



e-ISSN: 2319-8753 | p-ISSN: 2347-6710

# IJRSET

International Journal of Innovative Research in  
**SCIENCE | ENGINEERING | TECHNOLOGY**



# INTERNATIONAL JOURNAL OF INNOVATIVE RESEARCH

IN SCIENCE | ENGINEERING | TECHNOLOGY

Volume 13, Issue 3, March 2024

**ISSN** INTERNATIONAL  
STANDARD  
SERIAL  
NUMBER  
INDIA

Impact Factor: 8.423

# Microwave Irradiation as a Rapid Route for Metal Oxide Nanocomposite Synthesis

Janagamsetty Sivakrishna<sup>1</sup>, Dr.Praveen Meena<sup>2</sup>

Research Scholar, Department of Chemistry, Sikkim Alpine University, Sikkim, India.<sup>1</sup>

Professor, Department of Chemistry, Sikkim Alpine University, Sikkim, India.<sup>2</sup>

**ABSTRACT:** A systematic investigation was carried out on SnO<sub>2</sub> samples doped with x mol% Sb (x = 0, 6, 10, 14, and 18). The materials were synthesized using the polymeric precursor route (Pechini method) and calcined for 4 hours at temperatures ranging from 800 °C to 1200 °C. Structural parameters, including lattice constants, crystallite size, and microstrain, were determined through X-ray diffraction analysis followed by Rietveld refinement. Incorporation of Sb<sub>2</sub>O<sub>3</sub> into SnO<sub>2</sub> resulted in the formation of thermally stable nanoparticles exhibiting reduced particle growth at elevated temperatures. With increasing calcination temperature, all compositions showed a tendency toward similar lattice dimensions; however, the absolute values depended on Sb concentration and reached a maximum at 1100 °C. The observed structural variations suggest that the oxidation state of antimony is influenced by its concentration in the SnO<sub>2</sub> matrix.

**KEYWORDS:** Chemical synthesis; Electron microscopy; X-ray diffraction; Sb-doped SnO<sub>2</sub>; Pigment materials

## I. INTRODUCTION

Microwave irradiation has emerged as an effective alternative heating technique in chemical research and synthesis. Its growing application in both inorganic and organic chemistry is largely attributed to the remarkable enhancement in reaction rates when compared with conventional heating methods. In addition to accelerating reactions, microwave-assisted processes often provide improved product yields, minimize unwanted side reactions, and produce cleaner reaction profiles.

These advantages primarily arise from the distinct mode of energy transfer involved in microwave heating. In conventional synthesis, heat is supplied externally and transferred to the reaction medium through conduction. The energy must pass through the walls of the reaction vessel before reaching the solvent and reactants. Such a process is inherently slow and inefficient, as it relies on the thermal conductivity of the container material and the reaction medium. Consequently, achieving thermal equilibrium between the vessel and the reaction mixture may require extended heating periods, sometimes lasting several hours (Hoogenboom & Schubert, 2007; Sinnwell and Ritter, 2007; Chauveau et al., 2008)

The rapid development of petrochemical-derived polymers has significantly improved modern living standards by enabling durable, lightweight, and cost-effective materials. However, the widespread use of non-degradable petroleum-based plastics has led to serious environmental challenges. Accumulation of plastic waste has disrupted ecosystems, and its management has become a global concern due to limited disposal options. Incineration releases substantial amounts of carbon dioxide along with toxic gases, contributing to global warming and posing health hazards. Landfilling is also constrained by the scarcity of suitable sites and the long persistence of plastic materials in the environment. These issues highlight the pressing need for sustainable, renewable resource-based biodegradable plastics, particularly for short-term packaging and disposable applications. Such materials should be environmentally compatible, free from toxic constituents during production, and capable of undergoing composting alongside natural biodegradable waste.

According to definitions provided by the American Society for Testing and Materials and the International Organization for Standardization, degradable plastics are materials that undergo significant alterations in their chemical structure under specific environmental conditions. Biodegradable plastics, in particular, are decomposed by naturally occurring microorganisms such as bacteria and fungi. A key advantage of these polymers is their ability to undergo biodegradation and subsequent mineralization after disposal, ultimately converting into environmentally benign end products such as carbon dioxide and water through composting processes.

