

Research Article Sol-Gel Synthesis and Characterization of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq 1.0$) Nanoparticles

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The $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5) nanoparticles have been synthesized by sol-gel technique. X-ray diffraction, scanning electron microscopy, optical absorption spectroscopy, and electron paramagnetic resonance spectroscopy were used to characterize the sample. The X-ray diffraction results indicate the formation of nanocrystalline materials in tetragonal lattice with P4₂/nnm space group. The identical distribution of elements were confirmed by scanning electron microscopy with energy dispersive X-ray spectrometry and X-ray mapping. Electron paramagnetic resonance lineshapes of the samples are obtained at various (13 K, 77 K, and 300 K) temperatures. The isotropic lineshapes of the sample B1 are attributed to dipole-dipole interaction of Ti³⁺ ions. The incorporation of Al³⁺ ions into the sample B2–B5 the isotropic nature of the lineshapes are collapsed due to the distraction in crystal field. Optical absorption spectra results reveal the presence of Ag-TiO₂ nanoparticles.

1. Introduction

Titanium-based nanoparticles have been studied intensively owing to its widespread industrial applications such as cosmetics [1], ceramics [2], superconductivity [3], solar cells [4], magnetic [5], shape memory alloys [6], and photocatalysis [7]. However, the basic research and industrial development of TiO₂ containing samples are hot topics because of its extensive potential importance, and existing difficulties are still tough to solve [8, 9]. The key problems of TiO₂ containing samples show short wavelength excitation, it may crossover by the addition of noble metals such as Ag, Au, Pt. Among them, silver and gold nanoparticles are promising due to the remarkable chemical stability and a characteristic absorption peak in visible wavelength range [10-12]. The enriched photoactivity in visible light is attributed to the silver nanoparticles act an electron traps aiding electron-hole separation [13].

In favor of some specific advanced application (optics, sensor, photocatalysis, etc.), materials are appropriate to shape as films, tubes, and fibers but in most cases TiO_2 samples are required as a powder. Various wet chemical

methods such as coprecipitation [14], hydrothermal [15, 16], sol-gel technology, and combustion method [17, 18], have been developed for materials synthesis. Since the sol-gel technology is widely used and it is an effective process to produce Ag-TiO₂ nanoparticles is due to its advantages of low processing cost, energy efficiency, high production rate, and rapid productivity of fine homogeneous powder [19–23]. Characterization of materials are the root of modern technology for further progress and future requirements of advance materials. The structural and chemical properties of the materials have been progressively investigated by several advanced techniques. Electron paramagnetic resonance (EPR) spectroscopy is a very powerful and sensitive technique to ascertain the electronic structures of materials associated with unpaired electrons.

The present work focus on the synthesis of $Ag_{3(2+x)}$ $Al_x Ti_{4-x}O_{11+\delta}$ ($0 \le x \le 1.0$; B1–B5) nanoparticle by solgel process at room temperature. The aim is to determine the structure and property of the samples by X-ray diffraction, scanning electron microscopy (SEM), differential scanning colorimetry (DSC), optical absorbance spectroscopy, EPR spectroscopy, and vibrating sample magnetometer (VSM).

2. Experimental

2.1. Synthesis of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq 1.0$) Nanoparticles. The $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ nanoparticles were prepared by the sol-gel technology. Reagent grade chemicals were used without further purification. The calculated amounts of $Al(NO_3)_3$, $Ag(NO_3)_2$ and TiO_2 were mixed in $2 \text{ mol}\cdot\text{L}^{-1}$ nitric acid by stirring for 1 h at pH~4-5, followed by addition of 30 mL of 1.5 mol L⁻¹ citric acid solution. The resulting solution turned to yellowish sol then the sol was stirred continuously using a magnetic stirrer at 60°C until it became a transparent sticky gel. At that point, the gel was dried in an air oven at 200°C for 1 h which leads the formation of light weight porous materials due to the enormous gas evolution, and it was sintered at 850°C for 4 h to get the fine homogeneous dense powder. A pictorial representation of the synthesis method is given in Figure 1.

