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Review Paper

Versatility of Hydrothermal Synthesis: Experimental Design and Comparative Insights

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Abstract

Hydrothermal crystallization is a naturally occurring process involved in mineral formation and has been adapted in laboratories as a versatile approach for synthesizing advanced materials. Over the years, hydrothermal synthesis has gained considerable attention because of its ability to facilitate crystal growth under controlled temperature and pressure conditions, enabling the preparation of materials with tailored structural, morphological, and functional properties. This paper presents an overview of the experimental setup and mechanism of hydrothermal synthesis, with particular emphasis on the roles of major experimental components, comparative results and applications. Its performance is discussed with other commonly employed synthesis methods, including sol–gel and co-precipitation, using findings reported in the scientific literature.These comparisons highlight the potential of hydrothermal synthesis to achieve controlled morphology, improved crystallinity, and desirable functional properties in various material systems, although the outcomes depend on the material composition and experimental parameters. The method is also discussed in the context of green synthesis because water can serve as a relatively safe reaction medium and may reduce the need for certain hazardous organic solvents. Finally, the diverse applications of hydrothermally synthesized materials are discussed to demonstrate the relevance of this approach in materials science and related technological fields. This review aims to provide a concise understanding of the experimental requirements, comparative outcomes and applications.

Keywords: Hydrothermal method, Synthesis, Experimental setup, Comparative analysis.

Introduction

Surface modification is an important approach for tailoring the physicochemical properties of materials while largely preserving their bulk characteristics. By modifying surface morphology, chemical composition, crystallinity, defect concentration, and interfacial properties, various functional characteristics, including wettability, corrosion resistance, catalytic activity, optical response, electrical conductivity, and biocompatibility, can be enhanced. Such modifications are particularly important in functional materials, as surface properties govern their interactions with the surrounding environment and significantly influence their overall performance[1,2].

Among the various techniques employed in materials processing and surface engineering, the hydrothermal method has gained considerable research interest owing to its versatility, relatively simple experimental setup, and ability to control material growth through solution-based reactions. The hydrothermally executed one-pot synthesis successfully integrates multiple target components or dopants into a single reaction vessel, avoiding intermediate isolation and fostering a strong interfacial chemical bond[3].

This method involves the treatment of suitable precursors in a sealed reactor at elevated temperatures and autogenous (self-pressurizing system) pressures. Under these conditions, processes such as dissolution, nucleation, crystal growth, and phase transformation can be controlled by adjusting parameters including reaction temperature, duration, pH, precursor concentration, and mineralizer content[4,5]. The flexibility of these conditions allows the formation of diverse surface architectures, such as nanorods, nanosheets, nanoparticles, and hierarchical structures, depending on the nature of the material and the selected reaction conditions[6].

Another important aspect of hydrothermal processing is its potential to support more sustainable approaches to materials synthesis. Water can be used as the primary reaction medium, reducing the need for certain organic solvents. Moreover, controlled crystallization may eliminate or reduce the need for additional high-temperature treatment in some material systems[4,7]. From an economic perspective, hydrothermal processing can provide a cost-effective alternative to techniques that require expensive vacuum systems, sophisticated deposition equipment, or specialized energy sources. Its relatively simple experimental requirements, flexibility in processing conditions, and ability to produce crystalline materials within a single

processing step make it attractive for laboratory research and selected technological applications[5,8]. However, its economic and environmental advantages depend on factors such as energy consumption, reactor design, precursor selection, purification requirements, and waste management. Therefore, its sustainability and cost-effectiveness must be evaluated in relation to the specific process and competing techniques.

The growing interest in hydrothermal surface modification is also associated with its ability to facilitate controlled material growth, surface coating, functional-layer formation, and micro structural engineering. Hydrothermal processing has been explored for developing functional oxides, semiconductor nanostructures, composite interfaces, and materials for applications in energy storage, photocatalysis, sensing, environmental remediation, and biomedical technologies[3,6,9]. In suitable systems, nanostructures can also be grown directly on substrates, potentially reducing the need for separate deposition steps and binder-assisted assembly. Despite these advantages, challenges related to batch-to-batch reproducibility, reaction time, pressure management, substrate compatibility, and large-scale production continue to require attention[7,10].

