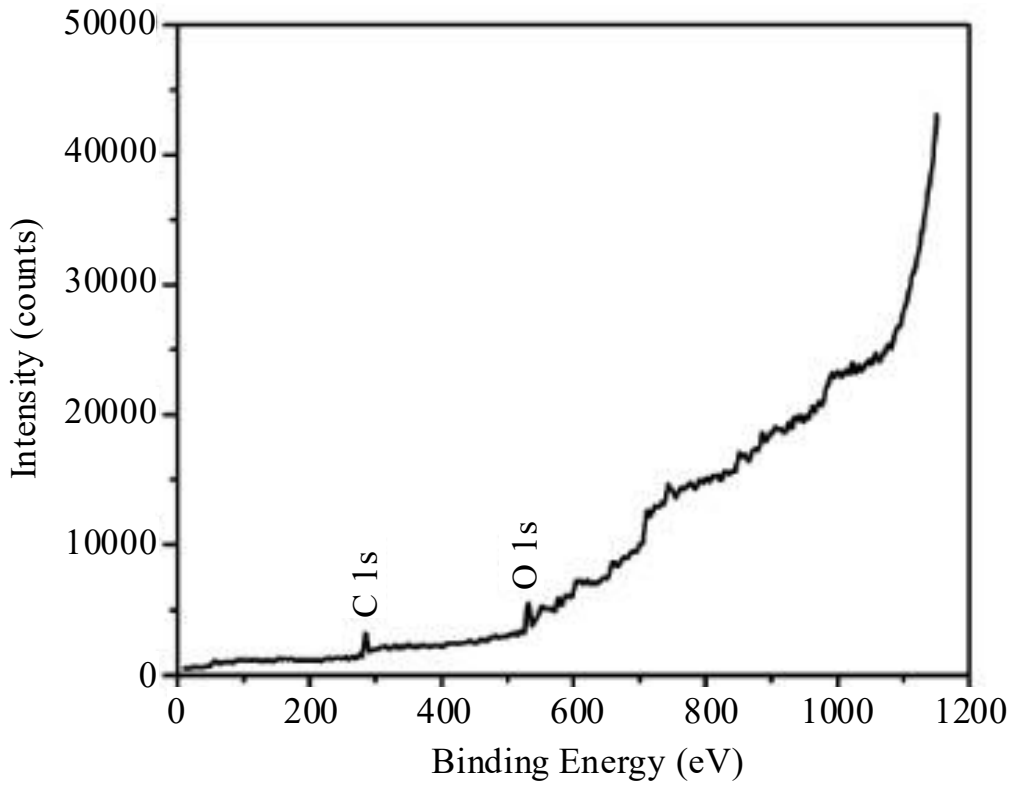

Figure 9. X-ray photoelectron survey spectrum of CeO_2G5

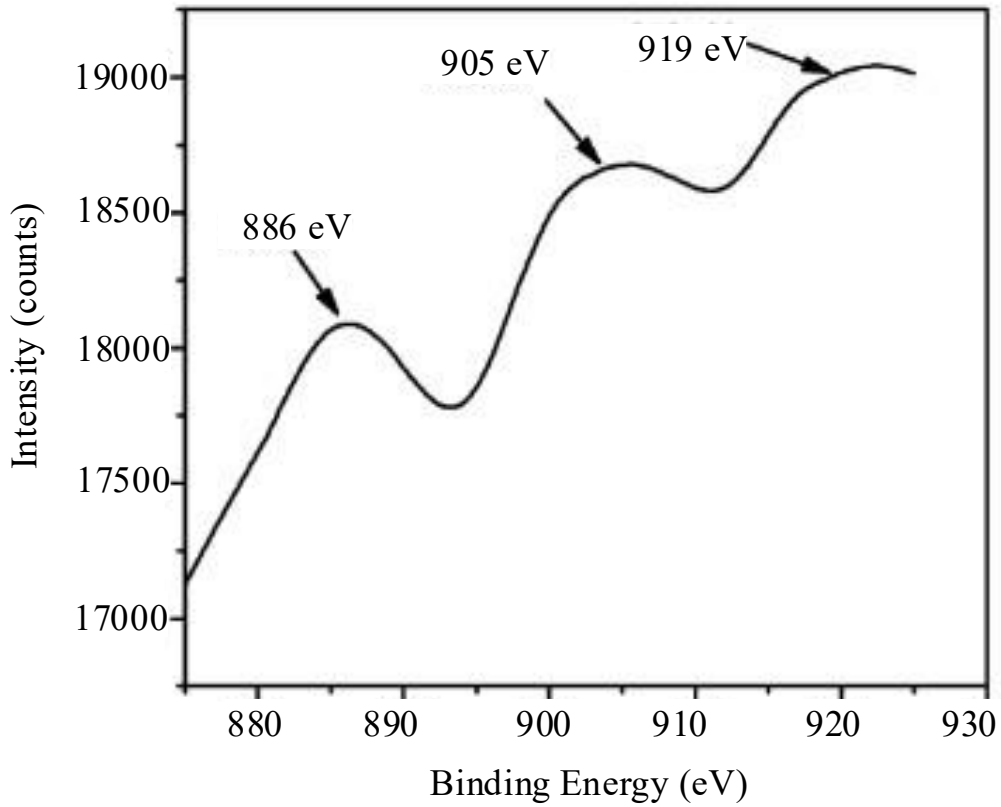

Figure 10. Core level X-ray photoelectron spectra of Cerium 3d electron

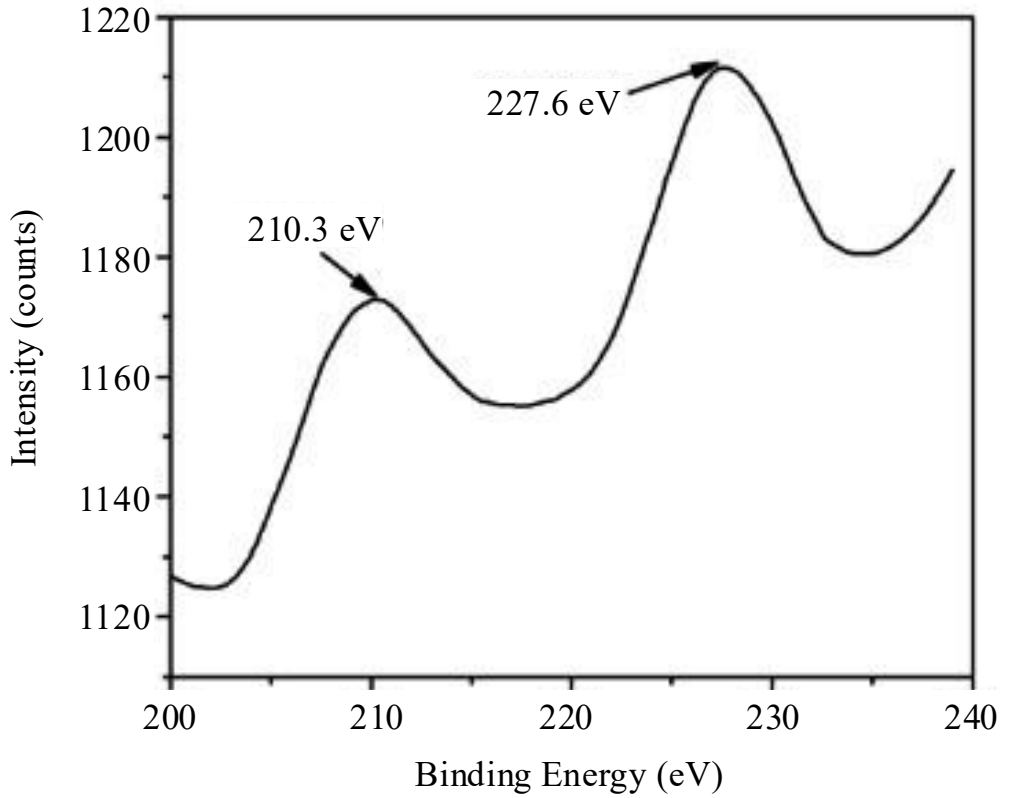

Figure 11. Core level X-ray photoelectron spectra of Cerium 4p electron

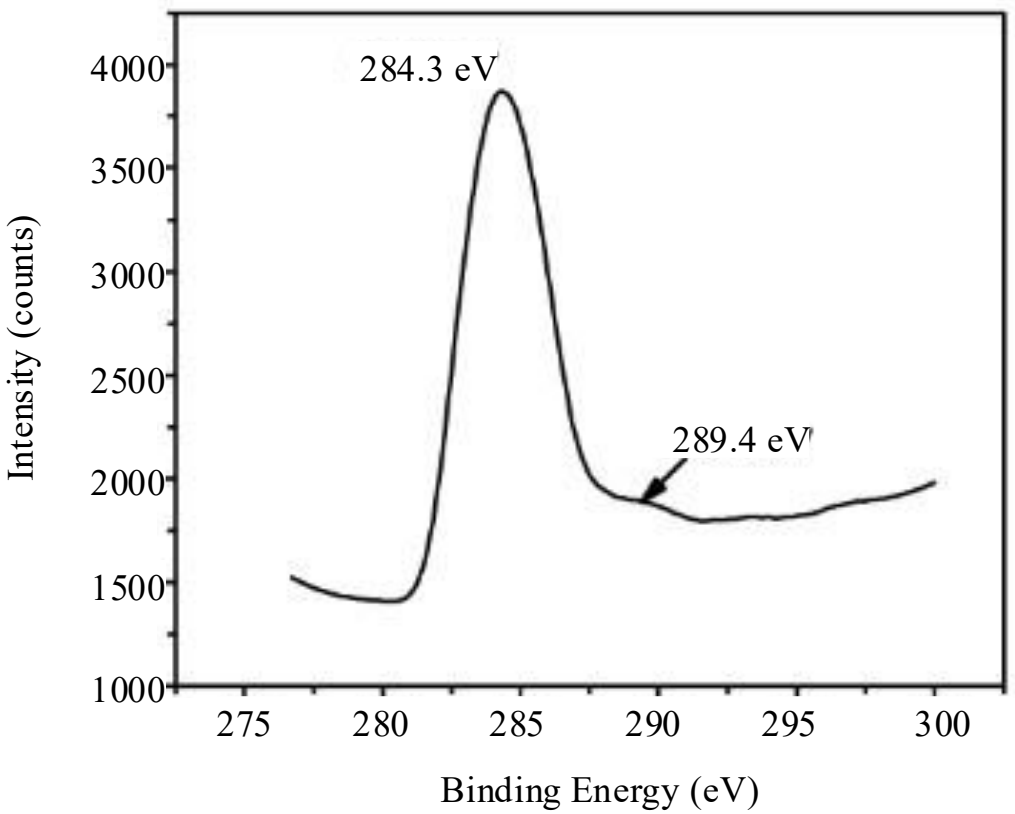

Figure 12. Core level X-ray photoelectron spectra of Carbon 1s electron

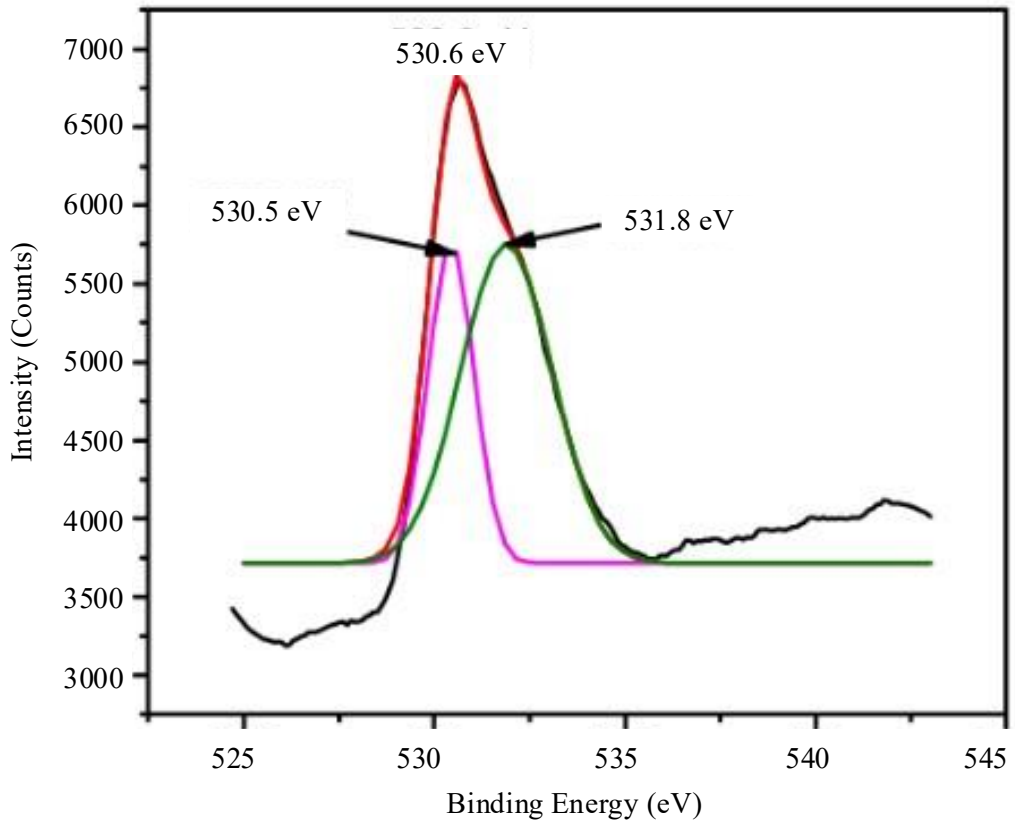

Figure 13. Core level X-ray photoelectron spectra of Oxygen 1s electron

Cyclic Voltammetric Studies

The investigation of electrochemical characteristics of pristine CeO_2 and the CeO_2G composite has been conducted through Cyclic Voltammetry. Figures 14-17 display the cyclic voltammogram outcomes of Pure CeO_2, CeO_2G1, CeO_2G3, and CeO_2G5, correspondingly in a 1M H_2SO_4 electrolyte solution. According to Figure 14, the unadulterated CeO_2 electrode exhibits responses akin to capacitance within the examined potential range [41]. Nonetheless, there is an increase in