

Synthesis of Nanocrystalline SiC via Sol–Gel Method: Effect of Low-Temperature Heat Treatment on Phase Formation and Nanostructures

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Abstract: Silicon carbide (SiC) is one of the most important silicon-based compounds, owing to its favourable physical, chemical, and biological properties, and is widely employed in various fields, including electronics, the chemical industry, and quantum computing. Several methods have been reported for synthesizing SiC nanoparticles, including chemical vapor deposition (CVD), hydrothermal synthesis, carbothermal reduction, and sol–gel processing. Among these, the sol–gel method has attracted significant attention due to its high yield, process controllability, biocompatibility, accessibility of precursors, and ability to produce nanoparticles. In this study, SiC nanosized powders were synthesized through the sol–gel route combined with carbothermal reduction, using tetraethyl orthosilicate ($(C_2H_5)_4SiO_4$) and sucrose ($C_{11}H_{22}O_{11}$) as the silicon and carbon sources, respectively. The silica/sucrose composite was subjected to carbothermal reduction under an argon atmosphere at 10 mTorr in a vacuum furnace at 1350°C for 3 h. The structural properties of the synthesized SiC nanopowders were analyzed using X-ray diffraction (XRD), while their optical characteristics were investigated through FTIR, diffuse reflectance spectroscopy (DRS), and photoluminescence (PL). This work demonstrates a greener, lower-temperature route to phase-controlled SiC nanoparticles with optically active vacancy centers.

Keywords: Silicon carbide nanoparticles, Sol–gel, Carbothermal reduction.

1. INTRODUCTION

Silicon carbide (SiC), one of the earliest known semiconductors, has recently gained renewed attention for its ability to operate under extreme conditions, including high temperatures, high power, high frequency, and intense radiation. It has also been widely applied in advanced electronic, optoelectronic, and quantum technologies due to its ability to generate single photons. SiC is a wide-bandgap semiconductor that exists in various polytypes, the most common of which are 4H, 6H, 15R, and 3C. The wide bandgap and remarkable optical emission features (such as donor–acceptor pairs and colour centres) have broadened the material's optical applications [1, 2]. The existence of multiple polytypes further enables the design of devices adaptable to advanced environments and suitable for quantum photonic platforms [3]. In recent years, the presence of color centers—particularly silicon vacancies (V_{Si})—in the SiC lattice has established it as a promising platform for room-temperature single-photon emission and spin-based qubits. Unlike nitrogen-vacancy (NV) centers in diamond, SiC offers CMOS compatibility, paving the way for integration of quantum and classical technologies. However,

the efficient synthesis of SiC nanoparticles with simultaneous control over particle size, phase purity, and quantum properties remains a significant challenge [4]. Several approaches have been developed for SiC nanostructure synthesis, such as carbothermal reduction [5], chemical vapor deposition (CVD) [6], direct carbonization of metallic silicon [7], high-energy mechanical milling [8], and sol–gel processing. Most of these methods, however, either require extremely high processing temperatures (above 2000°C) or involve toxic precursors such as HF and HNO_3 , rendering the synthesis costly, hazardous, and environmentally undesirable [9]. Furthermore, specialised equipment or complex technology requirements in some methods increase production costs and often result in low efficiency and high impurity levels. By contrast, the sol–gel method, an aqueous chemical process widely employed for the synthesis of inorganic and ceramic materials, has been widely used to produce metal oxides, nanocoatings, aerogels, and nanocomposites. Starting from a metal precursor (commonly a metal alkoxide or salt), which largely determines the final structure, the sol–gel process proceeds through sequential steps: hydrolysis (formation of reactive groups), polymerization (formation of metal–oxide bonds,