Biodegradable polymers can be broadly categorized into natural and synthetic types. Natural polymers include polysaccharides such as starch and cellulose; protein-based materials like gelatin, wool, and silk; lipid-derived substances such as fats and oils; microbially or plant-produced polyesters such as poly(hydroxyalkanoates) (PHAs); and bio-based polyesters like poly(lactic acid) (PLA). Other materials in this category include natural rubber and various biocomposites. The second group comprises biodegradable synthetic polymers derived from petroleum resources, including poly(butylene succinate) (PBS) and poly(caprolactone) (PCL). Certain biodegradable polymers, such as polyanhydrides and poly(vinyl alcohol), do not strictly fall into either category. Among these materials, biodegradable polyesters have attracted considerable attention as promising substitutes for conventional petrochemical-based thermoplastics. In recent years, aliphatic biodegradable polyesters have gained significant importance due to their expanding applications in consumer products, as well as in medical and healthcare devices.

Microwave-assisted chemical synthesis has been recognized since 1986, when the first reports demonstrating the effect of microwave irradiation on chemical reactions were published by Giguère et al. and Gedye et al. Since then, microwave heating has been widely explored as an efficient technique in materials chemistry, particularly for the preparation of inorganic solids. Its application in ceramic synthesis and the theoretical modeling of microwave–ceramic interactions has been extensively investigated. For example, it was demonstrated that a mixture of silicon and carbon (charcoal) powders, placed in a silica crucible and exposed to microwave radiation at 2.45 GHz in a domestic microwave oven for 4–10 minutes, yields silicon carbide (SiC). SiC is a high-volume engineering ceramic with significant industrial importance, especially in grinding wheels and abrasive tool manufacturing. Microwave technology has also made substantial contributions to nanoscience. It has been applied in the synthesis of single-site catalysts, antimicrobial nanocomposites, flame-retardant materials, advanced electro-optical devices, sensors, ultra-soft magnetic materials, and drug delivery systems. In analytical chemistry, microwave irradiation has been employed for sample digestion and solvent extraction procedures. A notable development in this area is the high-pressure asher focused microwave system, operating at 2.45 GHz with a power output of 650 W, which has been used effectively for the digestion of biological reference materials such as bovine liver.

The application of microwave irradiation in polymer synthesis has gained momentum over the past decade. Microwave-assisted techniques have been successfully implemented in radical polymerization, step-growth polymerization, ring-opening polymerization (ROP), and polymer modification processes. Early work demonstrated microwave-assisted radical crosslinking of unsaturated polyester with styrene. Subsequent reviews have emphasized the efficiency of microwave irradiation in ring-opening polymerization and related polymerization strategies. For instance, the synthesis of polyacrylamide (PAM) under microwave conditions has been reported to proceed efficiently.

In recent years, increasing attention has been directed toward the microwave-assisted synthesis of biodegradable polymers. Examples include the preparation of poly(caprolactone) (PCL) and poly(lactic acid) (PLA). PCL, in particular, has been synthesized via ring-opening polymerization of  $\epsilon$ -caprolactone using stannous octoate [Sn(Oct)<sub>2</sub>] as a catalyst, a compound approved by the U.S. Food and Drug Administration for use as a food additive. These advancements highlight the versatility and effectiveness of microwave irradiation as a powerful tool in modern polymer and materials chemistry.

## II. LITERATURE REVIEW

The crystallization characteristics of blends composed of nylon-11 and poly(vinylidene fluoride) (PVDF) have been systematically examined. Across the full composition range, the presence of distinct melting and crystallization temperatures confirmed that the two polymers crystallize independently rather than forming co-crystalline structures. Nevertheless, blending significantly influenced the crystallization behavior of each component. Incorporation of nylon-11 led to a reduction in the crystallization temperature ( $T_c$ ) of PVDF, while simultaneously enhancing its crystallization rate. Conversely, when nylon-11 was blended with PVDF, both its  $T_c$  and crystallization rate decreased. The crystallization mechanisms of both polymers were altered in the blended state compared to their pure forms. Specifically, the Avrami exponent (Ozawa index) of PVDF in the blend decreased from 3 to 1 as  $T_c$  decreased from 137 °C to 131 °C, whereas in pure PVDF it decreased from 2.5 to 1.3 over the temperature range 145 °C to 135 °C. Similarly, for nylon-11 in the blend, the Avrami exponent decreased from 2 to 1 as  $T_c$  decreased from 159 °C to 153 °C, compared to a decrease from 2.5 to 0.75 in pure nylon-11 over the same temperature interval (Gao and Scheinbeim, 2023).

Microwave irradiation has also been employed for the efficient synthesis of various (meth)acrylamides directly from (meth)acrylic acid and amines under solvent-free conditions. This approach enables high product yields while

preserving the integrity of the (meth)acrylic functional group. Furthermore, when an initiator such as azobisisobutyronitrile (AIBN) is introduced into the reaction mixture, polymerization proceeds in a single step to yield poly(meth)acrylamides (Goretzki et al., 2021).

