Comparative SEM Analysis Study of Titanium Dioxide (TiO_2) Nanoparticles Synthesized by Sol-Gel Method

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Abstract

This paper describes the comparative SEM Analyses study of various samples of nanoparticle Titanium Dioxide (TiO_2) synthesized by the Sol-Gel method with different changes in the parameters. Titanium dioxide is not very reactive with chemicals and doesn't harm the environment, so it is widely used as a colour in industry. It comes in three forms: anatase, rutile, and Brookite. Scanning electron microscopy was used to examine the morphological changes of the produced TiO_2 nanoparticles at various calcination temperatures as well as the precursor ratio. SEM analysis was carried out at 10 kV acceleration Voltage. According to the SEM results, the achievement of high temperatures, the alteration of the precursor ratio, and the presence of distinct contents of the two crystalline phases of titanium dioxide are the causes of the increase in particle size and the apparent aggregation of TiO_2 nanoparticles. These results are in agreement with XRD results that showed the particles size of the anatase phase smaller than particles grown at the higher temperature.

Keywords: Titanium Dioxide (TiO_2) nanoparticles, Sol-Gel Method, SEM Analysis, Anatase Phase, Particle size.
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Introduction

Titanium Dioxide (TiO_2) which is also defined as titania is one of the most versatile and widely studied materials in modern science and technology. Known for its exceptional chemical inertness, thermal stability, and environmental friendliness, one of titanium's natural oxides is TiO_2. There are three main crystalline forms of it: rutile, brookite, and anatase [1]. Each has distinct chemical and physical characteristics. Among these, the anatase and rutile phases are of particular interest due to their remarkable photocatalytic performance and optical characteristics. The anatase phase is known for its high reactivity and is often used in photocatalysis, while the rutile phase exhibits better thermal and structural stability. Due to its non-toxic nature and strong UV light absorption, TiO_2 has found widespread applications across various industries. In the pigment industry, it serves as an essential component for paints, coatings, and plastics, providing superior opacity and brightness. In the cosmetics sector, it is used in sunscreens and skincare products to protect against harmful UV radiation [2]. Titanium Dioxide used in the pigment industry can also be defined as titanium white or pigment white. Furthermore, TiO_2 has gained significant attention in environmental applications, such as air purification, water treatment, and self-cleaning surfaces, owing to its photocatalytic properties that enable the breakdown of organic pollutants [3,4].

In recent years, TiO_2 has emerged as a key material in renewable energy technologies. It is extensively employed as a photocatalyst for the water splitting process that produces hydrogen and in dye-sensitized solar cells (DSSCs). Its ability to function as a photocatalyst under UV light has also led to innovations in energy-efficient coatings and anti-bacterial surfaces. Moreover, its abundance in nature, low cost, and ease of synthesis have contributed to its growing importance in industrial and research domains [5,6].

Extensive studies are being conducted to further enhance the efficiency and functionality of TiO_2, particularly by tailoring its properties through methods such as doping, surface modification, and nano-structuring [7]. These advancements aim to optimize its performance for specific applications and explore its potential in emerging fields like biomedical devices, sensors, and advanced energy storage systems [8,9].

Overall, the versatility, affordability, and eco-friendly nature of Titanium Dioxide make it an indispensable material in both traditional industries and cutting-edge technological advancements [10].

The Sol-Gel method is a cost-effective and versatile chemical route for synthesizing metal oxide (MO) nanoparticles, offering precise control over particle size and morphology. X-ray diffraction (XRD) is a fundamental tool for phase identification, lattice parameter determination, and crystallite size estimation. This study aims to compare the structural properties of TiO_2 nanoparticles synthesized via the Sol-Gel method under various conditions using XRD analysis [11].

Experimental Methods

• Materials

Table 1: Materials used in the synthesis of TiO_2

Chemical Name	Chemical Structure
Titanium tetraisopropoxide (TTIP) [the titanium precursor]	$Ti(OCH(CH_3)_2)_4$
Acetic acid	CH_3COOH
Distilled water	H_2O

• Methodology

The Sol-Gel method is a versatile chemical process widely used for synthesizing advanced materials, such as nanoparticles, thin films, and ceramics. It involves a transformation from a liquid solution (sol) into a solid network (gel) through hydrolysis and condensation reactions of metal alkoxides or inorganic salts. This technique allows precise control over material properties, including particle size, morphology, and composition [12].