voltammetric current accompanied by a positive shift in electrode potentials during both positive and negative sweeps, particularly at a comparatively swift scan rate. In comparison to alternative electrodes, the CeO_2G5 electrode exhibits the most pronounced current response, showcasing a clear redox current within the potential range of (-0.6 to +0.6 V), as illustrated in Figure 17. Analyzing the curves 18 and 19 reveals that the peak current of CeO_2G5 results from the elevated proportion of graphene, which significantly boosts the double layer capacitance characteristics. The precise capacitance measurements for CeO_2 and CeO_2G composites across various scan rates have been determined by analyzing the area of the CV curve utilizing Equation (4.1).

$$C_s = {}^3IdV/vmV. \quad (1)$$

In this context, I represents the response current, V denotes the potential, v indicates the potential scan rate, and m signifies the mass of the active material present on the electrode. The distinct capacitances for the active substances at different scanning velocities are organized in Table 2. In the case of CeO_2G nanocomposite, the specific capacitance values diminish as the scan rate escalates [42]. The highest specific capacitance recorded was 65.4 Fg^{-1} at a scan rate of 10 mVs^{-1} for the CeO_2G5 sample. Furthermore, the connection between the oxidation peak current at various scan rates and the square root of the scan rate exhibits a linear relationship, suggesting a process governed by diffusion [43]. It is understood that every redox reaction encompasses the movement of electrons. The remarkable electrochemical performance of CeO_2G5 is attributed to the synergistic effect between the components: CeO_2 contributes to pseudocapacitance via reversible redox reactions between Ce^{3+} and Ce^{4+} states, while graphene acts as a highly conductive backbone that facilitates rapid electron transfer and reduces the charge transfer resistance (Rct). The intimate contact between CeO_2 and graphene minimizes the diffusion path length for ions, leading to the observed diffusion-controlled kinetic behavior [44].

