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Facile and Eco-friendly One Step Low Temperature Synthesis of Very Large Scale $ATiO_3$ (A = Ca, Sr, Ba and Cd)

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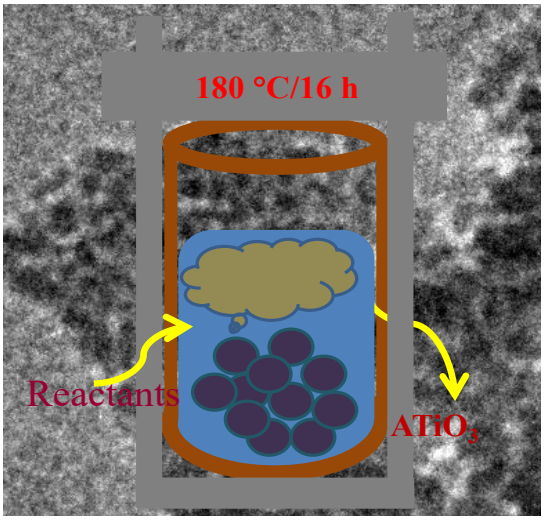
Abstract

The primary motivation of this work is to develop a low-cost and scalable methodology for the synthesis of industrially significant metal titanates. In this study, $ATiO_3$ (A = Ca, Sr, Ba, and Cd) perovskite nanoparticles were successfully synthesized via a simple, one-step hydrothermal process carried out at 180 °C for 16 hours. By varying the titanium precursor from TiO_2 to titanium isopropoxide, nanoparticles with different sizes and morphologies were obtained. This approach offers a general and practical route for the large-scale synthesis of $ATiO_3$ perovskite materials.



Development of a low-cost methodology for the hydrothermal synthesis of titanates with varying size and morphology

Highlights

- Low temperature general synthesis protocol for 4 titanates
- Variation of reactant phase from solid to liquid and their effect of morphology
- Hollow rectangle shape morphology of $CaTiO_3$
- Enhancement in the surface area of $BaTiO_3$
- Scope of applications in various industry

Keywords: Hydrothermal, Titanium Isopropoxide, Titanates, Hollow

1. Introduction

Nanostructured materials such as nanoparticles, nano cubes, nanorods, nanotubes and nanowires are under intensive investigation due to their size and shape dependent properties and potential application in the all field of technology due to their thermal, electrical, mechanical and optical properties [1-5]. Among the nanostructured materials, perovskite with general formulae ABO_3 constitute important classes of materials in solid state chemistry due their wide applicability in ferroelectrics, dielectric, piezoelectric nonlinear optics properties with their technology importance for fabricating the different class of devices [6-10]. On the commercial scale, these oxide powders are produced at high-temperature which's chemical in homogeneity, reactivity problems and very little control on the size, shape and agglomeration. Due to these above problems, there is demand for the low temperature synthesis route to yield high-purity fine powders with controlled size and morphology for miniaturization of these materials to the nanometer scale for the next generation of electronics.

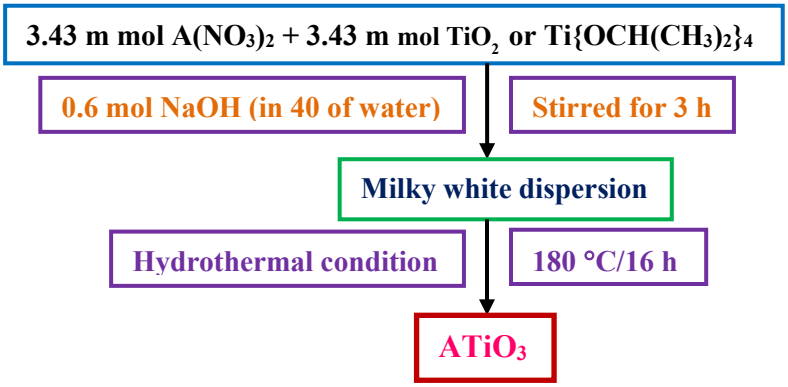
All the titanates are useful class of materials due to their industrial application in various technologies. Among them $BaTiO_3$, $SrTiO_3$, and $CaTiO_3$ has more importance. $BaTiO_3$ has ferroelectric property, high dielectric constant and positive temperature coefficient of resistivity (PTCR) used as multilayered ceramic capacitors (MLCCs), embedded decoupling capacitors (EDC), thermistor, waveguide modulators, IR detectors, microwave absorbers, dynamic random access memory and field - effect transistors [11-15]. $SrTiO_3$ is an important n-type semiconductor with band gap between 3.2 – 3.4 eV have high dielectric constant, thermal and chemical stability. It has a wide application in field of thin-film capacitors, nonlinear optics, optical memories, oxygen gas sensors, photocatalyst and photoelectrode for splitting of water, solar cell and electrooptic modulators [16-19]. $CaTiO_3$ crystallizes in orthorhombic crystal system with space group (Pbnm). It has various applications such as component of capacitors, immobilization of nuclear wastes, mineralogy, microwave dielectric applications (as resonators and filters), luminescent material and biomedical area [20-23]. $CdTiO_3$ crystallizes in non-ferroelectric ilmenite rhombohedral crystal system. Below the 1000 ^{0}C annealing temperature is an intelligent material due to their application in optical fiber and sensor [24, 25]. It has also piezoelectric, pyroelectric, magneto restrictive and photostrictive properties [26-28].