The influence of microwave irradiation on the crystallization behavior of poly(ethylene terephthalate) (PET)/poly(ethylene-2,6-naphthalate) (PEN) blends has been investigated using wide-angle X-ray diffraction (WAXD) and differential scanning calorimetry (DSC). Microwave exposure was found to markedly modify the crystallization characteristics and significantly increase the degree of crystallinity. For PET/PEN (90/10) blends, crystallinity increased from 15% to 45%, while for PET/PEN (60/40) blends, it rose from 1% to 36%. However, the enhancement in crystallinity was not indefinite; it reached a maximum at an optimal irradiation duration. The cold crystallization enthalpy ( $\Delta H_{cc}$ ) progressively decreased with longer microwave exposure, and the melting enthalpy ( $\Delta H_m$ ) also declined after extended irradiation. These findings indicate that microwave treatment plays a crucial role in modifying polymer crystallization behavior, with the underlying mechanism being dependent on irradiation time and blend composition (Li et al., 2020).

### III. MICROWAVE SYNTHESIS AND MEDICINAL CHEMISTRY

Enhancing research and development productivity remains a major challenge for the pharmaceutical sector, particularly as numerous drug patents approach expiration. To sustain market competitiveness and maintain growth, pharmaceutical companies must adopt strategies that compensate for declining revenues from off-patent drugs. At the same time, advances in genomics and proteomics have dramatically increased the number of potential biological targets for therapeutic intervention. While current treatments are based on a relatively limited number of validated targets, future projections suggest a substantial expansion in target availability. This shift has driven extensive investment in high-throughput genomic and proteomic technologies, combinatorial chemistry, and large-scale biological screening platforms. Despite these advances, lead optimization and medicinal chemistry continue to represent critical bottlenecks in the drug discovery pipeline, as the design and synthesis of biologically active molecules remain both time-intensive and costly. Consequently, considerable attention has been directed toward technologies capable of accelerating the synthesis and evaluation of candidate compounds.

Medicinal chemistry has greatly benefited from progress in combinatorial chemistry and high-throughput synthetic methodologies. These developments have enabled rapid design, preparation, purification, and characterization of extensive compound libraries. Such technological advancements have significantly improved both lead identification and lead optimization processes. Once an efficient synthetic route to a promising lead structure is established, hundreds of structural analogues can be generated and purified in parallel. This approach facilitates rapid exploration of structure–activity relationships (SAR), thereby shortening development timelines and enhancing the probability of identifying potent drug candidates.

A principal limitation of conventional parallel and combinatorial synthesis lies in the optimization of reaction conditions to achieve satisfactory yields and purity. Many synthetic pathways involve prolonged heating steps, making optimization laborious and time-consuming. Microwave-assisted heating under controlled conditions has emerged as a powerful alternative, particularly in medicinal chemistry and drug discovery. By enabling uniform and rapid energy transfer, microwave irradiation can drastically reduce reaction times—from hours or days to minutes or even seconds. This efficiency allows rapid evaluation of multiple reaction parameters within a short timeframe, expediting optimization. Furthermore, microwave-assisted synthesis supports the rapid generation of compound libraries in parallel or automated sequential formats. Beyond accelerating established reactions, this technique also facilitates the discovery of novel reaction pathways, thereby broadening chemical diversity and expanding the scope of biologically relevant chemical space.

Microwave-assisted synthesis has demonstrated significant potential to influence medicinal chemistry across three key stages of the drug discovery pathway: lead generation, hit-to-lead development, and lead optimization. Medicinal chemistry fundamentally seeks to address biological and clinical challenges by designing and refining molecular entities with desired therapeutic profiles. Initial efforts focus on generating suitable molecular probes for mechanistic validation, followed by systematic optimization of biochemical activity, pharmacokinetic behavior, pharmacological efficacy, clinical performance, and competitive positioning of candidate molecules. Throughout these stages, rapid progression is critical. Accelerated timelines translate into competitive advantage, improved utilization of limited resources, faster elucidation of structure–activity relationships (SAR), strengthened intellectual property positioning,

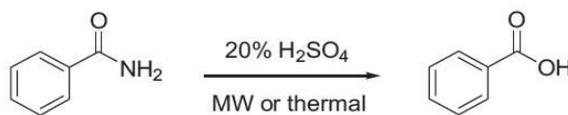
and earlier availability of essential medicines. Within this context, time efficiency directly impacts economic outcomes, and microwave-assisted chemistry has become an indispensable tool in this fast-moving and resource-intensive domain.