One of the main benefits of the Sol-Gel method is its ability to synthesize materials at relatively low temperatures, which is an advantage over traditional high-temperature techniques. The process generally starts with the preparation of a precursor solution, typically using metal alkoxides like titanium (IV) isopropoxide. This solution undergoes hydrolysis and condensation, leading to the formation of a gel. Subsequent drying and calcination convert the gel into the desired crystalline phase with specific structural characteristics [13,14].

The Sol-Gel method is highly adaptable and can produce materials in various forms, such as powders, thin films, fibers, or monolithic structures. It is widely employed in applications like optical coatings, catalysts, sensors, and bioactive materials. The technique is especially valued for producing highly pure and homogeneous materials, essential for many advanced technologies [15].

In recent developments, the Sol-Gel process has become increasingly popular for synthesizing metal oxide nanoparticles, including titanium dioxide (TiO_2). This method provides precise control over the structural and surface properties of nanoparticles, making it ideal for applications in photocatalysis, energy storage, and environmental cleanup [16]. Its simplicity, cost-effectiveness, and scalability have made the Sol-Gel method a favoured choice in both academic research and industrial manufacturing [17,18].

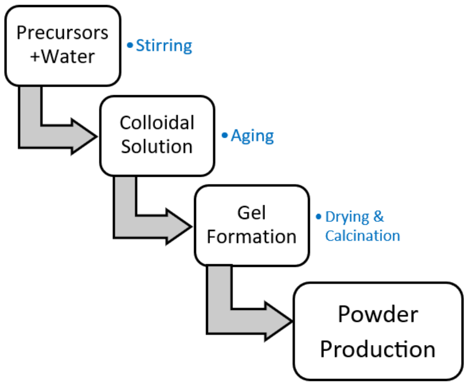


Figure 1: The flowchart of Sol-gel Synthesis.

The Sol-Gel process for Titanium Dioxide involved the hydrolysis and condensation of TTIP. The steps were as follows:

1. Mix 20 ml of titanium isopropoxide with 40 ml of glacial acetic acid.
2. Stir the mixture using a magnetic stirrer until a homogeneous solution forms.
3. Gradually add 120 ml of deionized water to the solution, drop by drop, while stirring continuously for 2 hours to form the sol.
4. Place the solution in an oven set at 90 °C and heat for about 12 hours to facilitate gel formation.
5. Pulverize and dry the gel at 200 °C for 2 hours, resulting in the formation of a white powder [19].

Figure 2: Transformation of Sol to Gel.

I have carried out the experiment by changing different parameters like the precursor ratio, water amount and the calcination temperature by 200 °C, 400 °C, 500 °C which is shown in a table form as follows [20,21]:

Table 2: The change in the different parameters with the different samplings.

Sample	TTIP (ml)	Acetic Acid (ml)	Dist. Water (ml)	Temperature ˚C
S1	20	40	120	200
S2	30	40	120	200
S3	20	40	200	200
S4	20	40	120	400
S5	25	45	125	500

Results and Discussions

• Characterisation

The synthesized TiO_2 nanoparticles samples with different parameters were analyzed using Scanning Electron Microscopy (SEM), revealing the following results [22,23]:

Sample 1

The particles were roughly spherical in shape with a spongy texture.

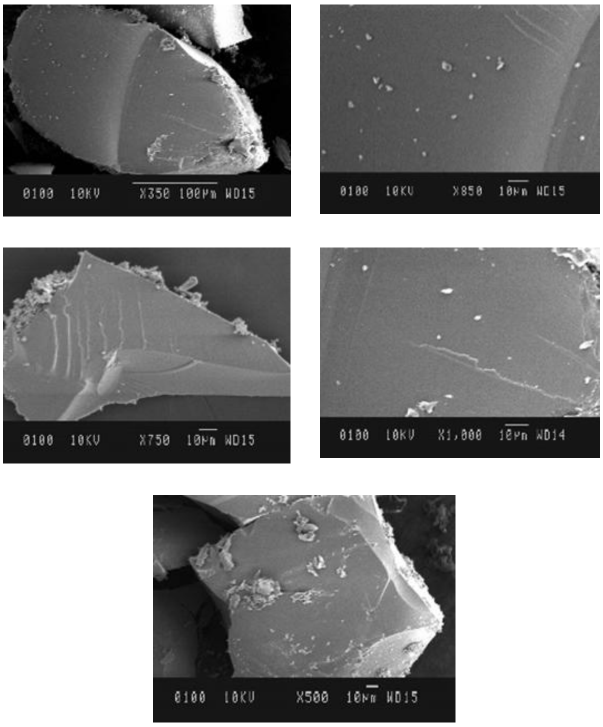


Figure 3: SEM images of the Sol-Gel synthesized TiO_2 nanoparticles (Sample1 as mentioned in Table 2)

Their sizes were in the range of less than 20 nanometres, and the particles were relatively well-distributed. This sample showed the best characteristics in terms of small particle size and distribution, making it highly effective for applications like photocatalysis [24].