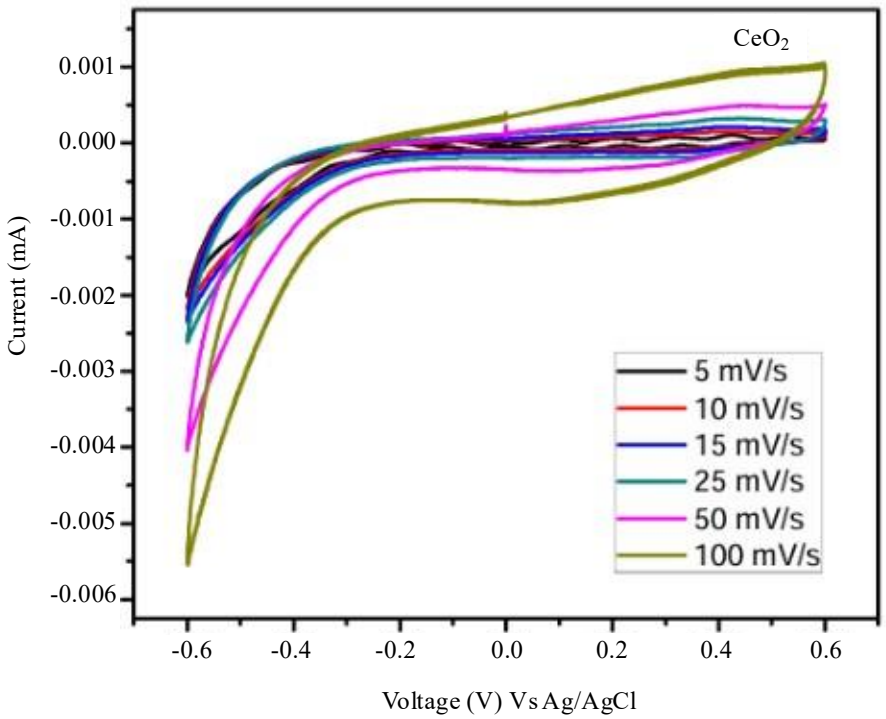

Figure 14. Cyclic voltammetric curves of pure CeO_2 under different scan rates

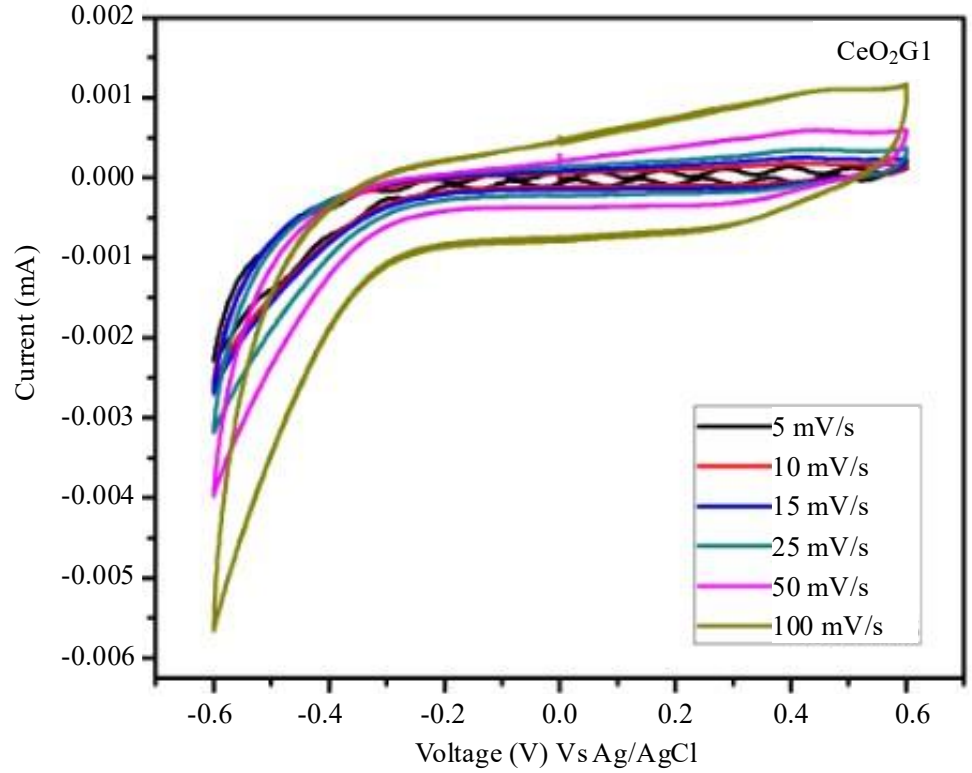

Figure 15. Cyclic voltammetric curves of CeO_2G1 under different scan rates

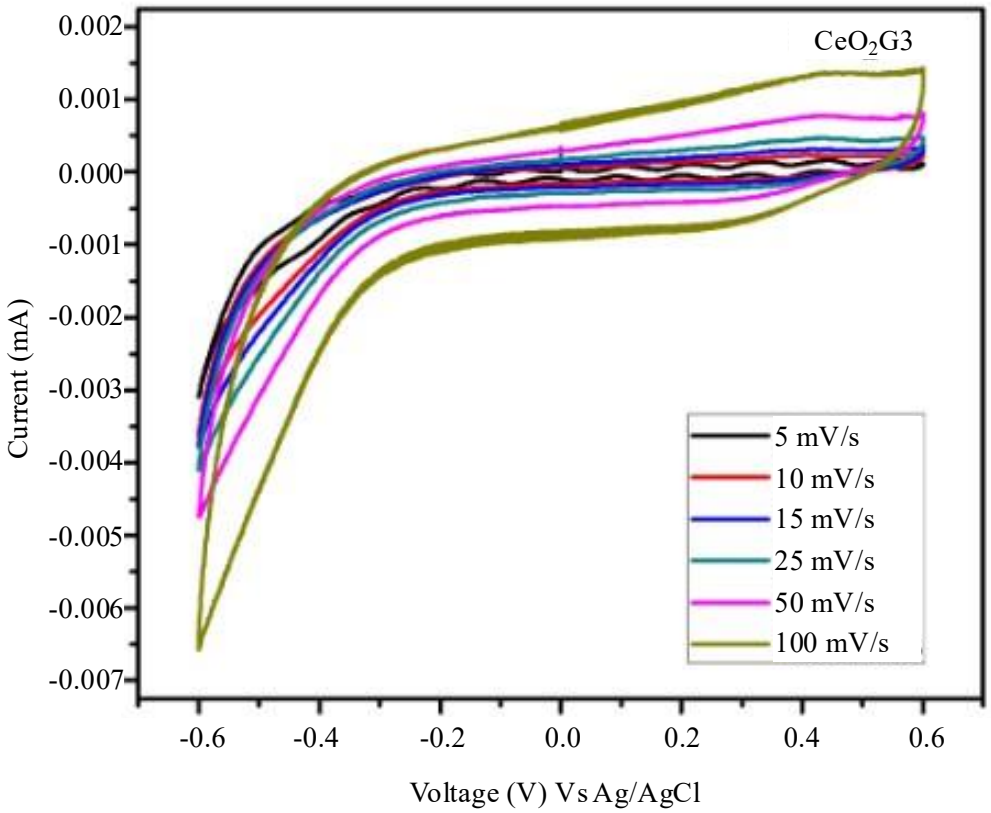