Scientific research, including medicinal chemistry, progresses through continuous cycles of hypothesis formulation, experimentation, and data-driven refinement. The markedly reduced reaction times achievable through microwave-assisted synthesis make it particularly suitable for rapid reaction screening and optimization. This capability enables multiple experimental iterations within short timeframes, thereby increasing the number of evaluative decision points per unit time. Although some experimental attempts may not yield the desired outcome, the minimal time investment associated with each trial reduces overall risk, while successful conditions can save hours or even days of conventional heating. The ability to test numerous reaction parameters within a single working session has transformed workflow dynamics, allowing strategic discussions in the morning to translate into experimental outcomes by the afternoon. Owing to these advantages, microwave-assisted synthesis has been widely adopted in pharmaceutical, agrochemical, and biotechnology research environments. It is routinely employed for compound library generation as well as lead optimization programs, where its capacity to accelerate chemical transformations significantly enhances the overall efficiency of the drug discovery process.

### Microwave-Assisted Organic Synthesis (MAOS) – A Brief History

The controlled application of heat has long been central to synthetic chemistry. Although open flames were once common, the invention of the Robert Bunsen burner in 1855 enabled more directed and consistent heating of reaction vessels. Over time, this device was largely replaced by heating mantles, oil baths, and hot plates. In recent decades, however, microwave energy has emerged as a powerful alternative heat source for driving chemical reactions. Originally utilized for food heating following its development by Percy Spencer in the 1940s, microwave technology was subsequently adopted across various industrial sectors, including food processing, drying technologies, and polymer manufacturing. Additional applications have extended to analytical chemistry (microwave digestion and extraction), biochemistry (protein hydrolysis and sterilization), pathology (tissue processing), and medical therapies such as diathermy.

Despite these early technical uses, microwave heating was not introduced into organic synthesis until the mid-1980s. The first reports describing microwave-assisted organic synthesis (MAOS) were independently published in 1986 by the research groups of Richard Gedye and Raymond J. Giguere. Early experiments were typically conducted in sealed Teflon or glass vessels placed inside domestic microwave ovens, often without accurate monitoring of temperature or pressure. Such conditions frequently resulted in uncontrolled heating and, in some cases, hazardous solvent explosions. To mitigate these risks, the 1990s saw the development of solvent-free or “dry-media” microwave methodologies. In this approach, reactants were adsorbed onto microwave-transparent supports such as silica, alumina, or clay, or onto strongly absorbing materials like graphite, sometimes in the presence of catalysts. While these strategies improved safety and enabled the use of standard open-vessel microwave ovens, challenges related to uniform heating, mixing efficiency, temperature measurement, and scalability remained significant limitations.



**Thermal: 1 h, 90 % yield (reflux)**

**MW: 10 min, 99 % yield (sealed vessel)**

**Scheme 2.1 Hydrolysis of benzamide. The first published example (1986) of microwave-assisted organic synthesis.**

An alternative strategy involved conducting microwave reactions in conventional organic solvents under open-vessel conditions. However, atmospheric pressure restricted achievable reaction temperatures to the solvent's boiling point. To overcome this limitation, high-boiling, microwave-absorbing solvents were often employed, though their use complicated product isolation and solvent recovery. The introduction of modern microwave reactors in the mid-1990s—particularly systems pioneered by Christopher R. Strauss—marked a turning point. These instruments allowed precise control and real-time monitoring of temperature and pressure in sealed vessels, significantly improving safety,

reproducibility, and scalability. Contemporary microwave systems now support parallel synthesis, automated sequential processing, and continuous- or stop-flow methodologies, making them highly adaptable for laboratory and industrial applications.

Since its inception, MAOS has prompted debate regarding the origin of its frequently observed reaction rate enhancements and occasional changes in product distribution. Early discussions centered on the possibility of “specific” or “nonthermal” microwave effects, particularly when microwave-heated reactions produced outcomes differing from those obtained under conventional heating at similar apparent temperatures. Current consensus suggests that most rate accelerations arise from thermal and kinetic factors associated with rapid and efficient dielectric heating of polar materials. Nevertheless, the potential contribution of unique microwave–matter interactions continue to be explored in academic research.

