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Synthesis of $SiO_2@TiO_2$ by Sol–Gel Method for Battery Electrode Applications

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Abstract: The $SiO_2@TiO_2$ nanostructured electrode material was effectively produced by a sol–gel technique. The produced material's structural, morphological, optical, and electrochemical properties were comprehensively examined using X-ray diffraction (XRD), UV-visible spectroscopy, scanning electron microscopy (SEM), and electrochemical methods. XRD examination verified the synthesis of crystalline rutile TiO_2 in conjunction with silica, demonstrating the successful creation of a $SiO_2@TiO_2$ composite structure. SEM investigation identified spheroidal particles exhibiting a size distribution from the nanometre to submicron scale. Energy-dispersive X-ray spectroscopy (EDX) verified the existence of Si, Ti, and O elements in a near-stoichiometric composition. UV–visible absorption analyses indicated an optical band gap of roughly 3.03 eV. The electrochemical performance was assessed by cyclic voltammetry (CV) and galvanostatic charge-discharge (GCD) tests within a three-electrode configuration. The electrode demonstrated a specific capacitance of 25 F g^{-1} at a scan rate of 5 mV s^{-1} and 37 F g^{-1} with a current density of 1 A g^{-1}, signifying battery-type behaviour. The results indicate that $SiO_2@TiO_2$ is a viable electrode material for energy storage applications.

Keywords: *$SiO_2@TiO_2$ Nanostructures; Sol-Gel Fabrication; Rutile TiO_2; Battery Electrode; Energy Storage.*

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I. INTRODUCTION

Accelerated industrialization and technical progress have resulted in a substantial rise in environmental pollution and energy consumption. The overproduction of industrial waste and the exhaustion of traditional energy resources have prompted academics to investigate sustainable and eco-friendly energy alternatives [1–4]. Semiconductor materials have garnered significant interest owing to their prospective applications in energy conversion, storage, and environmental cleanup [5–7].

Titanium dioxide (TiO_2) is a highly researched semiconductor material due to its remarkable chemical stability, non-toxicity, affordability, and appropriate band-edge positions [8–10]. Fujishima and Honda initially demonstrated the photocatalytic activity of TiO_2 in 1972 via photoelectrochemical water splitting under ultraviolet light [11]. Since that time, TiO_2 has been extensively utilized in applications like photocatalysis, dye-sensitized solar cells, gas sensors, and lithium-ion batteries [12–16]. Titanium dioxide primarily occurs in three crystalline forms: anatase, rutile, and brookite. Rutile TiO_2 demonstrates exceptional thermal stability and structural integrity, rendering it appropriate for electrochemical energy storage applications.

The integration of silica (SiO_2) with TiO_2 has been documented to augment structural stability, inhibit particle agglomeration, and boost electrochemical performance by increasing surface area and promoting ion diffusion [17–19]. Specifically, $SiO_2@TiO_2$ core-shell or composite architectures are attractive prospects for battery electrode materials owing to their enhanced mechanical stability and electrochemical longevity.

This study involved the synthesis of $SiO_2@TiO_2$ nanostructured material using a sol–gel technique. The structural, morphological, optical, and electrochemical properties were methodically examined to assess their applicability for battery electrode uses.

II. EXPERIMENTAL SPECIFICATIONS

➢ *Materials and Synthesis*

The $SiO_2@TiO_2$ composite was manufactured via a sol–gel technique to attain uniform molecular mixing of titanium and silica. Tetraethyl orthosilicate (TEOS) functioned as the silica precursor, whereas titanium (IV) isopropoxide (TTIP) acted as the titanium source. Ethanol served as the solvent, distilled water facilitated hydrolysis, and dilute hydrochloric acid (HCl) acted as a catalyst.

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TEOS was initially dissolved in ethanol while being continuously stirred magnetically to achieve a transparent solution. A measured quantity of distilled water, accompanied by several drops of weak HCl, was added incrementally to commence hydrolysis, and the mixture was agitated for 30–45 minutes to produce a homogeneous silica sol. TTIP was independently dissolved in ethanol and continually swirled to avert premature hydrolysis.

The TTIP solution was thereafter introduced dropwise into the silica sol while being vigorously stirred. The pH of the mixture was sustained within the acidic range (pH = 2–3) to facilitate regulated condensation and consistent dispersion of Ti species throughout the silica matrix. The resultant sol was agitated for 2–3 hours till gelation transpired.