controlling particle size, surface area, and structural homogeneity), sol formation, gelation (stabilizing the final structure), drying (influencing porous or dense morphology), and thermal treatment (enhancing crystallinity). In recent years, sol–gel processing has emerged as a highly attractive method, offering high-purity products, molecular-level mixing, and favorable chemical reactivity [10]. This approach enables the synthesis of nanocrystallites smaller than 100 nm at relatively low temperatures (below 1500°C). For instance, Najafi et al. (2022) synthesized β -SiC at 1500°C by optimizing acidic pH conditions during the sol–gel process. When pH was below 4, the solution became more stable, and β -SiC nanoparticles as small as 10 nm were achieved; however, in the pH range 4–7, solution stability decreased, and particle size increased significantly. Nevertheless, the use of HF remained a limiting factor [11]. Similarly, Li et al. (2023) employed tetraethyl orthosilicate (TEOS) and phenolic resin as precursors, with oxalic acid (OA) and hexamethylenetetramine (HMTA) as catalysts [12]. Their findings revealed that OA primarily regulated solution pH and influenced TEOS hydrolysis, whereas HMTA facilitated coagulation and gelation. Using this dual-catalyst system, SiC nanoparticles of ~60 nm were synthesised at 1500°C, but polytype control was not achieved [12]. Zhang et al. successfully synthesized SiC nanoparticles using a carbon–SiO₂ mixture [13]. This approach significantly reduced particle size and enabled synthesis at much lower temperatures than the Acheson process,

yielding β -SiC nanoparticles of ~30 nm at 1500°C, though particle agglomeration was observed. Other methods, such as plasma pyrolysis by Garcia (2019) at 1700°C [14], carbothermal–mechanical synthesis by Chen (2021) at 1550°C [15], and CVD by Wang (2022) at 1580°C [16], not only required higher temperatures but also produced particles predominantly larger than 100 nm.

Although sol–gel synthesis provides advantages such as compositional uniformity and precise structural control, challenges remain. The most critical factor is the accurate adjustment of the precursor ratio between silica and the carbon source, as an imbalance can result in residual silica or free carbon, reducing the purity of the SiC phase. Moreover, complete conversion of the gel to SiC generally requires high temperatures (typically above 1400°C), which may promote excessive grain growth or the crystallization of unwanted phases. Intermediate phases such as Si–O–C may also form, which, if incompletely reacted, degrade the final material properties. In addition, cracks or nonuniform porosity may develop during gel drying due to rapid solvent evaporation, reducing structural integrity. These issues highlight the need for precise control and optimisation throughout the sol–gel process [17]. Figure 1 provides a schematic overview of the key sol–gel steps and resulting products.

This figure has inspired many visual representations of the sol-to-gel transition, and similar diagrams are frequently used in the literature to illustrate the sol–gel process [18].

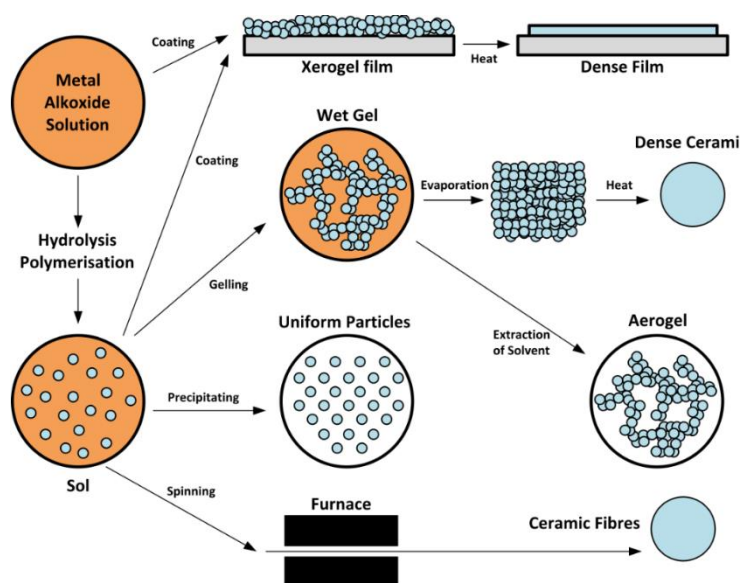


Fig. 1. Schematic view of the sol-gel process [18]