There are various reports for synthesizing such titanates by solid state, sol – gel, hydrothermal, microwave hydrothermal method, spray pyrolysis, microemulsion, polymeric precursor and coprecipitation [29-37]. There are few reports for general methodology of the $MTiO_3$ nanoparticles [38-41]. There are reports of the synthesis of $SrTiO_3$ and $BaTiO_3$ at low temperature however there is no any such report on $CaTiO_3$ and $CdTiO_3$ at low temperature [42-46]. There is no general approach reported for synthesizing the above four compounds by a single step on large scale.

In this letter, we report the synthesis of $ATiO_3$ (A = Ca, Sr, Ba, and Cd) perovskite materials using two different titanium sources: titanium dioxide and titanium isopropoxide. The resulting compounds were thoroughly characterized using powder X-ray diffraction (PXRD), field emission scanning electron microscopy (FESEM), transmission electron microscopy (TEM), Brunauer–Emmett–Teller (BET) surface area analysis, and UV-Visible diffuse reflectance spectroscopy (DRS).

2. Synthesis and Characterization

$ATiO_3$ nanoparticles were synthesized by hydrothermal route. In typical synthesis process 3.43 m mol of $A(NO_3)_2$ was dissolve in 20 ml of double distilled water followed addition of 3.43 m mol of TiO_2 (Titanium isopropoxide also). The above solution was stirred for 30 min and then 0.6 mol NaOH dissolved in 40 ml of water were added and stirred for 3 h. The obtained milky white solution was transferred to teflon beaker and putted inside the stainless-steel hydrothermal bomb and placed in oven at 180°C for 16 h. The schematic diagram for the synthesis of $ATiO_3$ nanoparticles of different morphology is given in scheme 1.