The resultant gel was aged at ambient temperature for 12–24 hours, thereafter dried at 80–100 °C for 10–12 hours to provide a xerogel powder. The dried powder underwent calcination at 650 °C for 2–3 hours in a muffle furnace, with a heating rate of 5 °C per minute, to facilitate crystallization and enhance the generation of rutile TiO_2.

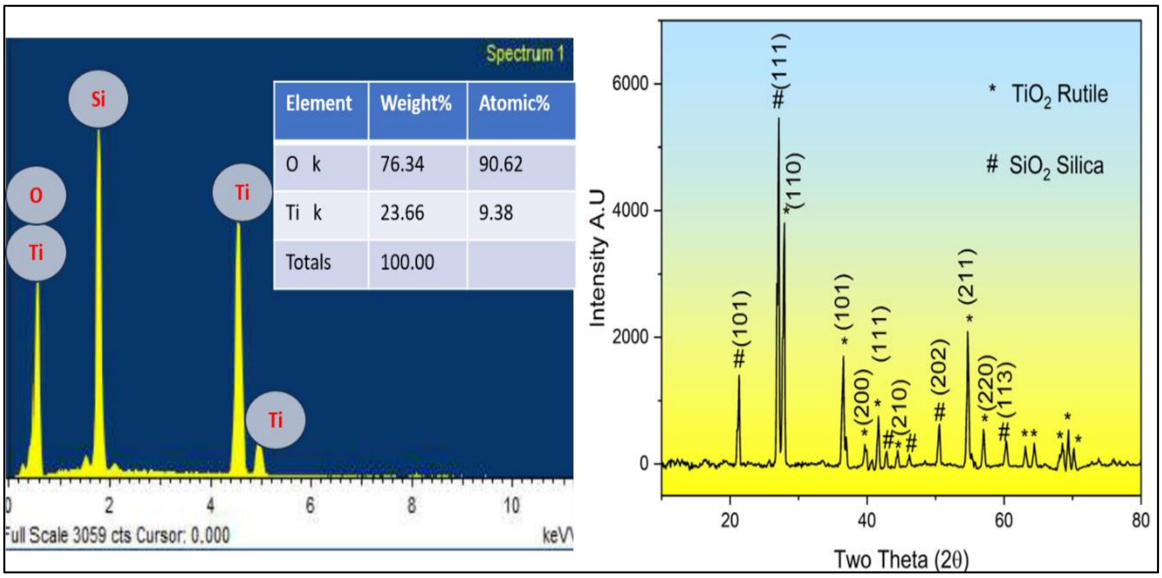
➢ *Techniques for Characterization*

Phase identification and crystallinity were assessed via X-ray diffraction (XRD) utilizing Cu Kα radiation (λ = 1.5406 Å). Morphological examination was conducted via scanning electron microscopy (SEM), and particle size distribution was assessed using ImageJ software. The optical characteristics were examined by UV-visible spectroscopy. Electrochemical assessments, comprising cyclic voltammetry (CV) and galvanostatic charge–discharge (GCD), were conducted utilizing a three-electrode configuration.

III. FINDINGS AND ANALYSIS

➢ *X-Ray Diffraction Examination*

The crystalline structure of the produced $SiO_2@TiO_2$ composite was examined via XRD within the 2θ range of 10–80°. The diffraction pattern in Fig.1 displayed distinct and well-defined peaks, signifying high crystallinity. The most prominent peak at 2θ = 27.4° is attributed to the (110) plane of rutile TiO_2. Additional peaks at 36.1°, 41.2°, 54.3°, 56.6°, and 69.0° correspond to the (101), (111), (211), (220), and (301) planes, respectively, aligning with JCPDS card No. 21-1276.



Element	Weight%	Atomic%
O k	76.34	90.62
Ti k	23.66	9.38
Totals	100.00	

Fig.1: XRD, EDS Pattern of $SiO_2@TiO_2$

Diffraction peaks identified at 20.8°, 26.6°, 39.5°, 45.8°, and 60.0° were correlated with the crystalline silica phase (SiO_2) (JCPDS No. 46-1045). The lack of impurity peaks verifies the high phase purity of the manufactured composite.