As mentioned above, all previous methods required synthesis temperatures at least 50–250°C higher than in this work and produced particles larger than 35 nm. Furthermore, none simultaneously achieved three critical goals: (i) controlled polytype synthesis (3C/6H), (ii) engineering of quantum vacancies (silicon-vacancy centers) for single-photon emission, and (iii) elimination of toxic chemicals. Despite previous reports on SiC nanomaterials spanning the micro- to nanoscale, precise control over nanostructure growth remains a critical challenge. In this research, we demonstrate a modified sol–gel method using TEOS and sucrose in an ethanol-based medium, with optimized calcination at 1350°C. This approach offers several unprecedented achievements: (i) reduction of the synthesis temperature to 1350°C, (ii) formation of SiC nanoparticles in the 15–30 nm range, (iii) generation of silicon-vacancy quantum centers with verified single-photon emission at 860 nm (confirmed by PL analysis); and (iv) complete elimination of hazardous corrosive chemicals such as HF, by employing an ethanol-based gel system, thereby reducing environmental risks and enhancing scalability.

2. EXPERIMENTAL PROCEDURES

Ethanol (all supplied by Merck, Germany) was used as a precursor. The experimental steps are schematically illustrated in Figure 2. As shown, first, a sol containing 2.2 ml of TEOS, about 10 ml of ethanol, 0.5 ml of distilled water, and 2 g of sucrose was prepared. Then, 2 mL of hydrochloric acid was added as a catalyst to the solution. This solution was completely homogeneous and uniform for 3 hours by magnetic stirring.

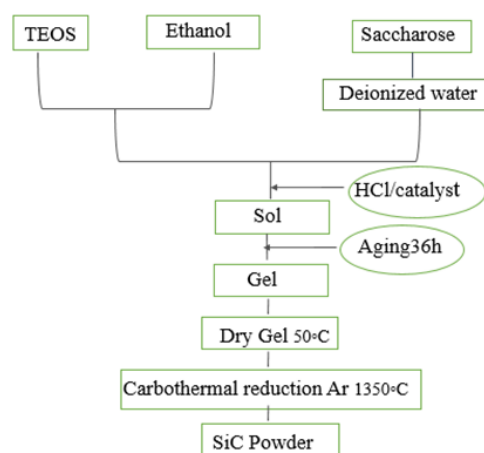


Fig. 2. Schematic of the preparation of nanoparticles by the sol-gel method

The resulting sol was transparent and colorless. The prepared sol was left to undergo gelation for 40 h at room temperature, after which it was dried at 50°C, yielding a dark brown powder. The obtained powder was then placed in an alumina crucible and heat-treated in a vacuum furnace under an argon atmosphere at 1350°C for 3 h. The furnace chamber was initially evacuated to approximately 2×10^{-2} mbar. Argon gas was then introduced at a flow rate of about 100 sccm. After allowing the system to stabilize for approximately 10 minutes, the heating program began. The temperature was first raised to 1100°C at a heating rate of 5°C/min, and then increased from 1100°C to 1350°C at a controlled rate of 3°C/min. For cooling, the furnace was switched off and allowed to return to room temperature naturally over several hours in ambient conditions. The final powder appeared dark. To characterize the synthesized powder, several analytical techniques were employed. The bonding types among the sample constituents were investigated using Fourier-transform infrared spectroscopy (FTIR) in the range of 400–4000 cm^{-1} . Phase identification and average crystallite size determination were performed via X-ray diffraction (XRD), employing the Debye–Scherrer method. For accurate grain size measurements, a Zeiss EM900 transmission electron microscope (TEM) was used. The morphology of the particles was examined using field-emission scanning electron microscopy (FESEM, Zeiss, model Sigma 30-HV). Photoluminescence (PL) spectra were recorded with 200 nm excitation to evaluate the optical emission characteristics of the synthesised SiC. Diffuse reflectance spectroscopy (DRS) was carried out with an Avantes Avaspec-2048 spectrometer in the range of 200–1100 nm. The Kubelka–Munk function was applied to convert the diffuse reflectance data for bandgap determination. Finally, Raman spectroscopy (Avantes, model uRaman-532-Ci) with a 532 nm laser over 100–1400 cm^{-1} was employed to analyse lattice vibrations and confirm the crystalline structure of the synthesised SiC nanoparticles.