Figure 16. Cyclic voltammetric curves of CeO_2G3 under different scan rates

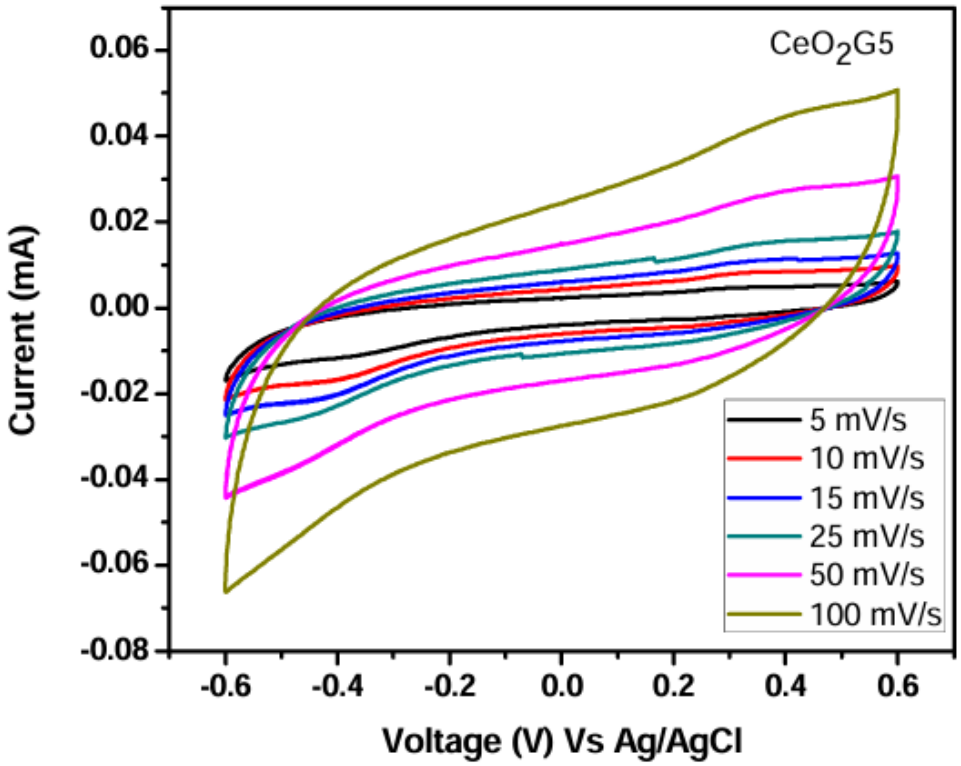

Figure 17. Cyclic voltammetric curves of CeO_2G5 under different scan rates

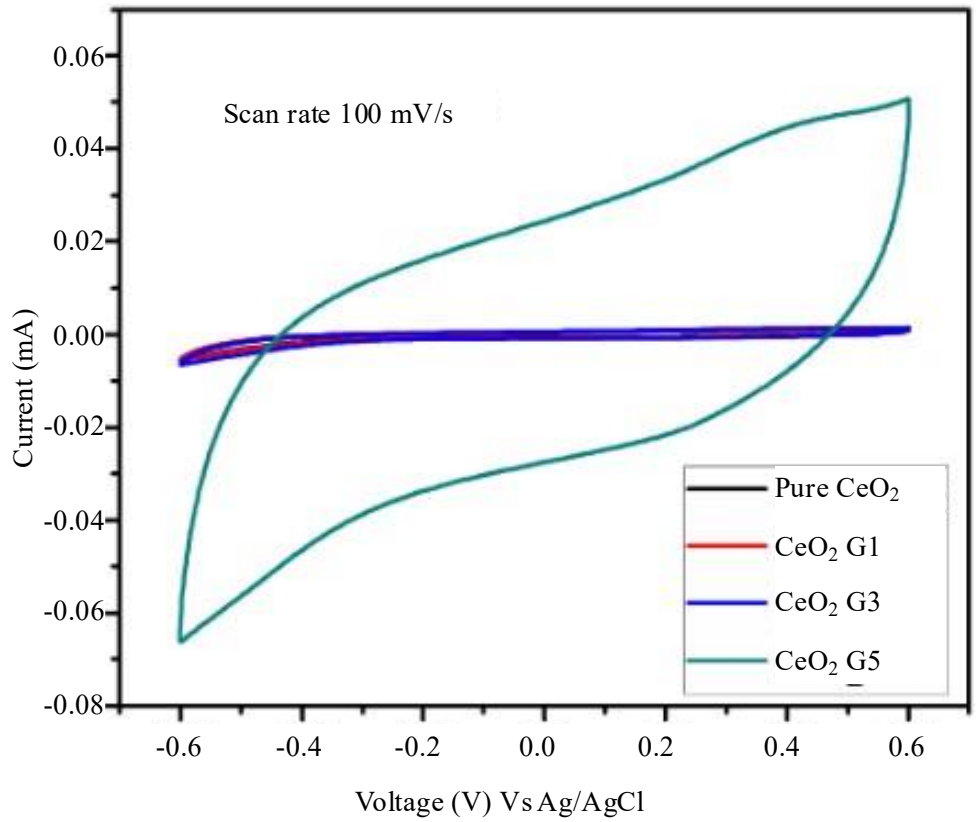