The lattice parameters of rutile TiO_2 were determined to be a = 4.59 Å and c = 2.96 Å, aligning with established values. The average crystallite size, as determined by the Scherrer equation, was calculated to be 20–25 nm.

➢ *Scanning Electron Microscopy Analysis*

SEM pictures in fig.2 captured at magnifications between 5 µm and 0.5 µm displayed spheroidal particles of submicron size. Analysis of particle size distribution via ImageJ software revealed sizes between around 28 nm and 476 nm, with a mean particle size of almost 500 nm. The existence of diminutive particles is advantageous for battery applications owing to augmented surface area and improved electrochemical activity.

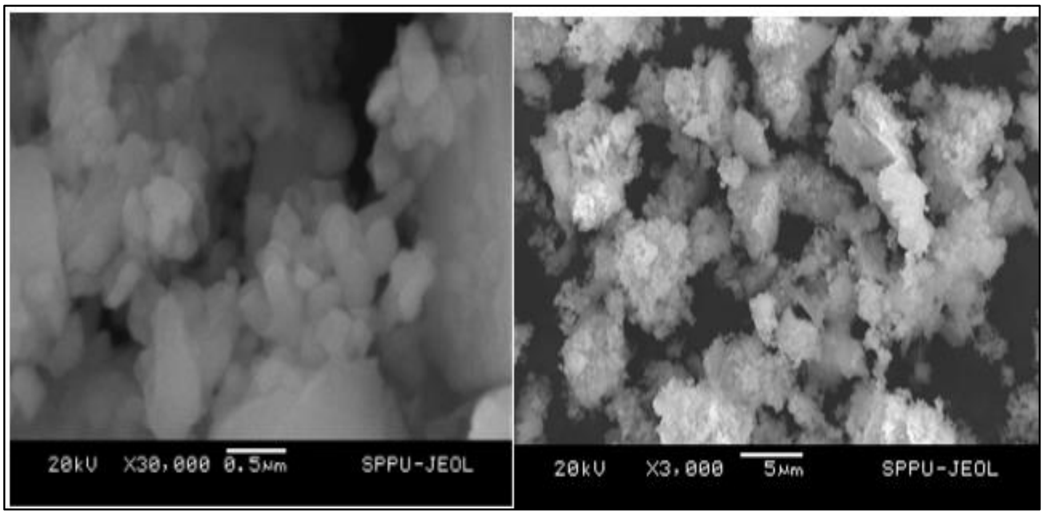


Fig.2: SEM of $SiO_2@TiO_2$ at Low and High Magnification

- *Ultraviolet–Visible Absorption Investigations*

The UV-visible absorption spectra exhibited significant absorption in the ultraviolet range, with a peak near 300 nm. The optical band gap, determined by the Tauc plot, was approximately 3.03 eV, aligning with rutile TiO_2.