3. RESULTS AND DISCUSSION

3.1. Structural Study of Silicon Carbide Nanoparticles

Initially, the X-ray diffraction spectrum of the resulting powder was taken. According to Figure 3, no peaks related to carbon gas or SiO_2 are

observed in the sample, which indicates that the carbothermal reduction reaction is almost complete. The synthesised sample shows peaks at 2θ of 33.6, 35.8, 41.4, 60.04, 71.8, and 75.5 degrees. As shown in Figure 3, the 2θ values and the intensities of the main peaks at 35.6, 41.4 and 60.04° are in good agreement with the established values (JCPDS Card No. 29-1129) and can be attributed to the diffraction of the β -SiC (111), (200) and (220) planes, respectively [19]. The pronounced, sharp peaks indicate that the SiC nanoparticles exhibited good crystallinity. Therefore, β -SiC was confirmed as the primary crystalline phase of the nanosized particles with a cubic structure. Also, the 2θ values and the main peaks at 33.6, 35.8, 71.8 and 75.5° degrees are in good agreement with the determined values (JCPDS card number 29-1131) and can be attributed to the diffraction of (101), (102), (116) and (0012) α -SiC planes, respectively, which indicates the presence of a hexagonal structure alongside the cubic structure [20].

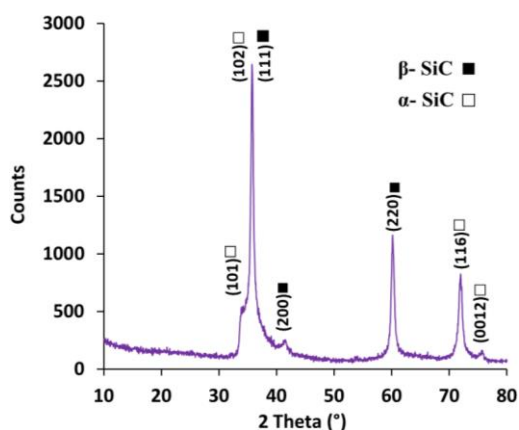


Fig. 3. X-ray diffraction spectrum showing the formation of bi-structured α -SiC and β -SiC nanoparticles

Using the X-ray diffraction spectrum and the Scherrer equation, the crystal dimensions can be determined as follows [21]:

$$D = (k \times \lambda) / (\text{FWHM} \times \cos\theta) \quad (1)$$

In this relation, D is the crystal size, k is the lattice constant (approximately 0.89), and λ is the wavelength of the incident beam, which is here the wavelength of the copper target and is considered to be 1.5406 angstroms. Also, θ is the diffraction angle, and FWHM shows the width at half maximum of the diffraction peak. Based on Scherrer's calculations, the crystal sizes for each phase in the sample are shown in Table 1.