Initially, the adoption of microwave technology in synthetic chemistry was gradual, largely due to concerns about safety, reproducibility, and limited understanding of dielectric heating mechanisms. The absence of dedicated instrumentation capable of accurately controlling reaction parameters further hindered early acceptance. However, advances in reactor design now allow precise regulation of time, temperature, and pressure, ensuring reproducible protocols and facilitating scale-up and inter-laboratory transferability. As a result, microwave-assisted synthesis has evolved from a laboratory novelty in the late 1980s into a well-established and widely utilized technique in modern organic synthesis, with substantial growth in related scientific publications since the early 2000s.

#### IV. MICROWAVE CHEMICAL SYNTHESIS

Previous investigations have highlighted multiple advantages of microwave-assisted wet chemical synthesis, including significant reduction in reaction times, improved product yields, enhanced physicochemical characteristics, and the formation of novel material phases. These benefits hold considerable promise for industrial-scale applications. However, successful implementation at the industrial level requires clear differentiation between purely thermal effects resulting from rapid temperature rise and potential non-thermal (athermal) microwave-specific effects. Systematic process optimization is therefore essential to fully harness the technological and economic advantages of microwave-assisted methodologies.

##### Advantages and Significance of Fe<sub>2</sub>O<sub>3</sub>-Based Magnetic Materials

Iron oxide (Fe<sub>2</sub>O<sub>3</sub>)-based magnetic materials are widely used in recording media due to their favorable magnetic properties. With the advent of nanomagnetic and spintronic technologies, increasingly stringent requirements have been imposed on magnetic materials. The fabrication of ultrahigh-density and high-speed data storage systems demands nanoscale magnetic particles with precisely controlled size and morphology.

Conventionally, Fe<sub>2</sub>O<sub>3</sub> powders with monodispersed particles are synthesized through forced hydrolysis of ferric nitrate or ferric chloride solutions, yielding  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub> (hematite), which is subsequently converted to  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> via high-temperature treatment. Achieving strict control over particle size and shape—critical parameters influencing magnetic performance—requires highly dilute precursor solutions. For micron- and submicron-sized particles, ferric ion concentrations are typically maintained around 0.02 M, while nanoscale powders demand even lower concentrations. Additionally, conventional hydrolysis must be carefully regulated and often requires 2–7 days of processing. Even under optimal conditions, the production rate of nanosized iron oxide powders remains extremely low. Industrial deployment of nanomagnetic materials therefore necessitates substantial acceleration of synthesis rates along with increased precursor concentrations.

Microwave-assisted hydrolysis has emerged as a promising alternative. Studies have demonstrated that crystalline hematite can be synthesized via microwave-hydrothermal routes at rates significantly faster than conventional hydrothermal techniques. In certain cases, processing times have been reduced to as little as 30 minutes while enabling higher ferric ion concentrations, thereby markedly increasing production efficiency. An additional advantage is the direct formation of nanoscale  $\gamma$ -Fe<sub>2</sub>O<sub>3</sub> particles, eliminating the need for subsequent high-temperature phase transformation from  $\alpha$ -Fe<sub>2</sub>O<sub>3</sub>. This not only simplifies the synthetic procedure but also avoids microstructural degradation associated with calcination and phase conversion.

### Insertion Electrode Materials

Intercalation or insertion reactions play a pivotal role in rechargeable battery technology. Since the early development of titanium disulfide-based prototype cells, insertion electrode concepts have evolved into commercially successful lithium-ion battery systems employing lithium cobalt oxide cathodes and carbon-based anodes. These materials exhibit high electrical conductivity upon ion intercalation, making them attractive for both small-scale electronics and large-scale energy storage systems.

Extensive research on insertion materials has explored a variety of layered and intercalated compounds, including titanium disulfide, lithium cobalt oxide, intercalated vanadium oxides, layered manganese dioxide structures, and pillared manganese oxides. More recently, vanadium oxide nanotubes have attracted attention due to their unique structural features, such as distinct electrical contact regions and electrolyte-accessible channels, which enhance electrochemical performance.

Conventionally, insertion electrode materials are synthesized via hydrothermal methods that often require several days to complete. Microwave-assisted hydrothermal synthesis has been investigated as a strategy to overcome these limitations. For example, layered vanadium oxides intercalated with tetramethylammonium ions have been prepared within minutes under microwave irradiation. Moreover, extended microwave treatment has been shown to produce new structural phases not accessible through conventional heating. Adoption of microwave techniques for insertion material synthesis offers significant advantages, including reduced processing time, lower production costs, and the potential discovery of novel structures with enhanced electrochemical properties.