Although hydrothermal synthesis has been extensively investigated for various material systems, a comprehensive understanding of the relationships between processing parameters, surface characteristics, and functional performance remains essential. In this context, the present review focuses on the fundamental principles of hydrothermal surface modification, the effects of processing conditions on surface morphology and composition, the resulting structural and functional changes, and their relevance to different applications. Particular emphasis is placed on sustainability, cost-effectiveness, existing challenges, and future research opportunities to provide a critical assessment of hydrothermal processing as a versatile route for developing advanced surface-functional materials.

Mechanism of Hydrothermal Method

Hydrothermal synthesis is an aqueous solution-based technique in which materials are synthesized through chemical reactions in an aqueous medium under elevated temperature and autogenous pressure inside a sealed reaction vessel. The formation of the final product generally involves precursor dissolution, hydrothermal reactions, super saturation, nucleation, crystal growth, and subsequent recovery of the synthesized material.

Liquefaction of the Precursor: As shown in Figure-1 the process begins with the dissolution or dispersion of the selected precursor materials in a suitable solvent, generally deionized (distilled) water. Soluble precursors initiate the process of creating a reactive medium by releasing their constituent ions or molecular species into the solution. Suitable chemical environment is achieved with proper precursor concentration, solvent composition, pH, and degree of dissolution influence, so that subsequent reactions can take place.

Hydrothermal Reactions and Super saturation: Formation of a new solid phase requires many prevailing conditions such as changes in solubility, ion mobility, and reaction kinetics leading to chemical transformations and the formation of product-forming species.

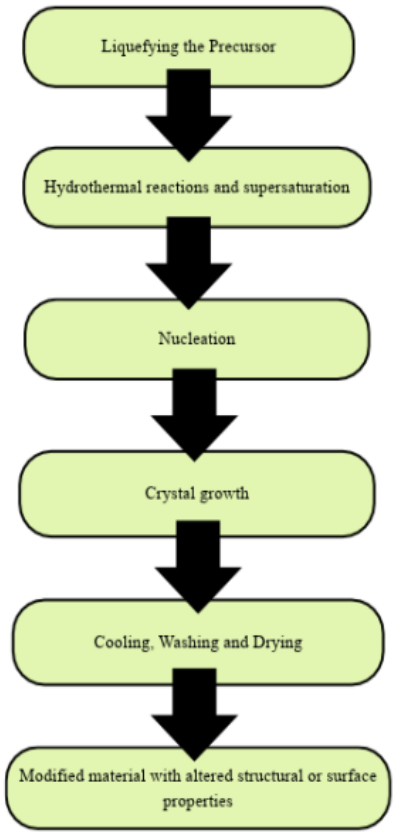


Figure-1 Working Mechanism of Hydrothermal Synthesis.

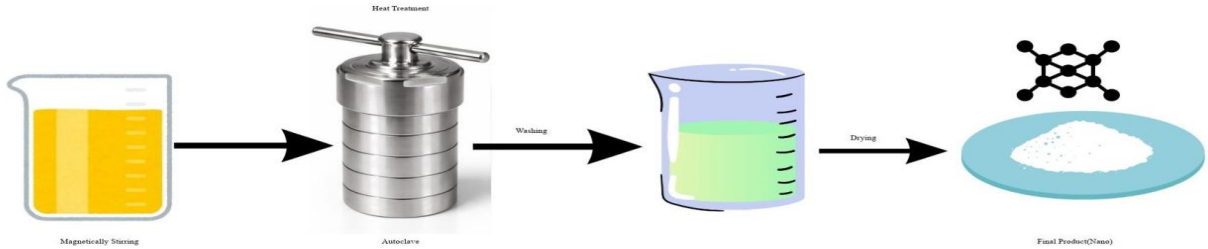


Figure-2: Pictorial Presentation of Hydrothermal Process.