Table 1. The determined average crystallite size of the sample obtained using X-ray diffraction spectrum

Poly type	2 θ	FWHM	D	Average
β	35.6	0.508	~16 nm	~14 nm
	41.4	0.62	~14 nm	
	60.04	0.819	~12 nm	
α	35.6	0.508	~16 nm	~16 nm
	71.8	0.62	~14 nm	
	75.5	0.52	~19 nm	

Based on these calculations, the average crystal size was determined to be about 15 nm, a desirable result compared to the work of others and the expensive, even risky, processes. The FTIR spectrum of the synthesized SiC nanoparticles is shown in Figure 4. An absorption peak was observed at 847 cm^{-1} , corresponding to the characteristic Si-C bond vibrations, formed by the bonding of carbon from sucrose with hydrolyzed silicon from TEOS at the molecular level. In addition, weak and broad absorption bands were detected in the 2300–3700 cm^{-1} region, which is almost certainly due to O-H stretching vibrations from adsorbed water [22].

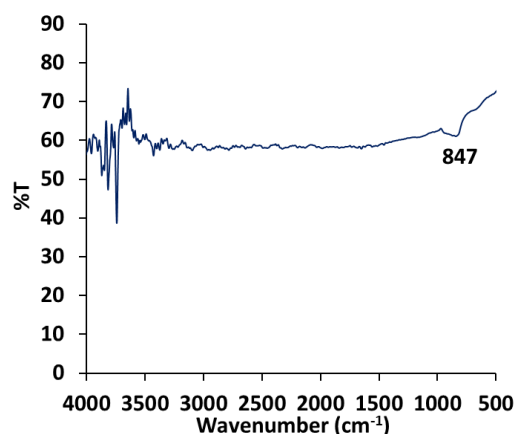


Fig. 4. Infrared Fourier transform curve of silicon carbide nanoparticles

Figure 5 shows the Raman spectrum of the synthesized powder. The presence of a characteristic peak around 782 cm^{-1} is observed, which is attributed to the transverse optical phonon (TO) mode in the cubic and hexagonal silicon carbide (6H-SiC, 3C-SiC) structure. This peak is a key indicator of the β -SiC and α -SiC phases and indicates their presence in the sample. In addition, a peak appears in the 930–970 cm^{-1} range, which is associated with the longitudinal optical (LO) phonon mode and is present in both 3C and 6H structures, in good agreement with the XRD analysis. In the higher wavenumber region (1330–1510 cm^{-1}),

peaks are also observed. They are attributed to second-order Raman processes, such as TO or TO+LO combinations, and are usually observed in structures containing free or amorphous carbon. The presence of these strong, distinct peaks in the Raman spectrum indicates the desirable crystallinity and high quality of the synthesised nanoparticles [23].

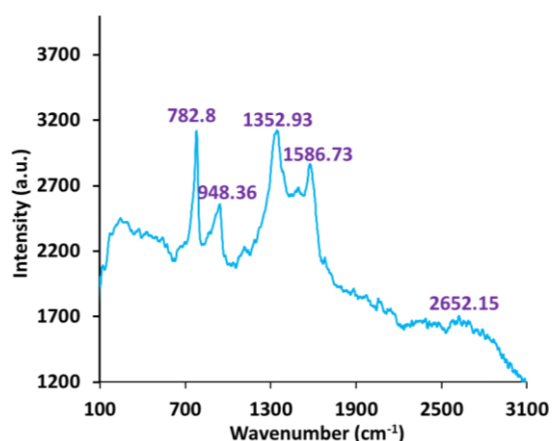


Fig. 5. Raman curve of synthesized silicon carbide nanoparticles

As shown in Figure 6, the morphology and structure of the synthesized SiC nanoparticles were studied using FESEM and TEM techniques. The TEM image in Figure 6(a) is a dark field image with (111) orientation of the selected area of the sample. The SEM images in Figures 6(b) and 6(c) show the formation of regular and spherical SiC nanoparticles. As shown, the synthesised

nanoparticles exhibit a relatively uniform size distribution in the 30–40 nm range [24]. Figure 6(c) shows the EDX spectrum of the synthesized silicon carbide nanoparticles. As shown by the percentage of elements, in addition to the formation of silicon carbide, a large number of carbon atoms are present in the sample, confirming the presence of free carbons at 1330 and 1510 in the Raman diagram (Figure 5) [25]. The low oxygen (O) content could be due to surface oxygen adsorption or the formation of a thin surface oxide layer, both of which significantly reduce the likelihood of SiO₂ bond formation. XRD (Figure 3) and Raman (Figure 5) analyses also confirm the absence of SiO₂.