2.2. Characterization. The room temperature powder Xray diffraction patterns were collected using a PANalytical X'Pert Pro diffractometer in Bragg-Brentano configuration. Monochromatic Cu K α 1, 2 (λ ~1.54060, 1.54443 Å) radiations were used as a source of 40 kV/30 mA power. Diffractograms were collected over 2θ -range 5–80 with 0.02 steps. Unit cell dimensions and initial structural refinements were carried out by Rietveld package FullProf 2003 [24]. SEM images were recorded using Hitchi-S3400 instrument operated at 20 kV, and EDX was done by a SUPER DRYER II instrument. The X-band EPR spectral measurement at 300, 77, and 13 K were done with a JEOL JES-TE 100 ESR spectrometer system with 100 kHz field modulation and a phase sensitive detector to obtain the first derivative signal. Quartz sample tubes were used for recording the EPR spectra of powder specimens. Samples were run at low temperatures using Air Products Helitran cryostat. The magnetic field was calibrated using a Varian NMR Gauss meter, and the microwave frequency was determined using an EIP frequency meter. Since a Gauss meter and frequency meter were used to record the lineshapes in these cases, a g-standard is not necessary. A Varian spectrophotometer (Model Carry 5000) at the range 200-800 nm recorded the Optical Absorption spectra in the solid phase.

3. Results and Discussion

The X-ray diffraction patterns of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ ($0.0 \le x \le 1.0$; B1–B5) nanoparticles are shown in Figure 2. In the XRD patterns, peaks were indexed using least square method to avoid the difference between experimental and theoretical patterns. The results of the indexed patterns are reported in Table 1. In the XRD patterns, a new weak peak is observed at 25.7° in B2–B5, and a peak at 55° is being reduced with increasing order of x value. The indexed patterns were undergone for Rietveld refinement analysis to estimate the lattice parameters. The profile was fitted with a pseudo-Voigt function, and a polynomial background subtraction was chosen for the refinement. The Rietveld refinement fit is shown in Figure 3. The lattice parameters of the samples are given in Table 2. The refinement results indicate the



FIGURE 1: Flow chart of the sample preparation of $Ag_{3(2+x)}$ $Al_x Ti_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5) nanoparticles.



FIGURE 2: The room temperature XRD pattern of $Ag_{3(2+x)}$ $Al_x Ti_{4-x} O_{11+\delta}$ (0.0 $\leq x \leq 1.0$; B1–B5) nanoparticles.

formation of nanocrystalline materials in tetragonal lattice with $P4_2/nnm$ space group, and *z* is 1 (*z*-number of atoms in the unit cell) for all the samples. The Lattice type, space group, and *z* values are found to be constant in the samples, possibly due to very low concentration of Al^{3+} ion present in the host lattice.

The average crystallite sizes of synthesized and sintered samples were calculated from the full width at half maximum (FWHM) of the peaks (112), (211), (213) (222), (223), (413), (512), and (514) using Debye-Scherrer formula [25]:

$$D = \frac{0.89\lambda}{\beta_{1/2}\cos\theta_{\rm hkl}}.$$
 (1)

Williamson-Hall equation is also used to calculate average crystallite size of the sample. The Williamson-Hall equation is given bellow:

$$B\cos\theta = \frac{K\lambda}{D} + 2\varepsilon\sin\theta,$$
 (2)

Journal of Nanoscience

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Sample code	Н	k	l	2TH-Obs	2TH-Calc	D-Obs
	2	1	1	27.477	27.480	3.2435
	2	0	2	30.186	30.170	2.9583
B1	3	0	1	36.135	36.078	2.4837
	2	2	2	38.169	38.226	2.3559
	2	2	2	44.376	44.360	2.0398
	5	1	2	64.511	64.516	1.4433
	1	1	2	25.301	25.262	3.5173
	2	1	1	27.428	27.445	3.2491
B2	3	0	1	36.070	36.035	2.4880
D2	2	2	2	38.112	38.170	2.3593
	2	2	3	44.293	44.284	2.0434
	5	1	2	64.432	64.430	1.4449
	0	0	3	25.270	25.269	3.5215
	1	0	3	28.269	28.259	3.1544
P 3	2	1	3	38.077	38.110	2.3614
D5	2	2	3	44.258	44.247	2.0449
	1	1	7	64.403	64.402	1.4455
	5	1	4	77.353	77.352	1.2326
	1	1	2	25.288	25.252	3.5191
	2	1	1	27.417	27.440	3.2505
P.4	3	0	1	36.063	36.029	2.4885
D4	2	2	2	38.096	38.160	2.3603
	2	2	3	44.281	44.269	2.0439
	5	1	2	64.421	64.417	1.4451
	2	0	2	25.341	25.355	3.5119
B5	3	0	0	27.474	27.530	3.2438
	4	0	0	36.993	36.994	2.4281
	4	1	0	38.144	38.176	2.3574
	2	2	4	44.331	44.329	2.0417
	6	1	3	64.471	64.470	1.4441
	2	0	2	25.341	25.355	3.5119

TABLE 1: Index lattice parameters of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5) nanoparticles.