Nucleation: When supersaturation reaches a sufficient level, small stable nuclei are formed through the aggregation and organization of product forming species. The initial structural units from which crystals develop are established by the nucleation process. The nucleation rate and number of nuclei are determined by factors such as supersaturation, temperature, precursor concentration, pH, and the presence of impurities or additives.

Crystal Growth: Following nucleation as shown in Figure-2, additional dissolved species are incorporated into the existing nuclei, and crystal growth is thereby produced. The resulting particle size, morphology, crystallinity, and structural characteristics are governed by the relative rates of nucleation and growth, as well as by the reaction temperature, duration, solution chemistry, and mass transport. Depending on the synthesis conditions, phase formation and structural reorganization may also be facilitated by hydrothermal treatment.

Cooling, Washing, and Drying: After the prescribed reaction period, the autoclave is cooled to a safe temperature before being opened. The solid product is separated from the reaction medium, typically by centrifugation or filtration, and is washed repeatedly with deionized water and, when appropriate, ethanol is used to eliminate residual soluble species and impurities. The recovered material is subsequently dried under controlled conditions to remove remaining solvent, yielding a powder suitable for further characterization or processing.

Formation of the Final Material: The final product is characterized by structural and surface features dictated by the precursor chemistry and hydrothermal processing conditions as shown in Figure-2. Depending on the material system, modifications may be manifested in phase composition, crystallite size, particle morphology, defect concentration, and surface chemistry. The synthesized powder is subsequently characterized for the intended application.

Methodology

The experimental conditions of hydrothermal synthesis play a crucial role in determining the structural, morphological, and functional properties of materials. Parameters such as temperature, reaction time, precursor concentration, pH, and solvent composition influence phase formation, crystallinity, particle growth, and surface characteristics. Appropriate control of these conditions enables the synthesis of materials with tailored properties for specific applications. Therefore, understanding the role of each component and processing parameter is essential for optimizing the hydrothermal synthesis process.

Magnetic Stirrer: A magnetic stirrer, with a hot plate facilitates uniform mixing and controlled heating of the precursor solution during the initial stage of hydrothermal synthesis. Magnetic stirring promotes the homogeneous distribution of precursor components, enhances dissolution, and minimizes concentration gradients within the solution. The hot plate maintains the desired temperature during solution preparation, which can influence precursor solubility and reaction kinetics. Together, these functions help to establish consistent experimental conditions prior to transfer of solution in the autoclave.

Autoclave: The autoclave, is the main component of the hydrothermal method, used to carry out chemical reactions under controlled high-temperature and high-pressure conditions. A strong stainless-steel outer vessel and a Teflon-lined inner container used to contain the precursor solution are the essential components of autoclave. The sealed vessel when kept at desired temperature provides favorable environment so that dissolution, nucleation, and crystal growth could take place. It is always safe to open autoclave when it cools completely naturally.

Hot Air Oven: A laboratory oven with temperature-controller provisions is required for hydrothermal synthesis. Once the precursor solution is placed in an autoclave, hot air oven is used to provide uniform heating. Sealed autoclave is carefully placed in hot air oven fixed at desired temperature as per requirement for a particular reaction period. The hydrothermal reaction takes place inside the sealed autoclave, while the oven provides the required heating conditions. This oven could also be used for the drying of sample at low temperatures.

Muffle Furnace: The muffle furnace is used in the hydrothermal synthesis process for the thermal treatment or annealing of the synthesized material after drying. The dried powder is placed in a suitable heat-resistant crucible and heated at a controlled temperature for a specified duration. This treatment can improve crystallinity, remove residual impurities or volatile substances, and modify the structural and functional properties of the material. The annealing temperature and duration depend on the material composition and the desired properties. After heating, the sample is allowed to cool under controlled conditions before further processing and characterizations.