3.2. Optical Study of Silicon Carbide Nanoparticles

To investigate the optical properties, the synthesised sample was analysed by photoluminescence spectroscopy at an excitation wavelength of 200 nm (Figure 7). As shown in this figure, there are distinct peaks in the UV-Vis and IR regions, each associated with different mechanisms of radiative recombination and atomic vacancies. The intense peaks at 400 and 600 nm are related to the first and second harmonics of the excitation wavelength. The sharp peak observed at about 361 nm is located in the ultraviolet region and is attributed to band-edge recombination in the synthesized sample structure. In the 4H-SiC structure, the indirect energy gap of about 3.3 eV corresponds to a wavelength of approximately 375 nm.

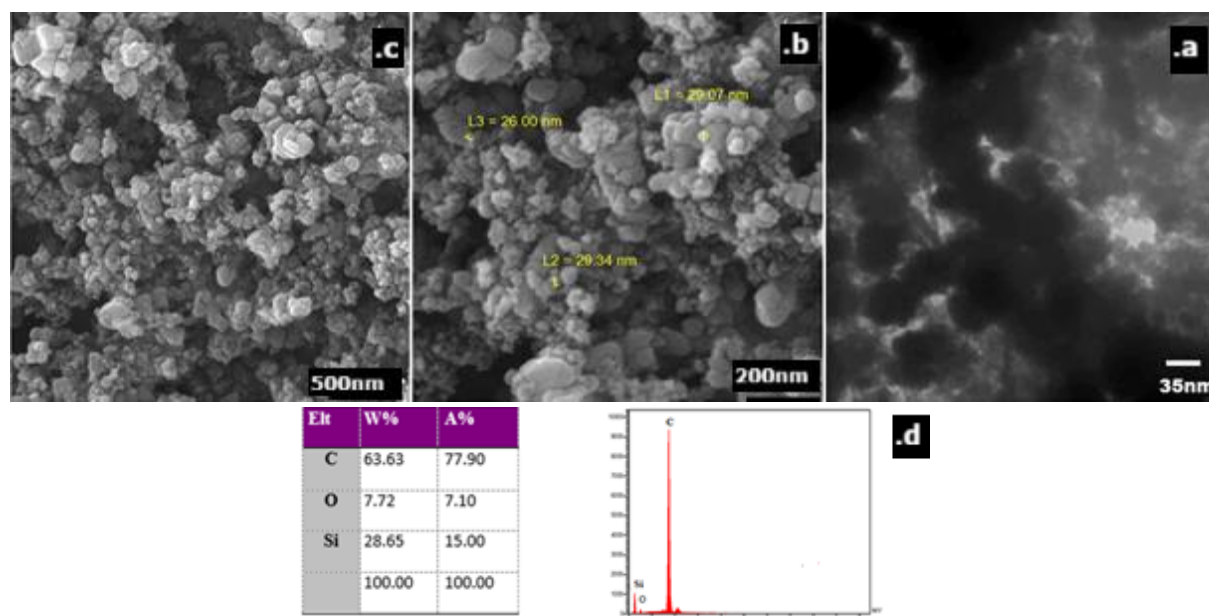


Fig. 6. a) TEM, b) and c) FESEM images, d) EDX of silicon carbide nanoparticles

The slight shift of this peak towards shorter wavelengths (361 nm) indicates quantum confinement in the nanoparticles, which increases the band gap. Therefore, this emission is mainly associated with the α -SiC phase and is influenced by the small particle size [26]. The peak observed at 461 nm is in the visible-blue region and is attributed to lattice defect emission. Stacking faults and layer discontinuities in hexagonal polytypes (α -SiC) can produce such optical emissions in the visible region. Hence, this peak is also attributed to structural defects in α -SiC [27]. The peak observed at around 731 nm is due to the donor-acceptor transitions in the β (3C-SiC) structure. These transitions result from the recombination of electrons trapped in donor levels with holes in acceptor levels within the energy gap, often due to intrinsic lattice defects such as silicon or carbon vacancies. The presence of this peak in the PL spectrum indicates that part of the sample forms a cubic polytype (β -SiC), and structural defects in this phase play an important role in generating red emission [28]. In the near-infrared region, peaks appear at around 820, 829, and 872 nm. These peaks, due to silicon vacancies (VSi) and the zero-phonon lines (ZPL) of the V1 centre in 4H-SiC, are located. The shift of