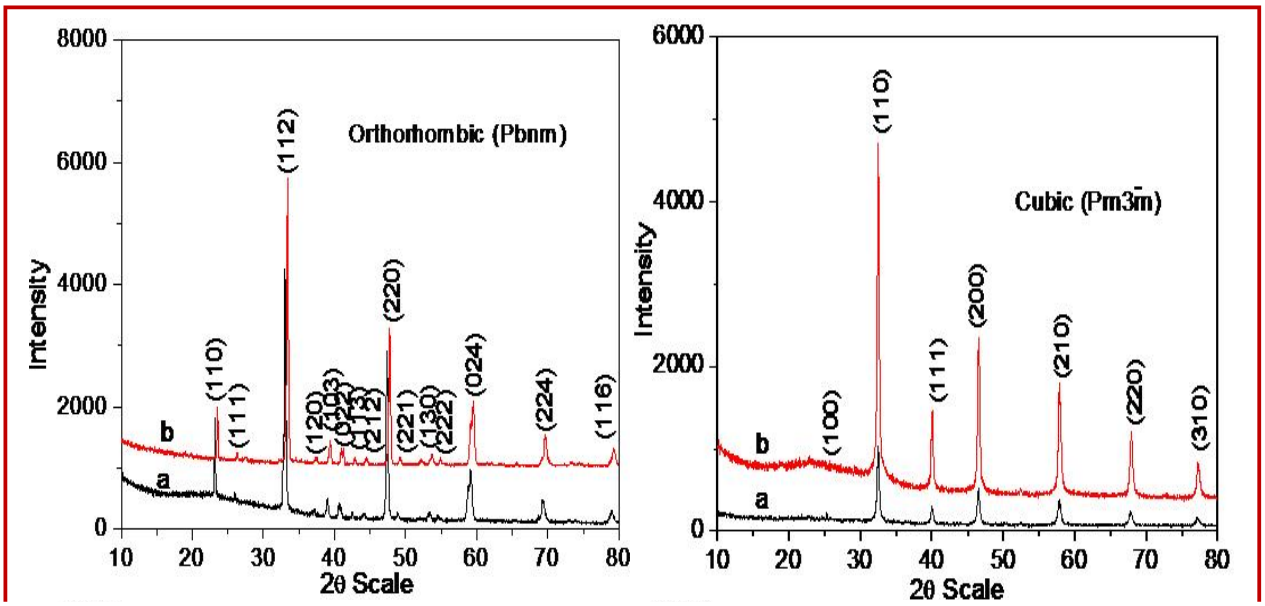


Scheme 1: General Procedure for the Synthesis of $ATiO_3$

Powder X-ray diffraction studies (PXRD) were carried out using Ni filtered Cu-Kα radiation. Normal scans were recorded with a step size of 0.02° and step time of 1 s. The $K_{\alpha 2}$ reflections were removed to obtain accurate lattice constants. Field emission scanning electron microscopy (FESEM) was carried out by FEI quanta 3D FEG – FESEM operated at 10 kV. The pellet had been coated with gold. Transmission Electron Microscopy (TEM) studies were carried out using a Tecnai G2 20 electron microscope operated at 200 KV. TEM specimens were prepared by dispersing the sample in ethanol by ultrasonic treatment, dropping onto a porous carbon film supported on a copper grid, and then drying in air. Nitrogen adsorption–desorption isotherms were recorded at liquid nitrogen temperature (77 K) using a Nova 2000e (Quantachrome Corp.) equipment and the specific area was determined by the Brunauer–Emmett–Teller (BET) method. The powder sample was degassed at 150 ºC for 6 h prior to the surface area measurements. Diffuse-reflectance spectra (DR) spectra were recorded on Shimadzu UV-2450 spectrophotometer where the baseline was fixed using a barium sulfate reference.

3. Result and Discussion

The aim behind this study is to develop a low-cost methodology for the synthesis of important titanates with different morphology at very low temperature. Here we have developed a common one step synthesis protocol at very low temperature (180 ºC) to synthesize titanates at gram scale (~ 1g) level by hydrothermal route. Here we fixed the hydrothermal reaction time, temperatures, reactant and base concentration. We varied the one of the reactant (Ti source) phase from solid to liquid to see change in the morphology. Using a NaOH base, we obtained pure phase of the nanocrystalline titanates $ATiO_3$ (Ca, Sr, Ba, Cd). Figures 1 (A, B, C, D) show the powder X-ray diffraction patterns of $CaTiO_3$, $SrTiO_3$, $BaTiO_3$ and $CdTiO_3$ respectively using both TiO_2 and titanium isopropoxide as Ti source. The PXRD patterns confirm the formation of monophasic nature for all the eight titanates. All the observed diffraction patterns were index on the basis of their crystal structures. The lattice parameters were refined and it observed that in case of titanium isopropoxide as Ti source the values was lower as compared to TiO_2 as Ti source due to decrease in the particles size of corresponding titanates.