Pellet Molding Machine: The pellet molding machine is used to convert synthesized powder into compact pellets for further characterization, particularly for electrical, dielectric, and electrochemical studies. The dried and finely ground powder is used to make a pellet of the anticipated dimension and shape using a suitable die and a specific pressure with the help of compressor. Sufficient mechanical strength of the pellet depends on the applied pressure leading to improved particle contact. These pellets could be used for subsequent measurements as per the need of the experiment.

Properties of Hydrothermally made materials

In order to provide an experimental base to the synthesis process

and resultant product material, role of characterization techniques comes into play. Identification of phase, morphology, chemical state, and functional properties of product should be validated properly. Different analytical methods offer complementary information about various properties of the materials (semiconductor oxides, doped materials, dielectric ceramics) to obtain systematic observations leading to specific applications. Following significant properties are relevant for hydrothermally synthesized samples

Structural Property: Commonly used techniques are SEM (Scanning Electron Microscopy), TEM (Transmission Electron Microscopy), XRD (X ray diffraction) and Raman spectroscopy to explore structural property of the sample[11,12]. Structural and vibrational analysis of ZnO is found to be useful for the design and development of a gas-sensor[11]. Similarly, hydrothermally prepared Mn–Zn ferrite nanoparticles were investigated using XRD, Raman spectroscopy, electron microscopy, and vibrating sample magnetometry. Their superparamagnetic response supports further investigation for magnetic and electromagnetic applications[12].

Morphological and chemical-state analysis: SEM reveals particle architecture and surface features, while XPS identifies surface chemical states and oxidation-state variations. Hydrothermally synthesized ZnO nanoflowers and nanorods exhibited morphology-dependent gas-sensing behavior, with hierarchical nanoflowers showing a strong response to nitrogen dioxide[13]. In another study, Fe-doped TiO_2 was prepared hydrothermally, and XPS was used to examine the chemical state of iron alongside investigations of phase formation and optical response. The doped oxide demonstrated improved visible-light photocatalytic activity for methylene blue degradation under the reported conditions[14].

Dielectric Property: Dielectric measurements are important for assessing oxides intended for capacitors, thermistors, and other electronic components. Boulos et al. synthesized $BaTiO_3$ powder hydrothermally and investigated the dielectric properties of the corresponding ceramics. Their results showed that the titanium precursor and sintering conditions influenced the dielectric permittivity and Curie temperature, demonstrating the importance of processing history in developing ferroelectric ceramics[15]. A separate investigation of hydrothermally prepared $BaTiO_3$ showed that controlling phase composition and particle size influenced densification and dielectric performance, with potential relevance to multilayer ceramic capacitors[16].

Magnetic Property: Magnetic hysteresis measurements provide coercivity, remanent magnetization, and saturation magnetization, which help evaluate the magnetic response of oxide nanomaterials. Allaedini et al. synthesized cobalt ferrite nanoparticles hydrothermally and used XRD, SEM, infrared spectroscopy, and VSM to establish their structural and magnetic characteristics. The measured hysteresis parameters demonstrated the magnetic functionality of the prepared ferrite, which is relevant to magnetic devices and catalyst-related applications[17].

Electrochemical Performance: Electrochemical performance is commonly assessed through cyclic voltammetry, galvanostatic charge–discharge, and impedance spectroscopy, which provide insight into charge storage capacity, rate capability, cycling stability, and electrode resistance. Xia et al. reported hollow Co_3O_4 nanowire arrays prepared hydrothermally, demonstrating their suitability as supercapacitor electrodes owing to a porous, self-supported architecture that enabled high capacitance and stable cycling [18].