these peaks towards shorter wavelengths can be due to electron-phonon interactions. Therefore, these emissions are directly related to VSi centers in the α phase, (4H-SiC). In general, the PL spectrum shows that both α and β phases are present in the synthesized nanoparticles. Still, the α phase predominates over the β phase, as indicated by structural analyses. The IR emissions, which are mainly due to vacancy centers, are of particular importance for applications in quantum technologies and biosensors [29-32].

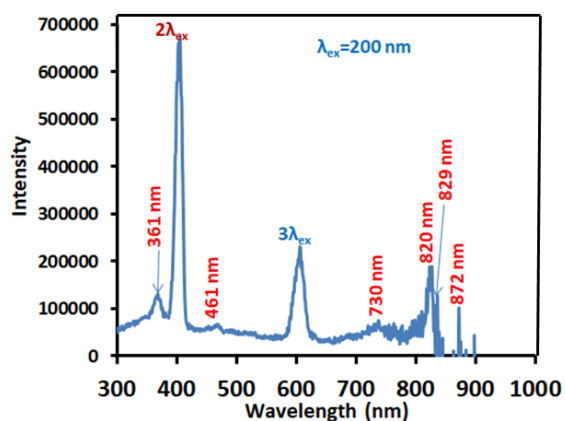


Fig. 7. PL spectrum of silicon carbide nanoparticles with 200 nm excitation

In the ultraviolet-visible region was also used. Given the sample's powdery nature and nanoscale structure, the direct UV-Vis absorption method could have yielded unrealistic results due to intense light scattering. For this reason, to eliminate the scattering effects, the diffuse reflectance was transformed into the Kubelka-Munk function, which is defined as $F(R) = (1-R)^2/2R$, where R is the relative reflectance. To determine the band gap energy, the Tauc relation for semiconductors with an indirect band gap and a power-law dependence on the square of the photon energy was used. In this method, the $(F(R) \cdot E)^{1/2}$ plot was plotted in terms of photon energy, and the band gap energy (E_g) was determined by extrapolating the linear part of the curve to the horizontal axis (Figure 8). The band gap energy obtained for the sample is about 3.2 eV, which corresponds to a wavelength of about 388 nm and is in relative agreement with the values reported for the β -SiC and α -SiC phases [33,34]. This value shows a slight increase relative to the band gap of the cubic face-centred cubic structure, which can be attributed to quantum confinement induced by the reduction in crystal size. When the crystal dimensions are reduced below the Bohr radius of the exciton, the electronic structure of the material changes, and the band gap increases. On the other hand, the relatively high synthesis temperature (1350°C) provides conditions for the formation of a stable hexagonal (4H/6H)-SiC phase alongside 3C-SiC, without leading to significant grain growth.