Figure 18. A comparison of cyclic voltammetric curves of Pure CeO_2, CeO_2G1, CeO_2G3 and CeO_2G5 under 100 mV/s scan rate and (b) Magnified curves to show the variations

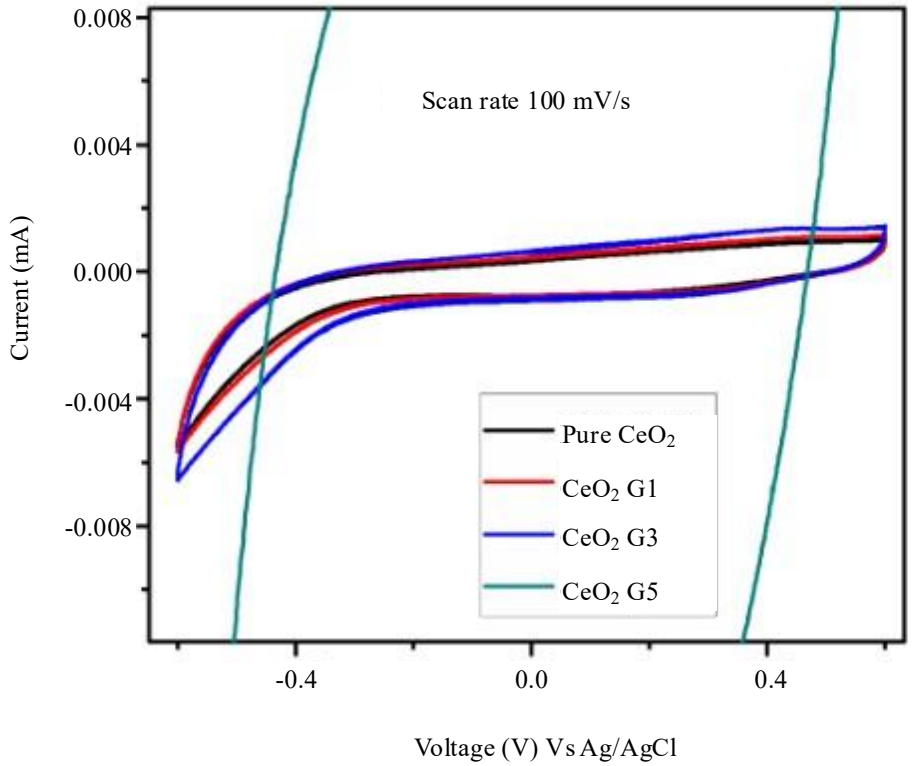

Figure 19: Magnified curves of pure CeO_2, CeO_2G1, CeO_2G3 and CeO_2G5 under 100 mV/s scan rate