Sample 2:

At a higher calcination temperature, the particles exhibited increased size and agglomeration.

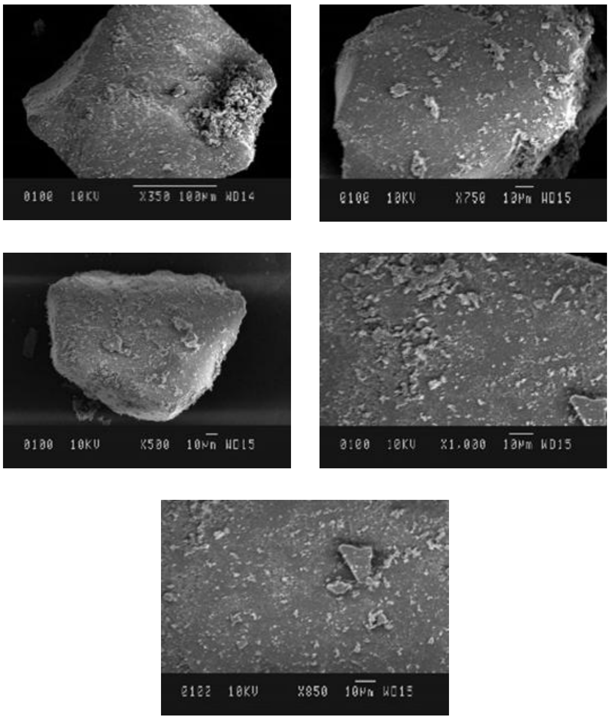
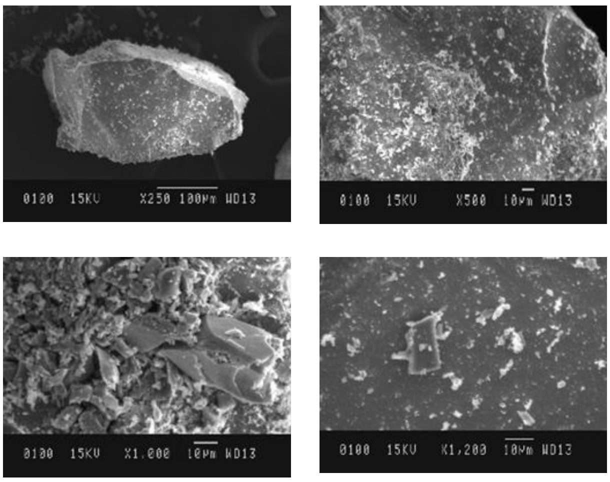


Figure 4: SEM images of the Sol-Gel synthesized TiO_2 nanoparticles (Sample2 as mentioned in Table 2)

The shapes became less uniform due to the initial particles clumping together as crystallinity increased.

Sample 3:

This result shows agglomerated particles with varying shapes and sizes.



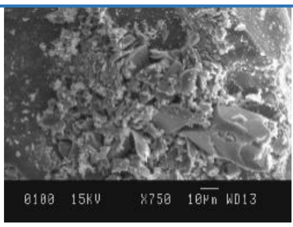


Figure 5: SEM images of the Sol-Gel synthesized TiO_2 nanoparticles (Sample3 as mentioned in Table 2)

Some regions appear porous and spongy, while others show larger, plate-like structures. There is noticeable particle clustering, suggesting possible agglomeration due to high calcination temperatures or synthesis conditions [24].

The presence of larger particles and irregular morphology could indicate increased crystallization and grain growth, leading to agglomeration.

Sample 4:

There appears to show a rough, uneven surface with noticeable cracks and interconnected structures.