TABLE 2: Lattice parameters of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5) nanoparticles.

Sample code	B1	B2	B3	B4	B5
Lattice type	Tetragonal	Tetragonal	Tetragonal	Tetragonal	Tetragonal
a (Å	7.7535	7.7618	7.1067	7.7629	9.7120
b (Å)	7.7535	7.7618	7.1067	7.7629	9.7120
c (Å)	9.1661	9.1875	10.5651	9.1925	10.1582
Unitcell volume	551.04	553.508	533.588	553.959	958.153
Space group	P42/nnm	P42/nnm	P42/nnm	P42/nnm	P42/nnm

TABLE 3: Density, crystallite sizes, and porosity of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5).

Sample coo	le	B1	B2	B3	B4	B5
X-value		0	0.25	0.50	0.75	1.0
Density (a/cm^3)	Experimental	2.74	4.87	5.15	2.39	4.45
Density (g/em)	Calculated	3.06	3.30	3.63	4.06	4.57
Crystallita sizas (nm)	Scherrer	44.0	18.8	18.8	18.1	18.0
	W-H plot	48.3 ± 2.6	19.9 ± 2.5	18.3 ± 2.6	18.2 ± 4.52	17.9 ± 4.4



FIGURE 3: Rietveld profile fit of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5) nanoparticles.



FIGURE 4: (a) The SEM morphology, (b) EDX- profile, and (c) X-ray mapping of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (x = 1.0) nanoparticles.

where *D*-crystallite size, *K*-Scherrer constant (K = 0.9), λ wavelength of Cu K α radiation, β -corrected full width half maximum (FWHM) of the diffraction peak, θ -Bragg angle of the X-ray diffraction peak and ε -lattice strain [26]. The average crystallite sizes of the samples in the range from 48 nm to 17 nm (B1–B5), and the values are reported in Table 3. The decreasing trend was observed in the crystallite size with increasing Al^{3+} ionic concentration; it may be

FIGURE 5: The room temperature optical absorption spectra of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5) nanoparticles.

FIGURE 6: (a) The EPR lineshapes of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq 1.0$; B1–B5) nanoparticles at 300 K. (b) The EPR lineshapes of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq 1.0$; B1–B5) nanoparticles at 77 K. (c) The EPR lineshapes of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq 1.0$; B1–B5) nanoparticles at 13 K.

TABLE 4: The spin Hamiltonian	a parameters of Ag _{3(2+x}	$Al_x Ti_{4-x} O_{11+\delta}$	$(0.0 \le x \le 1.0; B1-B5)$	i) nanoparticles
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	g-matrixes and A-matrixes									
Sample code	300 K		77 K				13 K			
	$\mathcal{G}_{\mathbb{I}}$	\mathcal{G}_{\perp}	$\mathcal{G}_{\mathbb{H}}$	g_{\perp}	A_{\parallel} (G)	A_{\perp} (G)	$\mathcal{G}_{\mathbb{I}}$	${\cal G}_{\perp}$	A_{\parallel} (G)	A_{\perp} (G)
B1	$g_{\rm iso} - 2.002$		$g_{\rm iso} - 1.992$		—	—	$g_{\rm iso} - 1.994$		—	_
B2	2.046	2.022	_	_	_	_	_	_	_	_
B3	2.277		2.087	2.030	168	20	2.075	2.019	169	21
B4	2.048	2.024	_	_		_	_		_	_
B5	2.047	2.018	2.077	2.016	167	20	2.086	2.019	162	21

attributed to dominating behavior of lower ionic radius of Al^{3+} (1.82 × 10⁻¹⁰ Å) ions that were replaced with higher Ti³⁺ (2.0 × 10⁻¹⁰ Å) ions.

Density of the samples were determined using liquid displacement method (Experimental) and lattice parameters (Calculated). The experimental density values are closer to the theoretical density. However, small differences in the densities are due to porous nature of the sample. The pictorial representation of the porous nature was identified by surface morphology. The crystallite sizes and density of the samples are reported in Table 3.

Surface morphology of the samples B1, B3, and B5 along with EDX profile and X-ray mappings of randomly selected particle in B5 is shown in Figures 4(a), 4(b), and 4(c), respectively. The morphology of the samples shows globular in shape. The purity was confirmed by EDX profile and quantitative results. The EDX results show the presence of Ag, Al, Ti, and O elements in the samples with expected ratio, and no other extra elements were observed; this conforms the formations of pure $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5) nanoparticles. The X-ray mapping has become an extremely powerful technique to understand the distribution of elements in the sample. The X-ray mapping (Figure 4(c)) of the sample B5 conforms the presence of all the constituent elements distributed homogenously with resemble of their relative quantity. The homogenous distribution of the elements is due to the existence of uniform particle size.