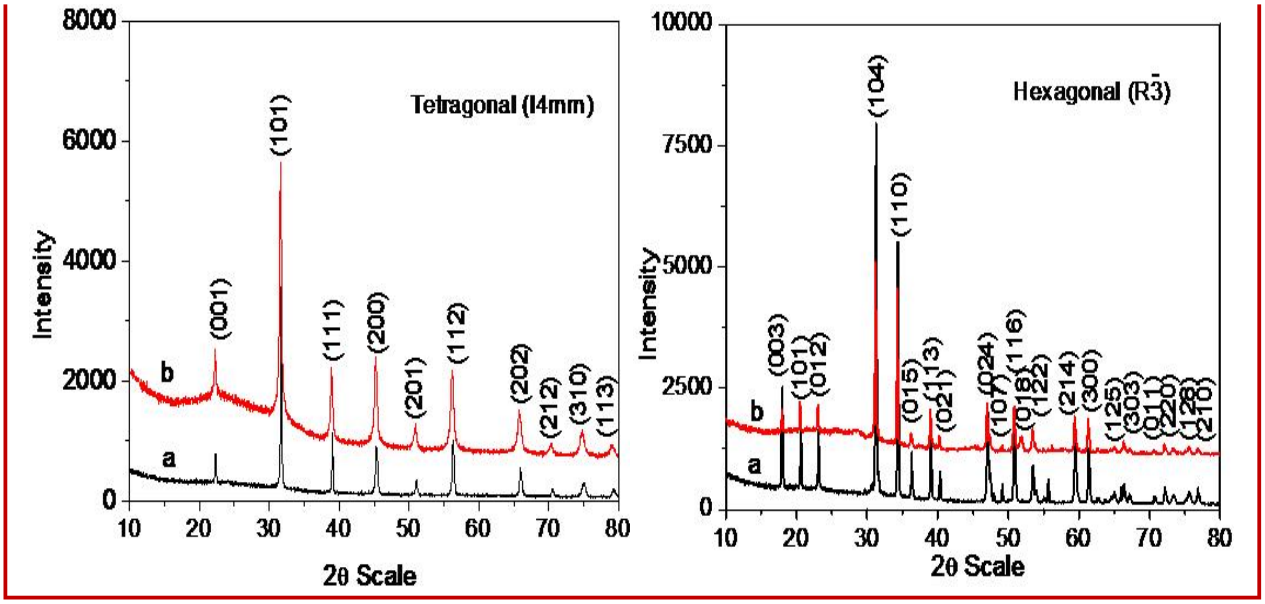
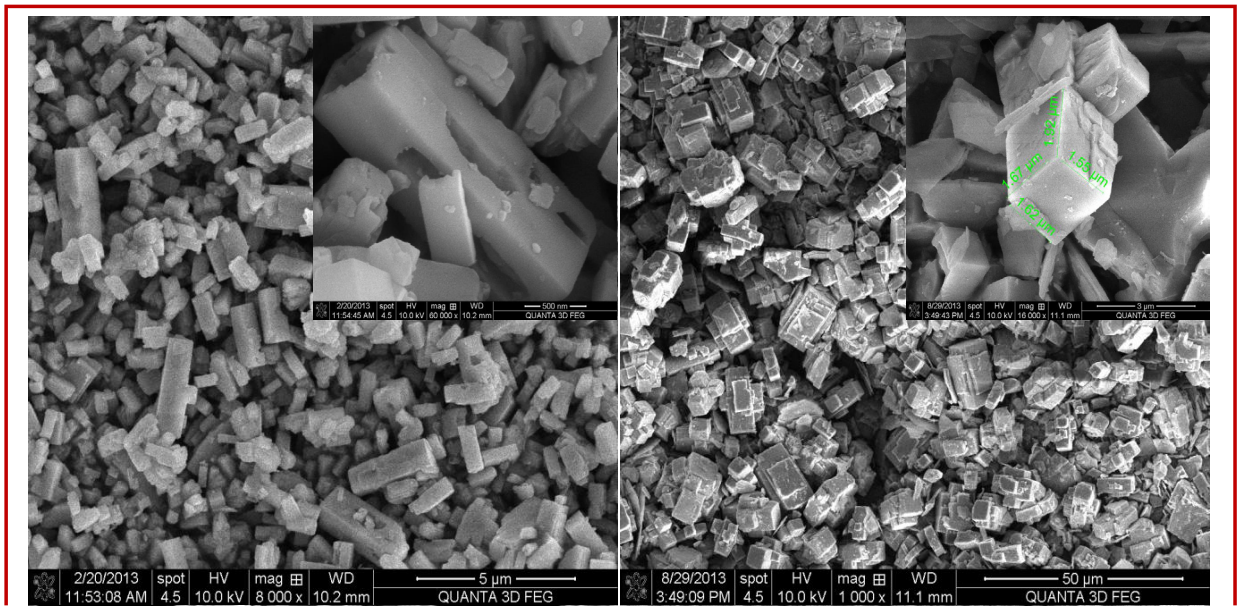


Figure 1: Powder x-ray Diffraction Pattern of (A) $CaTiO_3$(B) $SrTiO_3$(C) $BaTiO_3$(D) $CdTiO_3$

Field emission scanning electron microscopy was carried out to check the morphology of these synthesized titanates. Figure 2 and 3 show the field emission scanning electron micrograph of titanates. Figure 2a shows cube shape morphology of $CaTiO_3$ obtained using TiO_2 whereas in case of titanium isopropoxide (Figure 2b), rectangle hollow shape morphology was obtained. This result clearly indicates that with change in source phase from solid (TiO_2) to liquid (titanium isopropoxide) the morphology was changed. This hollow morphology type's materials are very interesting for various applications such as drug delivery and catalysis [47-49]. The $SrTiO_3$ (Figure 2c and 2d) the particles are spherical in nature having two different sizes for the two different titanium sources. For the titanium isopropoxide source, the particles are smaller as compared to titanium dioxide. In the case of TiO_2 source some of these nanoparticles (30 nm size) are aggregated and form cube shaped structures. The $BaTiO_3$ nanoparticles size distribution (Figure 3b) is very uniform in case of titanium isopropoxideas Ti source and it is found to be very small in size (further confirm by TEM) as compared to TiO_2 sources. The $BaTiO_3$ obtained from TiO_2 (Figure 3a) shows the formation of mixed morphology i.e. spherical and cube. For $CdTiO_3$, sheet like morphology was obtained in both the cases. The size of sheet is smaller and broken in case of liquid phase sources. These sheets like structures are very useful for the formation of 2-D materials for practical applications.