### Molecular sieves

Crystalline molecular sieves possess well-defined intraframework cages and channel systems with uniform microporous or mesoporous dimensions, tunable acidity or basicity, and excellent thermal stability. These structural characteristics have led to their extensive use in petroleum refining and petrochemical industries as catalysts and adsorbents. With the emergence of nanotechnology, molecular sieves have also gained importance as nanostructured hosts for advanced functional materials. Their ordered pore architecture enables the fabrication of materials exhibiting tailored optical, optoelectronic, and electrochemical properties suitable for applications such as molecular wires, quantum electronic devices, and nonlinear optical systems. Numerous studies have demonstrated the successful preparation of nanoscale entities within these templates, including organized metal clusters, metal oxides, metal sulfides, isolated conducting polymers, and confined supramolecular assemblies.

The structural diversity of molecular sieve frameworks is highly sensitive to synthesis conditions. Conventional hydrothermal preparation often requires several days to weeks to obtain a targeted crystalline phase. Although increasing the reaction temperature can shorten synthesis time, it may simultaneously promote the formation of undesired or competing structures. Frequently, multiple phases develop concurrently, leading to mixed products rather than a single, well-defined structure. Consequently, the preparation of phase-pure materials with tailored frameworks is often labor-intensive and time-consuming.

Microwave-assisted synthesis has been explored as an alternative strategy for molecular sieve preparation and has demonstrated substantial kinetic acceleration. For example, MgAPO-5 has been synthesized within 20 minutes under microwave conditions compared to 24 hours using conventional methods, while MCM-41 has been obtained in 4 hours instead of the 7–14 days typically required. In addition to shortening reaction times, microwave processing often enhances surface area and structural quality. For instance, microwave-synthesized MgAPO-5 exhibits a significantly higher multipoint surface area compared to conventionally prepared samples. Furthermore, microwave irradiation has proven effective in producing phase-pure crystalline materials. In the synthesis of AlPO<sub>4</sub>-11 & cloverite, microwave treatment yielded pure phases within a few hours, whereas prolonged conventional heating resulted in mixed phases or incomplete reactions. An additional advantage of microwave synthesis lies in improved control over reaction stages; in certain cases, pure products were obtained exclusively under microwave-activated reflux conditions, while conventional methods generated multiple coexisting phases.

### Organometallic Compounds

Organometallic compounds are characterized by the presence of direct metal–carbon bonds, conferring properties distinct from purely inorganic systems. This broad class encompasses a wide range of metals and structural types. Many organometallic species exhibit high reactivity and selectivity, making them valuable as catalysts and synthetic intermediates in organic transformations. Others possess substantial chemical stability and are utilized in applications such as microbiocides, pesticides, anticancer agents, water-repellent formulations, fuel additives for octane

enhancement, antifoaming agents, and mold-release materials. Additionally, organometallic precursors play a crucial role in the preparation of ultrahigh-purity metals and advanced functional ceramics, including semiconductors, conductive materials, magnetic materials, hard coatings, heat-resistant systems, and superconductors.

The synthesis of organometallic compounds typically involves reactions between metals, metal salts, or intermediate organometallic species with inorganic reagents, gaseous reactants, or organic substrates. Reaction durations vary widely: some transformations proceed within an hour, whereas others require several hours or even days under conventional conditions. Microwave-assisted synthesis has been shown to markedly enhance the kinetics of organometallic reactions. Early demonstrations revealed that triphenyltin chloride and triphenyltin hydroxide could be prepared in sealed vessels under microwave irradiation within minutes, compared to hours required by conventional reflux methods. Similarly, microwave-promoted redistribution reactions have enabled rapid synthesis of alkyl- and arylhalogermanes with high yields in open reactors, significantly reducing reaction times relative to traditional high-temperature procedures. Studies on Group 6 zerovalent metal carbonyl complexes (Cr, Mo, W) have further confirmed that microwave irradiation can provide rate enhancements of up to two orders of magnitude along with improved yields compared to conventional heating. These findings collectively underscore the efficiency and versatility of microwave-assisted methodologies in organometallic chemistry.