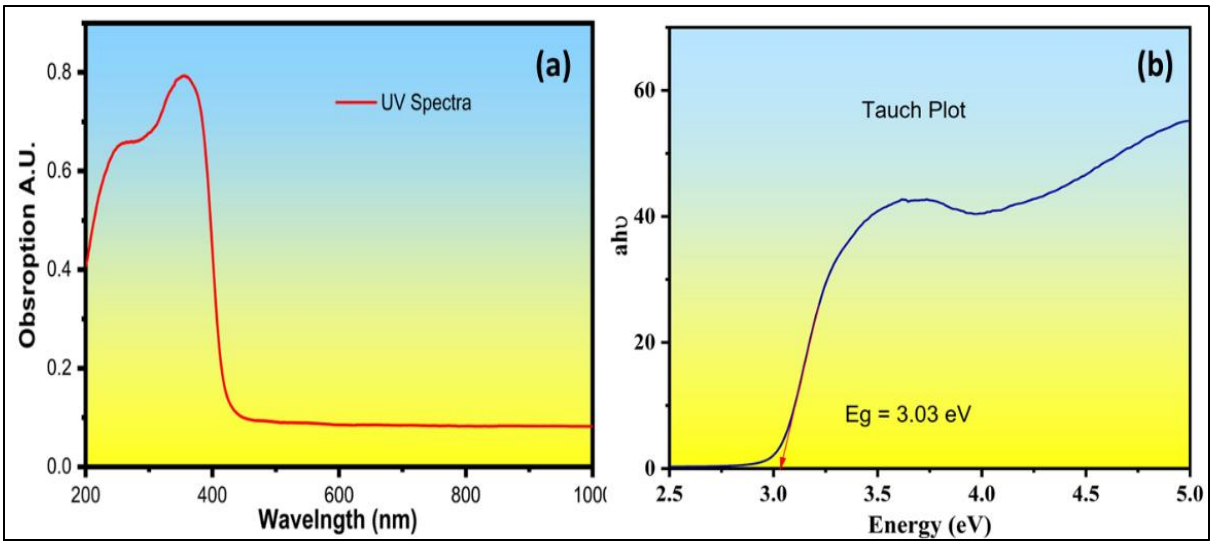


Fig.4: (a) UV Spectra (b) Tauc Graph of $SiO_2@TiO_2$

- *Electrochemical Performance*

Electrochemical investigations were conducted utilizing a three-electrode setup. Cyclic voltammetry demonstrated battery-type behaviour, achieving a maximum specific capacitance of 25 F g^{-1} at a scan rate of 5 mV s^{-1}. Galvanostatic charge–discharge experiments revealed a substantial specific capacitance of 37 F g^{-1} at a current density of 1 A g^{-1}, signifying exceptional charge storage capacity. The SC values calculated from CV and GCD curves are shown in table 1 and illustrated in fig. 4.

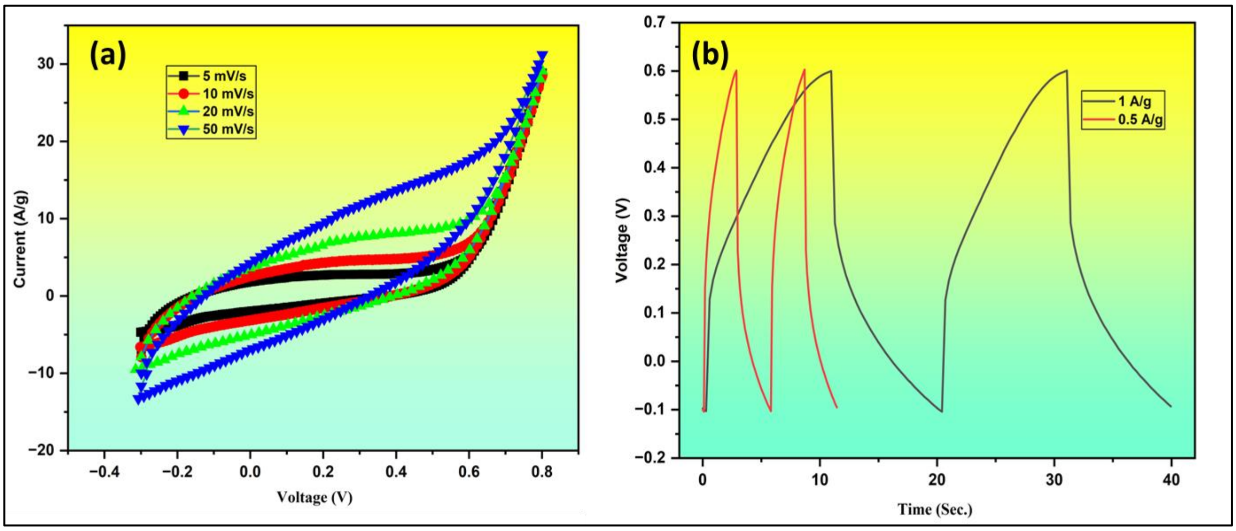


Fig.4: (a) Cyclic Voltamogram (b) GCD Curve of $SiO_2@TiO_2$

Table 1: Specific Capacitance from CV Curve (Right), GCD Curve (Left)

Scan Rate (mV/s)	Specific Capacitance (F/g)
5	25
10	14.975
20	9.625
50	5.4775

Current Density (A/g)	Specific Capacitance (F/g)
1	37
0.5	58.37

IV. CONCLUSION

$SiO_2@TiO_2$ nanostructured material was effectively produced using a sol–gel technique. Structural study verified the synthesis of nanocrystalline rutile TiO_2 on a silica substrate. The composite displayed advantageous morphological, optical, and electrochemical characteristics, exhibiting battery-type behaviour with elevated specific capacitance. The results indicate that $SiO_2@TiO_2$ is a viable electrode material for sophisticated energy storage applications.

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