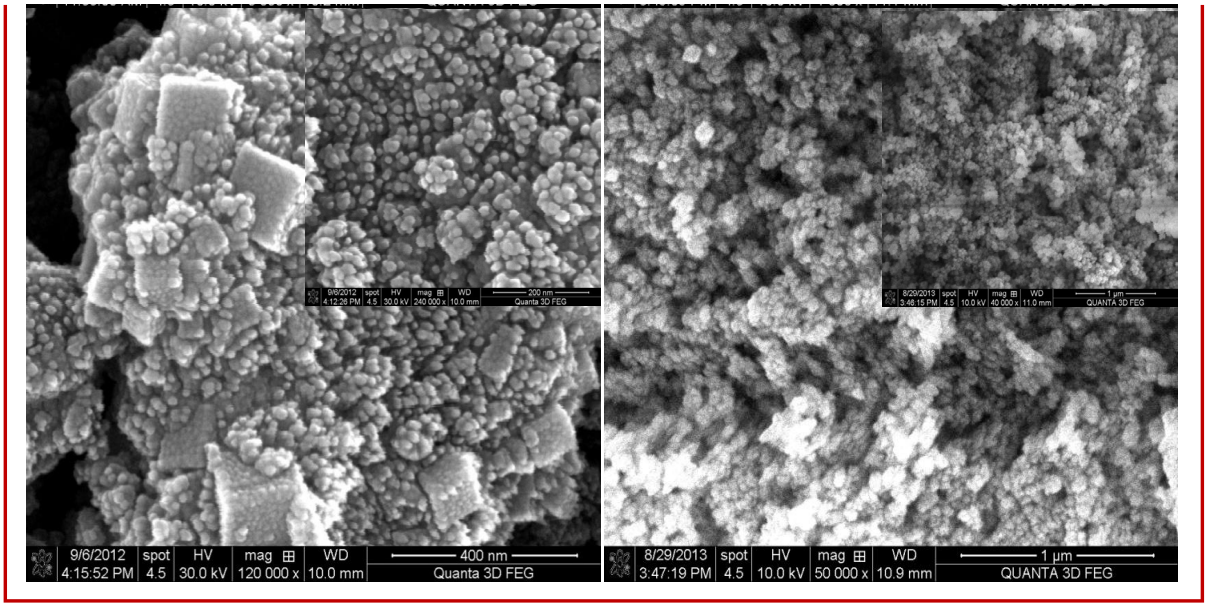


Figure 2: FESEM Micrograph of (a, b) $CaTiO_3$ (c, d) $SrTiO_3$ using TiO_2 and Ttitanium Isopropoxide as Ti Source Respectively