Room temperature optical absorbance spectra of the samples are shown in Figure 5. The absorption bands appeared at 320, 424, and 583 nm are assigned with the reported data. It reveals that the band at 320 nm is attributed to the Ti³⁺ ions, and 424 nm corresponds to the presence of Ag-TiO₂ nanoparticles. The same results lead to the samples which may be used for photocatalysis [27]. The band at 424 and 583 nm is also corresponding to Ti³⁺ ions. The 3d¹ electronic configuration of Ti³⁺ ionic ground state term is ²D. In the octahedral crystal field, the degeneracy is lifted; hence, the samples show broad absorption band at about 424 and 586 nm. The corresponding bands are attributed to ²B_{2g}-²B_{1g} (424 nm) and ²B_{2g}-²A_{1g} (583 nm) transitions of Ti³⁺ ions in distracted octahedral sites [28, 29].

Due to the paramagnetic nature of Ti³⁺ ions in the samples, the EPR technique has been widely employed in characterization of the samples. It is a single progress to understand the electronic and geometrical features of elements in different chemical environments. The EPR line-shapes of the $Ag_{3(2+x)}AI_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0; B1–B5) nanoparticles are shown in Figures 6(a)–6(c).

The spin Hamiltonian parameters of the samples were calculated using the following relation [30]:

$$h\nu = q\beta H,$$
 (3)

where *h*-Planck's constant, ν -resonance frequency, β -Bohr magnetron and *H*-field strength at which the resonance occurs. Ti³⁺ ion has five isotopes: ⁴⁶Ti, ⁴⁷Ti, ⁴⁸Ti, ⁴⁹Ti, and ⁵⁰Ti within this three isotopes, three are EPR inactive (⁴⁶Ti, ⁴⁸Ti, and ⁵⁰Ti) to zero nuclear spin (I = 0). The isotopes ⁴⁷Ti (I = 5/2) and ⁴⁹Ti (I = 5/2) are EPR active due to

nonzero nuclear spin. The EPR lineshapes of the sample B1 shows an isotropic nature with $g_{iso} \sim 1.991$ is due to dipoledipole interaction of Ti ions [31]. However, the isotropic exchange interaction usually dominates the effects of spinorbit coupling, and magnetic dipole interaction. The electron spin resonance of Ti³⁺ (d¹) ions has been detected in only a few cases.

Generally, Ti³⁺ (d¹) ions excited states are closer to the ground state in octahedral and tetrahedral symmetry in this case EPR lineshapes can only be detected at liquid-helium temperatures due to a very short spin-lattice relaxation time [24, 32]. Consequently, the sample B1 shows broad unresolved hyperfine line at most temperatures. Once Al³⁺ ions are incorporated into the sample B2-B5, the crystal field is distracted, as the effect or the spin-lattice relaxation time becomes longer. It results the possibility of hyperfine splitting at higher temperature [33, 34] as in the case of B2-B3. The hyperfine splitting of the sample B2-B5 shows six parallel and perpendicular lines with $g_{\parallel} \sim 2.0233$ and $g_{\perp} \sim 2.0233$, which are not well-resolved lineshapes due to the small distraction. The obtained spin Hamiltonian parameters of the samples at 13 K, 77 K, and 300 K were reported in Table 4. The EPR spectra of the samples are reasonable for 47 Ti (I = 5/2) isotopes. A single-broad line centered at q = 2.05 is observed, and the additional lines were observed on both sides of the central line, which are not six as expected. The collapse of the extra lines may be caused by dipolar interaction between Ti^{3+} (d¹) ions.

4. Conclusion

The nanoparticles of $Ag_{3(2+x)}Al_xTi_{4-x}O_{11+\delta}$ (0.0 $\leq x \leq$ 1.0) were successfully prepared by sol-gel method. The XRD results of the samples were conformed the formation of the tetragonal lattice with P4₂/nnm space group. The morphology of the samples shows globular in shape, and the constituent elements were distributed homogenously. The optical absorbance spectra conforms the formation of Ag-TiO₂ nanoparticles and the samples. The Ag-TiO₂ nanoparticles can be used for photocatalytic activity. The EPR result shows that the hyperfine splitting is due to the ⁴⁷Ti isotopes of Ti³⁺ ions. The samples show paramagnetic behavior in room temperature.

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