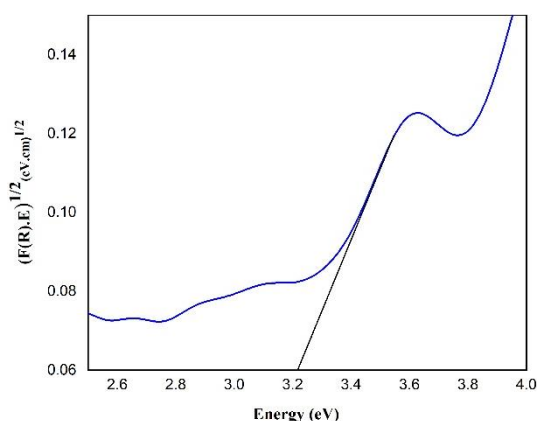


Fig. 8. DRS spectrum of silicon carbide nanoparticles for band gap determination

Overall, DRS results show that both α and β phases are present in the sample, but the dominant band gap measured is due to the α (4H/6H)-SiC phase. This finding is also consistent with the PL

spectroscopy results, confirming that the observed band-edge emission in the ultraviolet region is mainly attributed to the α -SiC structure. This indicates that maintaining nanoscale dimensions, along with the material's desired crystallinity, plays an important role in tuning its optical properties [35].

3.3. Comparison of the Results Obtained with the Work of Others

Table 2, compares the results obtained from this study with the results of other researchers.

As shown in Table 2, all previous methods have a temperature range of 100-250°C, higher than that in this study, and the particle size is greater than 35 nm. In addition, none of them have simultaneously achieved the three goals of controlling the 3C/6H polytype, preparing nanosized particles with silicon vacancies, and removing toxic materials. Despite the synthesis of SiC nanomaterials with dimensions ranging from micrometres to nanometers using various methods, controlling the growth of SiC nanostructures remains a significant challenge for the development of nanotechnology. In this study, by using a modified sol-gel method based on tetraethyl orthosilicate and sucrose in an ethanolic environment and optimizing the calcination conditions of 1350°C, we have achieved desirable results: Reducing the synthesis temperature to 1350°C, lower than all the studies compared in Table 2, forming SiC nanoparticles (with a size of 20-30 nm), the smallest size reported in similar

methods, creating Si vacancy quantum dots with single-photon emission at 872 nm confirmed by (PL) and complete removal of toxic corrosive materials such as (HF) and using an ethanol-based gelation system, to reduce environmental risk and increase scalability.

4. CONCLUSIONS

In this study, silicon carbide nanoparticles were successfully synthesized by the sol-gel method and carbothermal reduction at 1350°C. XRD and Raman results revealed the simultaneous presence of both α and β polytypes and indicated a favourable crystallinity. FESEM and TEM images showed that the synthesized particles had a uniform morphology and a size of about 30-40 nm. DRS analysis showed a band gap of about 3.2 eV, in perfect agreement with the band-edge peak in PL. Also, the PL spectrum revealed peaks associated with lattice defects and vacancy centres, in addition to band-edge emission, which, along with structural data, helps provide a deeper understanding of the phase composition and optical properties of the sample. The overlap between the results of different analyses indicates that the synthesis process was reliable and that the final product was of high purity and quality. On the other hand, the observed optical and structural properties indicate the broad capabilities of the synthesized SiC nanoparticles for application in advanced fields such as quantum technology.

Table 2. Comparison of recent studies with the present work

Study (Year)	Synthesis method	Temperature (°C)	Particle Size (nm)	Limitations
Najafi et al. (2022) [11]	Acid-catalyzed sol-gel	1500	~50	Requirement of HF, lack of polytype control
Li et al. (2023) [12]	Sol-gel + catalyst	1550	~60	Mixed phases
Garcia et al. (2019) [14]	Plasma pyrolysis	1700	>200	High equipment cost
Chen et al. (2021) [15]	Carbothermal + MA	1550	>100	High energy consumption
Wang et al. (2022) [16]	CVD	1580	100-300	Toxic precursors
Patel et al. (2023) [36]	Fe-assisted sol-gel	1520	~35	Iron contamination
This work	Modified sol-gel	1350	15-30	-----

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