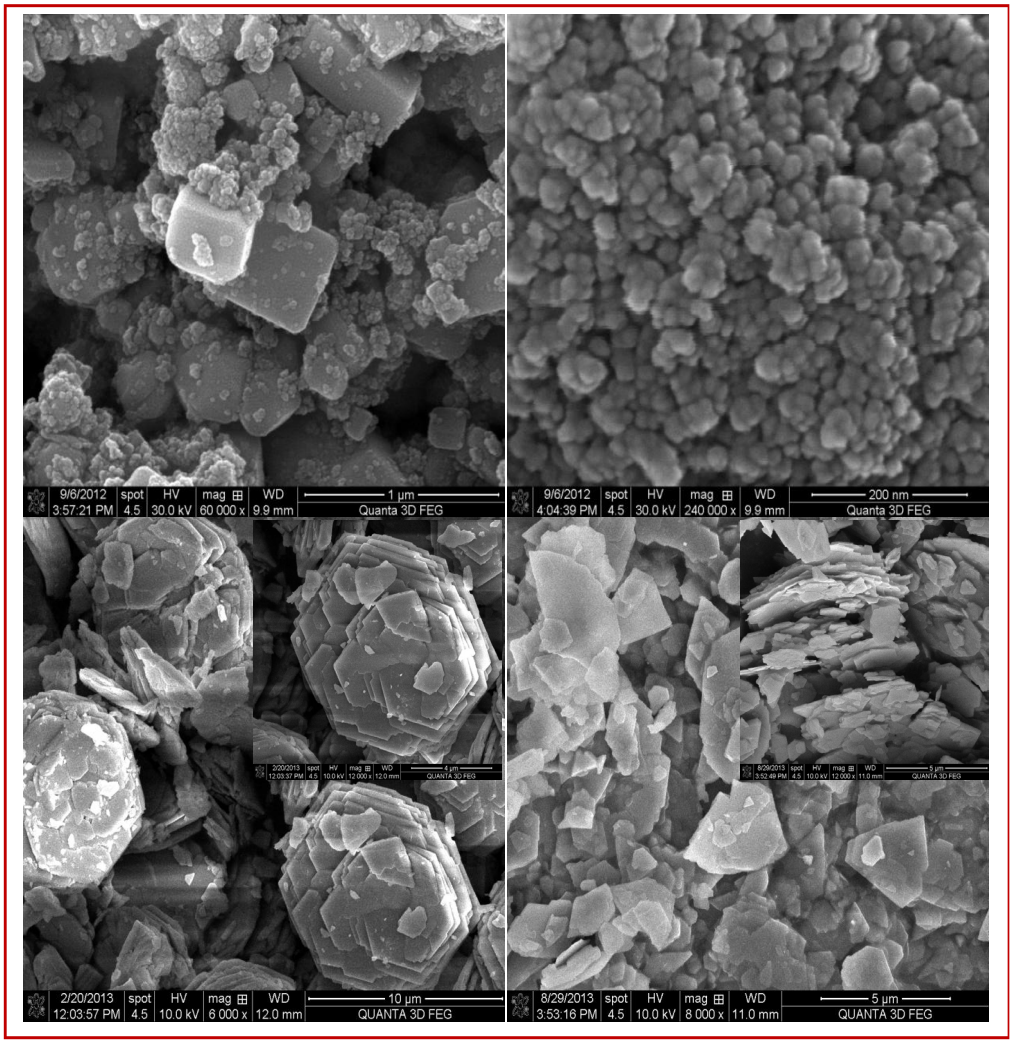


Figure 3: FESEM Micrograph of (a, b) $BaTiO_3$ (c, d) $CdTiO_3$ using TiO_2 and Titanium Isopropoxide as Ti Source Respectively

Transmission electron microscopy (Figure 4) was carried out to confirm the particle size of $SrTiO_3$ and $BaTiO_3$ nanoparticles. The $SrTiO_3$ nanoparticles (Figure 4a and 4b) are spherical in nature having average particles size of 30 nm and 12 nm for titanium dioxide and titanium isopropoxide respectively. There are very few reports of $SrTiO_3$ having particles of size less than 15 nm but none of the reports claim synthesis at gram scale level [50, 51]. The $BaTiO_3$ nanoparticles are uniformly distributed and spherical in nature in case of titanium isopropoxide as titanium sources (Figure 4d). The average diameter of the particles was found to be 5 nm. In case of metal titanates obtained from TiO_2 source showed mixed distribution of particle size (Figure 4c). There are only some reports in literature of $BaTiO_3$ of such small size nanoparticles however the synthesis method not showed a general protocol [42-46, 51].