Table 2. Specific capacitance for active materials at different scanning rates

Scanning rate (mV/s)	Specific capacitance of active electrode materials (Fg^{-1})			
	CeO_2	CeO_2G1	CeO_2G3	CeO_2G5
10	42.3	51.2	58.3	65.4
25	33.2	43.1	48.3	52.7
50	28.5	32.6	39.5	44
100	24.2	25.4	28.9	31.2

OBSERVATIONS AND DISCUSSION

The pursuit of eco-friendly energy has escalated because of the exhaustion of conventional energy sources such as coal, oil, and nuclear materials. In response to this, scientists are concentrating on innovative substances and advancements for energy transformation and retention. The combination of nanoscale metal oxides and graphene within composites has attracted considerable attention owing to their improved electronic characteristics, extensive surface areas, and ability to be recycled [45]. This section delves into the creation of graphene-CeO_2 composites, examining the influence of different weight ratios of graphene within these composites on their structural characteristics and electrochemical behavior. Graphene-CeO_2 composites have surfaced as exciting contenders for boosting the electrochemical efficiency of energy storage systems. Graphene exhibits remarkable electrical conductivity, whereas cerium oxide (CeO_2) is recognized for its outstanding redox characteristics, rendering it perfect for energy storage uses. Investigators have examined numerous graphene-metal oxide hybrids, yet the pairing of graphene with uncommon earth oxides such as CeO_2 has received limited attention [46]. This section tackles this void by combining CeO_2-graphene composites in various proportions and assessing their efficacy for application in energy storage solutions.

The fabrication procedure of graphene-CeO_2 nanocomposites encompassed the amalgamation of cerium oxide with graphene at different weight ratios (1%, 3%, and 5%). The CeO_2 powder was initially mixed with water, subsequently accompanied by the incorporation of synthesized graphene. Following

a duration of sonication, the blend was stirred at ambient temperature to guarantee consistent distribution [47]. The ensuing composites were subsequently desiccated under vacuum conditions. The composites created were labeled as CeO_2G1, CeO_2G3, and CeO_2G5, reflecting the 1%, 3%, and 5% graphene proportions, respectively. The approach employed facilitated meticulous regulation of the graphene incorporation within every composite. An examination utilizing X-ray diffraction (XRD) was conducted to evaluate the crystalline arrangement of graphene, unadulterated CeO_2, and the graphene-CeO_2 composite materials. The diffraction pattern of pristine graphene displayed merely two peaks, relating to the (002) and (100) planes, signifying the hexagonal crystalline arrangement of graphene. The XRD patterns of the CeO_2 and CeO_2G composites exhibited numerous distinct peaks, affirming their crystalline characteristics. The incorporation of graphene, nonetheless, led to a displacement in the diffraction peaks, especially evident in the CeO_2G composites, indicating the existence of graphene within the composite, though with minimal influence on the crystalline arrangement of CeO_2. The alteration in peak locations can be linked to lattice stress induced by the interplay between graphene and CeO_2 [48].

Fourier transform infrared (FTIR) spectroscopy was employed to identify functional groups present in the CeO_2, graphene oxide (GO), reduced graphene oxide (rGO), and CeO_2G composites. The FTIR spectra of unadulterated CeO_2 exhibited a significant peak at 553 cm^{-1}, indicative of the Ce-O stretching vibration. Conversely, the FTIR spectra of graphene oxide exhibited peaks near 1700 cm^{-1} and 1072 cm^{-1}, which are associated with C=O and C-O stretching vibrations, respectively [28]. The diminished graphene oxide (rGO) spectra exhibited a lack of these peaks, validating the reduction of graphene oxide. The composites exhibited an extra peak at 525 cm^{-1}, signifying the existence of Ce-O bonds, which appeared less pronounced in the CeO_2G5 composite, implying a potential modification in bonding as the graphene content rises. Raman spectroscopy was performed to examine the structural robustness and excellence of the graphene embedded in the composites. The Raman spectrum of pristine graphene showcased the distinctive D and G peaks at 1343 cm^{-1} and 1577 cm^{-1}, correspondingly. The ratio of intensity (ID/IG) for these peaks was utilized to evaluate the quality of the graphene. The CeO_2G composites displayed similar bands, yet the peak intensities fluctuated based on the amount of graphene present. Remarkably, CeO_2G5 exhibited an extra peak at 1161 cm^{-1}, linked to cerium oxide, indicating a connection between CeO_2 and graphene. The CeO_2G1 composite, characterized by reduced graphene presence, displayed feeble Raman signals, suggesting inadequate composite development [29].

The morphology of the CeO_2G composites was investigated using Field Emission Scanning Electron Microscopy (FE-SEM). The CeO_2G1 composite displayed a fairly consistent distribution of graphene throughout the CeO_2 framework; however, the graphene layers seemed inadequately fused, positioned seemingly at random among the CeO_2 particles. With the elevation of graphene concentration in the CeO_2G5 composite, graphene layers aggregated, creating flake-like formations [34]. This grouping suggested a more robust connection between the graphene and CeO_2 with elevated graphene amounts, potentially affecting the total electrochemical efficacy by enhancing electron movement and augmenting surface area. Images captured through high-resolution transmission electron microscopy (HR-TEM) of CeO_2G5 revealed that CeO_2 nanocrystals were spread across the surface of graphene sheets, exhibiting particle dimensions varying from 10 to 50 nm [14]. Under elevated magnifications, the graphene layers seemed wrinkled, and CeO_2 was detected on the exterior as well as nestled between the graphene sheets. The HR-TEM visuals additionally disclosed that graphene comprised numerous layers, with as many as 15 layers detected in certain areas. The findings validated that the graphene within the CeO_2G5 composite had been successfully transformed from graphene oxide, as demonstrated by the distinct lattice fringes apparent in the images [29].