More directly relevant to MoO_3, a 2024 study compared undoped, Ce-doped, and Fe-doped nanoparticles synthesized hydrothermally, employing XRD, SEM, cyclic voltammetry, and impedance analysis to probe structural, morphological, and electrochemical behavior. Among these, Fe-doped MoO_3 exhibited superior charge storage performance relative to the comparison samples[19].

Similarly, hydrothermally synthesized Cr-doped MoO_3 showed reaction-time-dependent properties, with the 12-hour sample achieving a specific capacitance of 465 F g^{-1} and ~89% retention after 5,000 cycles[20].

Overall, these investigations underscore that characterization is most meaningful when structural and chemical observations are interpreted in direct relation to application specific requirements. Integrating complementary techniques yields a more reliable assessment of how hydrothermal conditions, morphological control, and dopant incorporation govern the functional performance of metal oxides. This holistic approach establishes a clear link between synthesis strategy, material structure, and device applicability, reinforcing the importance of method-driven design in advanced functional materials research.

Comparative Analysis of Synthesis Methods

This section contrasts hydrothermal synthesis with alternative approaches employed for the preparation of similar materials. Table 1 summarizes representative results, underscoring the distinct advantages of the hydrothermal route. The comparative investigation highlights how synthesis methodology critically influences the resulting structural, optical, magnetic, and dielectric properties. In particular, hydrothermal processing demonstrates superior control over crystallinity, defect states, and functional performance, thereby establishing its significance as a versatile and effective strategy for tailoring multifunctional behaviour in transition metal oxides.

Table-1 highlights merits of hydrothermal synthesis as compared to other synthesis method, although intended application also favors some specific synthesis method as per the requirements of the experiments.

Table-1: Hydrothermal Versus Alternative Synthesis Method.

Material Synthesized	Alternative Synthesis Method	Outcome
Cu–Ag-Co doped ZnO/graphene	Sonochemical synthesis	Hydrothermal composite showed a lower band gap, stronger Zn–O–C interfacial bonding and better photocatalytic and antibacterial performance under the reported conditions. Structural and optical characterization, interfacial bonding, charge transfer, photocatalytic dye degradation and antibacterial activity were compared[21].
Spinel lithium manganese oxide ($LiMn_2O_4$)	Sol–gel	Higher specific discharge capacity and better cycling performance were reported for the hydrothermal sample. At the 100th cycle, reported discharge capacities were 99.78 and 114.36mAh g^{-1} for hydrothermal samples at two reported capacity values, compared with 60.25 and 98.67mAh g^{-1} for sol–gel samples, respectively[22].
Green-synthesized magnetic silver nanoparticles	Co-precipitation	More uniform, mono dispersed spherical particles were observed, and the reported magnetization was 3.91emu g^{-1} for hydrothermal synthesis versus 2.83emu g^{-1} for co-precipitation[23].
Titania–silica (TiO_2–SiO_2) composite nanoparticles	Conventional sol–gel	The combined sol–gel–hydrothermal route yielded a larger specific surface area, better resistance to phase transformation and particle growth, and higher photocatalytic activity for methylene-blue degradation[24].
$ZnSn(OH)_6$ (zinc tin hydroxide)	Grinding, co-precipitation and self-templating	PVP-assisted hydrothermal synthesis reportedly gave the highest photocatalytic activity among the compared preparation routes[25].

Conclusions

Beginning of hydrothermal synthesis of materials dates back to the year 1845. Till date this method is extensively used for generating materials. This paper discusses the versatile hydrothermal synthesis method, its basic mechanism and experimental setup. An overview of the performance of hydrothermal method as compared to other commonly employed synthesis methods has also been presented using available scientific literature.

The method is discussed in the context of green synthesis, as water is employed as a relatively safe reaction medium and the requirement for hazardous organic solvents may thereby be reduced. The diverse applications of hydrothermally synthesized materials are further highlighted to demonstrate the relevance of this approach within materials science and related technological domains. A concise understanding of the experimental requirements, comparative outcomes, and applications is provided through this review.

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