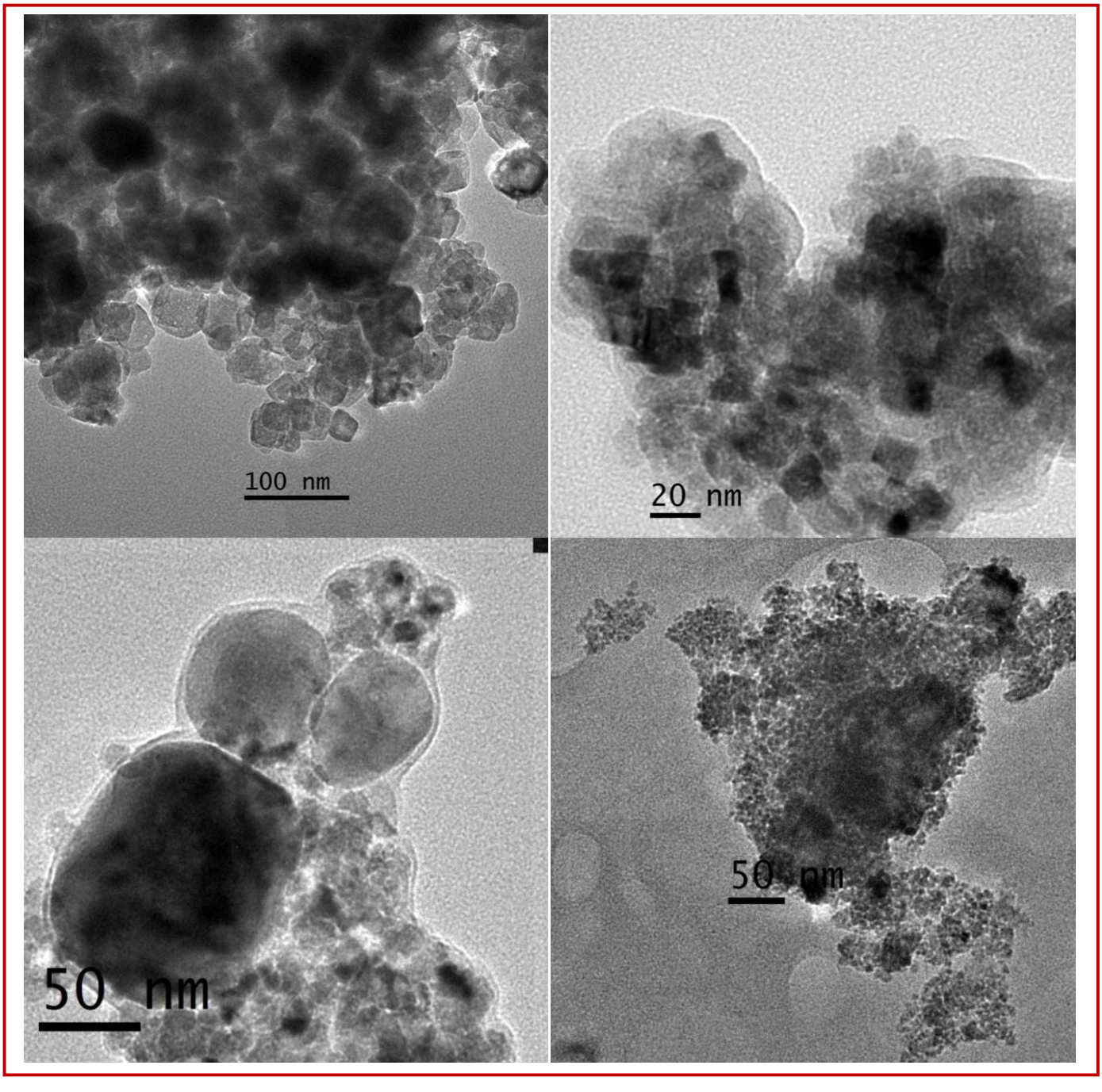


Figure 4: TEM Micrograph of (a, b) $SrTiO_3$ (c, d) $BaTiO_3$ using TiO_2 and Titanium Isopropoxide as Ti Source Respectively

N_2 adsorption-desorption measurement was carried out for surface area measurements of the nanoparticles (Table 1). It was observed that as titanium source changed from solid phase (TiO_2) to liquid phase (titanium isopropoxide) the surface area of the titanates enhanced along with decrease in particle size. In case of $CaTiO_3$ and $BaTiO_3$, the surface area is more enhanced due to the hollow shape particles ($CaTiO_3$) and small in $BaTiO_3$ in case of titanium isopropoxide as Ti sources.

Figure 5 shows the diffuse reflectance spectra of the all the titanates. It was observed that as titanium source changed from TiO_2 to titanium isopropoxide the band gap of the titanates increased due to decrease in particle size. All the titanates have band gap in the UV and close to the reported value. The optical properties (band gap) of nanomaterials are size and morphology dependent [52, 53]. For several cases, it is observed that a change in size and morphology can alter the band gap. The band gap of all the titanates is given in Table 1.