Energy dispersive spectroscopy (EDS) alongside elemental mapping was conducted to investigate the allocation of carbon, oxygen, and cerium throughout the composites. The elemental mapping revealed a uniform spread of these elements throughout the composites, accompanied by a significant rise in carbon levels as the proportion of graphene increased [23]. The elemental examination validated

that CeO_2G5 possessed the utmost percentage of carbon, signifying an elevated graphene presence. The even distribution of components indicates that the graphene was effectively integrated into the CeO_2 framework, facilitating the development of a thoroughly dispersed nanocomposite. X-ray photoelectron spectroscopy (XPS) was used to study the surface chemistry of the CeO_2G5 composite. The XPS spectra revealed the existence of both Ce^{3+} and Ce^{4+} oxidation states, which are typical of cerium oxide [24]. The carbon 1s spectrum displayed a prominent peak at 284.3 eV, indicative of sp2-hybridized carbon (C-C), accompanied by a satellite peak at 289.4 eV associated with carboxyl groups (C=O). The oxygen 1s spectrum revealed distinct peaks at 530.5 eV and 531.8 eV, associated with lattice oxygen and oxygenated groups, respectively. The findings demonstrate the effective incorporation of graphene within CeO_2 and the transformation of graphene oxide.

Cyclic voltammetry (CV) was employed to evaluate the electrochemical characteristics of the CeO_2G composites in a 1M H_2SO_4 electrolyte solution [25]. The findings demonstrated that CeO_2 displayed characteristics akin to capacitance, showcasing a notable rise in current when subjected to elevated scan rates. Conversely, the CeO_2G composites, especially CeO_2G5, exhibited remarkably improved electrochemical performance, showcasing more distinct redox peaks and elevated capacitance metrics. The CeO_2G5 composite exhibited the peak specific capacitance of 65.4 Fg^{-1} at a scan rate of 10 mV/s, suggesting that the augmented graphene content enhanced the electrochemical performance [13]. With elevated scanning velocities, the distinct capacitance of every composite diminished, which is characteristic of substances undergoing a diffusion-governed electrochemical mechanism [10]. The CeO_2G5 composite, nonetheless, exhibited superior capacitance at different scan rates, further validating that the incorporation of graphene boosts the electrode's overall efficacy. This conduct indicates that the electrochemical interactions of the composite are predominantly governed by diffusion rather than kinetic factors, probably owing to the enhanced conductivity enabled by graphene [14].

The distinct capacitance measurements for CeO_2 and CeO_2G composites were analyzed across various scan rates, showcasing a noticeable pattern: with the rise in graphene content, the specific capacitance exhibited an upward trend. CeO_2G1 exhibited the least capacitance, whereas CeO_2G5 showcased the utmost performance, credited to the elevated graphene concentration that facilitated superior electron transfer and improved electrochemical characteristics [44]. This discovery highlights the significance of graphene composition in influencing the electrochemical efficacy of nanocomposites. A reduction in particular capacitance alongside a rise in scan rate was noted for every composite [5]. This pattern is characteristic of substances involving diffusion-governed electrochemical mechanisms, where the speed of ion movement within the electrode substance restricts the capacitance at elevated scan velocities. Nonetheless, CeO_2G5 exhibited the smallest reduction in capacitance, suggesting that the graphene-CeO_2 hybrid provides superior ion movement and enhanced electrochemical resilience compared to unadulterated CeO_2 [11].

The graphene-CeO_2 nanocomposites exhibited remarkable electrochemical capabilities, with CeO_2G5 achieving the most outstanding outcomes attributed to its elevated graphene concentration. The successful creation of these composites was accomplished, and a range of characterization methods, such as XRD, FTIR, Raman spectroscopy, SEM, and TEM, validated the effective integration of graphene into CeO_2. Electrochemical evaluation via cyclic voltammetry demonstrated that the composites, particularly CeO_2G5, showcased improved capacitance, which was linked to the elevated graphene concentration. This research offers significant perspectives on the function of graphene in improving the electrochemical characteristics of metal oxide-derived nanocomposites, opening avenues for their application in cutting-edge energy storage solutions.

CONCLUSION

Graphene-CeO_2 nanocomposites were synthesized utilizing graphene at various mole percentages combined with cerium oxide through a direct solution blending technique. Structural investigations

were conducted utilizing powder X-ray diffraction examination. FTIR and Laser Raman spectral evaluations were conducted to ascertain the functional groups and to validate the creation of nanocomposites. The structure of the created compounds has been examined using FE-SEM and HR-TEM methods. Elemental mapping alongside energy dispersive spectrum was captured using the FE-EPMA method, and the mole ratios of different elements found in the composites were assessed. The analysis of X-ray photoelectron spectra was conducted, focusing on the binding states of different elements found within the composites. The electrochemical investigations reveal that the CeO_2G5 nanocomposite demonstrates commendable capacitance characteristics; however, the composites containing a reduced proportion of graphene failed to produce any notable influence on the electrochemical performance owing to the minimal quantity of graphene present.

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