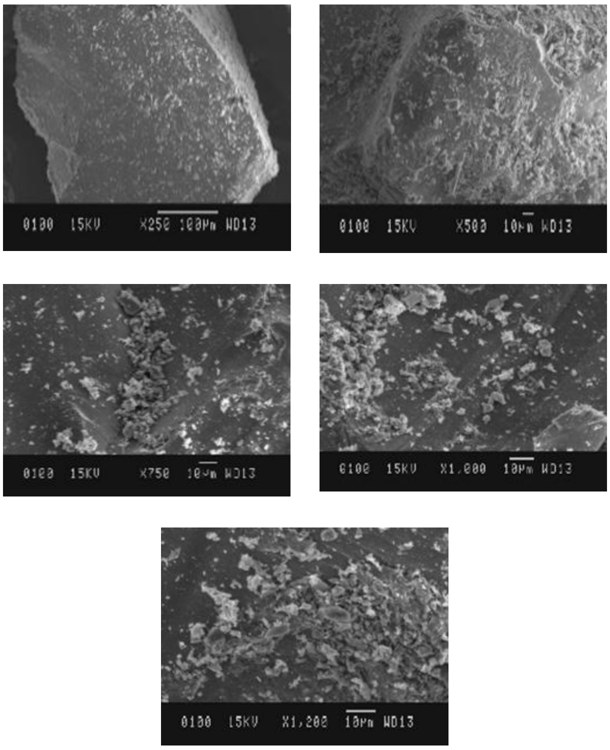


Figure 6: SEM images of the Sol-Gel synthesized TiO_2 nanoparticles (Sample4 as mentioned in Table 2)

The texture looks dense and compact, with some regions displaying porous structures. There are fine grain-like features compared to sample shown before.

Sample 5:

The trend of increased crystallinity continued, but there was a significant anatase-to-rutile phase transformation. This transformation impacts properties like photocatalytic activity and electron mobility [25].

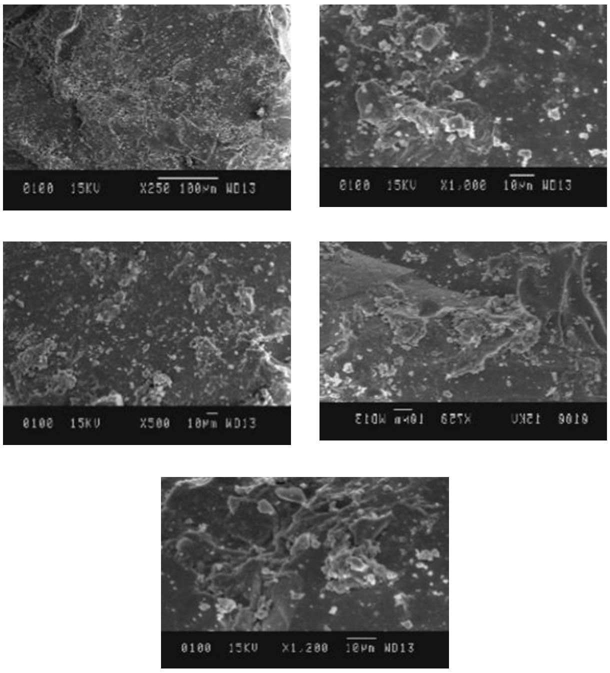


Figure 7: SEM images of the Sol-Gel synthesized TiO_2 nanoparticles (Sample5 as mentioned in Table 2).

Conclusion

1. Effect of Calcination Temperature:

At lower temperatures (e.g., 200°C), the TiO_2 nanoparticles maintained a smaller size and uniform distribution.

Increasing the temperature led to higher crystallinity, which, while beneficial for some properties, also caused agglomeration and phase transformations that may reduce effectiveness in specific applications.

2. Effect of Hydrous Solution Concentration:

Higher water content during synthesis resulted in smaller particle sizes but reduced crystallinity.

This trade-off indicates that optimizing water concentration is essential for achieving the desired balance between particle size and crystallinity.

In conclusion, the sol-gel method offers a versatile and controllable approach to synthesizing TiO_2 nanoparticles. The findings from this study suggest that:

- Sample 1, prepared at 200 °C, had the most promising properties, with smaller particle sizes and better distribution.

- Increasing calcination temperature enhances crystallinity but leads to agglomeration and phase transformations.

• Optimizing synthesis conditions is crucial for tailoring the properties of TiO_2 nanoparticles to suit specific applications, such as photocatalysis, where smaller and uniform particles are highly advantageous.

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