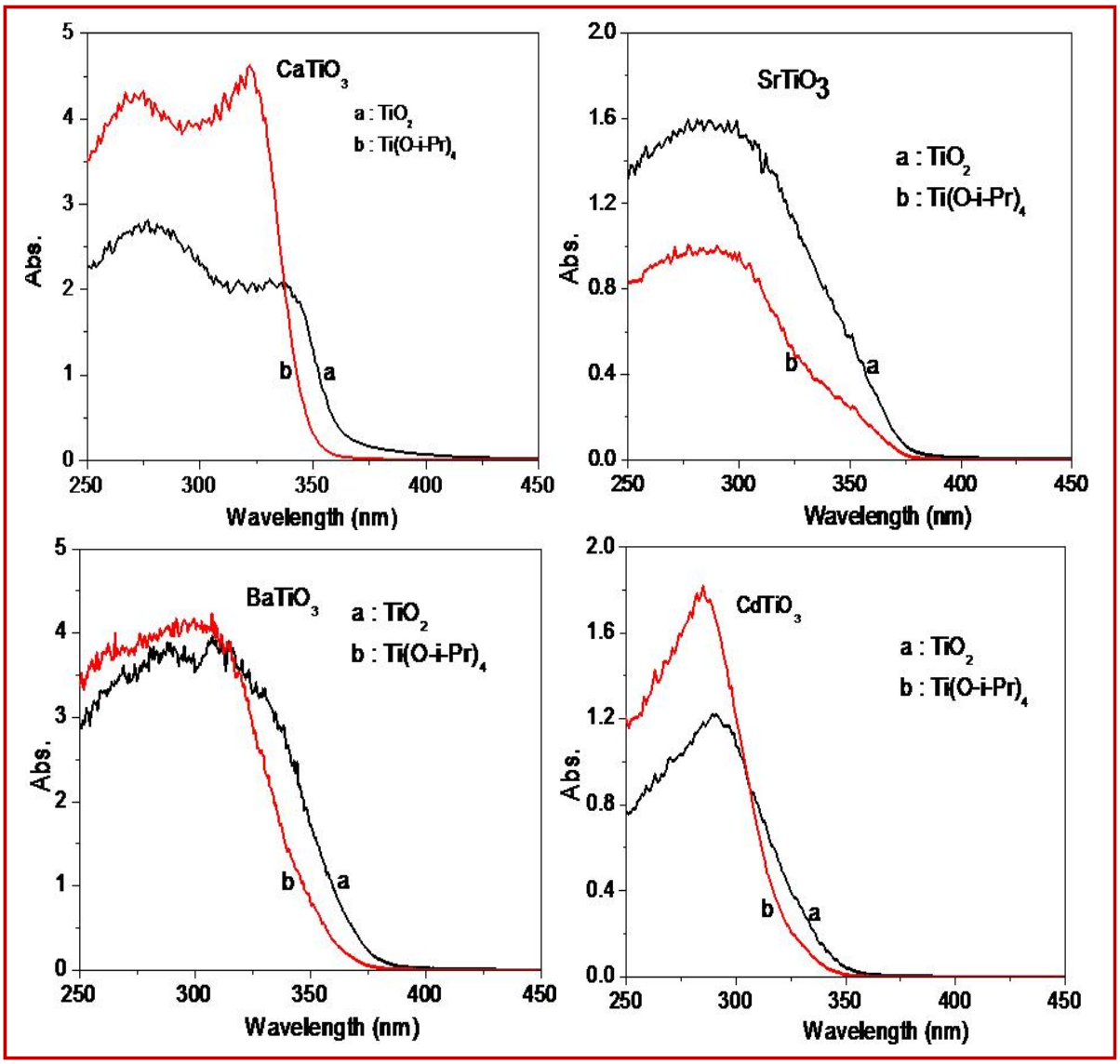


Figure 5: Diffuse Reflectance Spectra of(A) $CaTiO_3$(B) $SrTiO_3$(C) $BaTiO_3$(D) $CdTiO_3$

S. No.	Compound	Ti Source	Surface area (g/m^2)	Band Gap (eV)
1	$CaTiO_3$	TiO_2	49	3.4
		$Ti(O\text{-}i\text{-}Pr)_4$	152	3.5
2	$SrTiO_3$	TiO_2	55	3.3
		$Ti(O\text{-}i\text{-}Pr)_4$	64	3.35

3	$BaTiO_3$	TiO_2	58	3.3
		$Ti(O\text{-}i\text{-}Pr)_4$	184	3.4
4	$CdTiO_3$	TiO_2	67	3.6
		$Ti(O\text{-}i\text{-}Pr)_4$	95	3.7

Table 1: Surface Area and Band Gap of Synthesized Titanates

4. Conclusions

We report a general and cost-effective one-step hydrothermal protocol for the low-temperature synthesis of $CaTiO_3$, $SrTiO_3$, $BaTiO_3$, and $CdTiO_3$ nanoparticles. The morphology and particle size are strongly influenced by the titanium precursor, with liquid-phase titanium isopropoxide producing significantly smaller and more uniformly distributed nanoparticles than TiO_2; in particular, $BaTiO_3$ nanoparticles as small as ~5 nm were obtained. The size and morphology can be further tuned by varying hydrothermal parameters such as reaction time, temperature, base, and reactant concentration. The successful synthesis of ultra-small titanate nanoparticles at low temperatures and in large quantities demonstrates the scalability of this approach and highlights its potential for commercial production of nanostructured metal titanates.

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Author Contributions

Pramendra Kumar Pandey and Revati Raman Chaubey: Synthesis and draft writing, Gyandeshwar Kumar Rao, Suman Srivastava and Arvind Kumar Singh: Characterization, Bharat Kumar: Concept, synthesis modification, final draft writing and final submission. All the authors reviewed the manuscript.

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Declarations

Clinical Trial

Clinical trial is not applicable in the manuscript.

Consent to Publish Declaration

Not applicable

Ethics and Consent to Participate Declarations:

Not applicable

Competing Interests:

The authors declare no competing interests.

Data Availability

The authors declare that the data supporting the findings of this study are available within the paper file. Should any raw data files be needed in another format they are available from the corresponding author upon reasonable request.

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