Compound	Microwave vs reflux			
	Time (min)	Temp (°C)	Yield (%)	Rate enhancement
(dppe <sup>□</sup> )Mo(CO) <sub>4</sub>	0.5/25 #	180/120	80/68	50
(dppe <sup>□</sup> )Mo(CO) <sub>4</sub>	2/180	180/155	64/55	90
(bipy <sup>@</sup> )Mo(CO) <sub>4</sub>	0.5/90	180/110	99/88	180
(dppe)Cr(CO) <sub>4</sub>	0.5/13 5	180/135	86/41	270
(dppm*)W(CO) <sub>4</sub>	5/288 0	180/165	62/51	576

Table 1. Synthesis of Group 6 (Cr, Mo, W) zerovalent organometallic carbonyl compounds:

#synthesis method: microwave / reflux

□ = bis(diphenylphosphino)ethane, @ = 2,2'-bipyridine, \* = bis(diphenylphosphino)methane

### Polymers

Poly( $\epsilon$ -caprolactam), commonly known as nylon-6, is a widely used engineering thermoplastic valued for its balanced thermal stability, mechanical strength, and chemical resistance. Its synthetic fiber has extensive applications in textiles, industrial yarns, and engineering plastics, with global production exceeding several million metric tons annually. Industrially, nylon-6 is produced via hydrolytic polymerization of  $\epsilon$ -caprolactam under high-pressure conditions at temperatures of 250–270 °C for approximately 12–24 hours. This process is energy-intensive and time-consuming.

Microwave-assisted polymerization has been explored as an alternative approach to enhance efficiency. It has been demonstrated that poly( $\epsilon$ -caprolactam) can be synthesized under microwave irradiation in a nitrogen atmosphere at atmospheric pressure. At 250 °C, polymerization was completed within 2 hours, producing material with yield, purity, yield strength, and tensile strength comparable to conventionally manufactured commercial products. This substantial reduction in reaction time highlights the potential of microwave heating to improve productivity while maintaining material performance. Poly( $\epsilon$ -caprolactone) (PCL) is an important biodegradable polyester extensively used in biomedical devices, controlled drug delivery systems, and environmentally friendly packaging materials. It is typically synthesized through catalyzed ring-opening polymerization of  $\epsilon$ -caprolactone in bulk or solution. Despite extensive research into catalytic systems, conventional polymerization methods often require more than 10 hours, and in some cases several days, to reach completion.

Microwave-assisted ring-opening polymerization has provided a significantly accelerated alternative. Using sealed reaction vessels and a catalyst such as stannous octanoate [Sn(Oct)<sub>2</sub>] at a concentration of 0.1 mol%, PCL has been successfully synthesized at 180 °C within 30 minutes. The process achieved yields of approximately 90% and produced polymers with high average molar mass (around 124,000 g/mol). These findings demonstrate that microwave-assisted

techniques can drastically shorten reaction times while delivering high-quality biodegradable polymers suitable for advanced applications.

## V. CONCLUSIONS

The observed expansion of the SnO<sub>2</sub> unit cell is attributed to the substitution of Sn<sup>4+</sup> ions by Sb<sup>3+</sup> within the crystal lattice. At antimony concentrations corresponding to heat treatment above 900 °C, the coexistence of Sb<sup>3+</sup> and Sb<sup>5+</sup> species contributes to the stabilization of crystallite size and microstrain. In samples calcined at 800 °C, the proportion of Sb<sup>5+</sup> increases progressively with higher antimony content. High-resolution transmission electron microscopy (HRTEM) analysis revealed that undoped SnO<sub>2</sub> exhibits greater particle agglomeration compared to Sb-doped samples. These findings demonstrate that antimony doping is an effective strategy for regulating particle size and enhancing the thermal stability of SnO<sub>2</sub> by suppressing grain growth at elevated temperatures.

Both heat-bodying (HB) and microwave irradiation (MI) treatments were found to increase the viscosity of soybean oil. Pressurized differential scanning calorimetry indicated that HB significantly enhanced oxidative stability relative to untreated oil, while MI-treated samples exhibited even greater oxidative resistance than HB-treated counterparts. Notably, despite the increase in viscosity, both treatments—particularly microwave irradiation—substantially reduced the pour point compared to untreated soybean oil. Proton nuclear magnetic resonance (<sup>1</sup>H NMR) analysis confirmed the absence of oxidative degradation in all treated samples, suggesting that the observed decrease in pour point is likely associated with the formation of cyclic triacylglyceride structures that contribute to viscosity enhancement.

Tribological evaluation revealed increased friction coefficients for both HB- and MI-treated oils compared to untreated samples. Microwave-treated oil, in particular, produced more pronounced wear scars on the contact surfaces during lubricity testing. Although microwave irradiation shows potential for improving the low-temperature flow properties of soybean oil, it does not necessarily yield superior performance in vegetable oil-based lubricant applications.

## REFERENCES

- Fang, Q.; Hanna, M.A. Rheological properties of amorphous and semicrystalline poly(lactic acid) polymers. *Ind. Crop Prod.* 2022, 10, 47-53.
- Mathew, A.P.; Oksman, K.; Sain, M. Mechanical properties of biodegradable composites from poly lactic acid (PLA) and microcrystalline cellulose (MCC). *J. Appl. Polym. Sci.* 2020, 97, 2020-2021.
- Huda, M.S.; Mohanty, A.K.; Drzal, L.T.; Schut, E.; Misra, M. "Green" composites from recycled cellulose and poly(lactic acid): Physico-mechanical and morphological properties evaluation. *J. Mater. Sci.* 2022, 40, 4221-4229.
- Graupner, N. Application of lignin as natural adhesion promoter in cotton fibre-reinforced poly(lactic acid) (PLA) composites. *J. Mater. Sci.* 2023, 43, 5222-5229.
- Huda, M.S.; Drzal, L.T.; Mohanty, A.K.; Misra, M. Effect of fiber surface-treatments on the properties of laminated biocomposites from poly(lactic acid) (PLA) and kenaf fibers. *Compos. Sci. Technol.* 2008, 68, 424432.
- Avella, M.; Bogoeva-Gaceva, G.; Buzarovska, A.; Errico, M.E.; Gentile, G.; Grozdanov, A. Poly(lactic acid)-based biocomposites reinforced with kenaf fibers. *J. Appl. Polym. Sci.* 2008, 108, 3542-3551. *Materials* 2009, 2 923
- García, M.; Garmendia, I.; García, J. Influence of natural fiber type in eco-composites. *J Appl. Polym. Sci.* 2008, 107, 2994-3004.
- Bax, B.; Müssig, J. Impact and tensile properties of PLA/Cordenka and PLA/flax composites. *Compos. Sci. Technol.* 2008, 68, 1601-1607.
- Bodros, E.; Pillin, I.; Montrelay, N.; Baley, C. Could biopolymers reinforced by randomly scattered flax fibres be used in structural applications. *Compos. Sci. Technol.* 2007, 67, 462-470.
- Hu, R.; Lim, J.-K. Fabrication and mechanical properties of completely biodegradable hemp reinforced PLA composites. *J. Compos. Mater.* 2007, 41, 1655-1669.
- Tokoro, R.; Vu, D.M.; Okubo, K.; Tanaka, T.; Fujii, T.; Fujiura, T. How to improve mechanical properties of polylactic acid with bamboo fibers. *J. Mater. Sci.* 2008, 43, 775-787.
- Shikamoto, N.; Ohtani, A.; Leong, Y.W.; Nakai, A. Fabrication and mechanical properties of jute/PLA composites. In *22nd Technical Conference of the American Society for Composites 2007: Composites: Enabling a New Era in Civil Aviation*; Curran Associates, Inc: Red Hook, NY, USA, 2007; pp. 151:1151:10.
- Huda, M.S.; Drzal, L.T.; Mohanty, A.K.; Misra, M. Wood-fiber-reinforced poly(lactic acid) composites: Evaluation of the physico-mechanical and morphological properties. *J. Appl. Polym. Sci.* 2006, 102, 4856-4869.



14. Huda, M.S.; Drzal, L.T.; Mohanty, A.K.; Misra, M. Effect of chemical modifications of the pineapple leaf fiber surfaces on the interfacial and mechanical properties of laminated biocomposites. *Compos. Interface* 2008, 15, 169-191.
15. Zhao, Y.-Q.; Lau, K.-T.; Liu, T.; Cheng, S.; Lam, P.M.; Li, H.-L. Production of a green composite: Mixture of poly(lactic acid) and keratin fibers from chicken feathers. *Adv. Mat. Res.* 2008, 47-50, 1225-1228.
16. Cheung, H.-Y.; Lau, K.-T.; Tao, X.-M.; Hui, D. A potential material for tissue engineering: Silkworm silk/PLA biocomposite. *Compos. Part B-Eng.* 2008, 39, 1026-1033.
17. Wang, K.-H.; Wu, T.-M.; Shih, Y.-F.; Huang, C.-M. Water bamboo husk reinforced poly(lactic acid) green composites. *Polym. Eng. Sci.* 2008, 48, 1833-1839.



INTERNATIONAL  
STANDARD  
SERIAL  
NUMBER  
INDIA



# INTERNATIONAL JOURNAL OF INNOVATIVE RESEARCH

IN SCIENCE | ENGINEERING | TECHNOLOGY

 9940 572 462  6381 907 438  [ijirset@gmail.com](mailto:ijirset@gmail.com)



[www.ijirset.com](http://www.ijirset.com)

Scan to save the contact details