**Handbook of Greener Synthesis** of Nanomaterials and Compounds

## **VOLUME 1**

**Fundamental Principles** and Methods

> **Edited by** Boris I. Kharisov Oxana V. Kharissova



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**Volume 1: Fundamental Principles and Methods**

*Edited by*

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## **Preface**

Modern techniques to produce nanoparticles, nanomaterials, and nanocomposites are based on physical and chemical (of zerovalent (elemental) metals, their oxides and salts), various nanomaterials and nanocomposites are examined. Main (of zerovalent (elemental) metals, their oxides and salts), various nanomaterials and nanocomposites are examined. Main methods for green synthesis include the use of *natural products* (*i.e*., tea, banana, coffee, wine, glucose, sugar, extracts of various plants or parts of plants, simple amino acids), acting as capping, chelating agents, and/or reductants (mainly polyphenols) in these processes. Indeed, a variety of plant biomolecules (vitamins, alkaloids, sugars, enzymes/proteins, etc.) can contribute to the formation and stabilization of nanoparticles. These techniques are environmentally friendly, simple, and mainly one-step method as a part of biosynthesis applies several bacteria for nanoparticle formation. The yield of final products, size and method as a part of biosynthesis applies several bacteria for nanoparticle formation. The yield of final products, size and morphology of nanoparticles, extraction of biologically produced metal nanoparticles will be discussed.

Currently, in addition to the biological methods above, several *physical* and *chemical* routes for nanomaterials fabrication can be considered as green methods, for example UV-irradiation, ultrasound- or microwave-assisted reactions (dry media synthesis), use of special microreactors, water, liquid and supercritical  $CO<sub>2</sub>$ , some ionic liquids, supercritical fluids, glycerol and polyethylene glycols as nonharmful solvents, green catalysis (or catalyst-free reactions) in organic synthesis, incorporation of reactants in clays, zeolites, silica, or alumina. Methods of green synthesis are frequently simple, inexpensive, and efficient, without hazards for the environment.

We are extremely grateful to all authors for their hard work in creation of the present Handbook and hope that this collection will be a useful guide for developing novel materials and reaction routes for a sustainable and clean future.

> *Editors* Boris I. Kharisov Oxana V. Kharissova

## **Synthesis of nanomaterials by mechanochemistry**

# **11**

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#### 11.1 Introduction

During the previous decades, several achievements in the creation of nanomaterials have pulled in tremendous considerations in multidisciplinary research areas along with commercial [1], agricultural [2] and medicinal [3] packages. Moreover, nanomaterials usually possess great mechanical, magnetic and electrical residences etc., which lessen the impurity on fabric surfaces and lead to superior functional advantages [4].

Nano-size particles of less than 100 nm in diameter are currently attracting more attention for the wide range of new applications in many fields and industries because of some effects like small particle dimension, high surface to volume ratio and their quantum confinement [5,6].

Nano chemistry is the understanding and control of matter of sizes roughly in the range of 1 to 100 nanometers. If one of the dimensions is in this range, it is considered a nanoparticle. Bulk materials when reduced to the nanoscale show some properties (melting point, physical strength, surface area, penetration power, electric conductance, optical effect, magnetism etc.) which are different from what they exhibit on a macro scale enabling unique applications. These materials can be either natural or engineered.

At nanoscale, gravity would become less important, whereas surface tension and van der Waal forces would become more important [7]

In the meantime, the remarkable surface properties of nanomaterials enrich them as special materials with better practical exhibitions material to progressively sensitive conditions [8,9].

The name mechanochemistry became carried out to the kind of reactions accomplished via mechanical strength. A narrower area, tribochemistry, became used for reactions generated by way of friction at some point of the milling

Mechanochemistry deals with chemical transformations induced by mechanical means such as compression, shear, or friction. In mechanochemical processes, the energy required for the activation of chemical reactions is usually provided by mechanical force as similar to thermochemistry, photochemistry, or electrochemistry, where energy is provided by heat, light, or electrical potential, respectively.

Mechanochemical processes have a long history and continue to be of high importance because these can quantitatively and rapidly promote solid-phase reactions only using nominal amounts (wet milling or liquid-assisted grinding). In conventional chemical synthesis, the solvent often plays a key role in energy dispersion, dissolution/solvation and transportation of chemicals.

Mass and energy transport may be hampered in solvent-free reactions. The efficient mixing process under ball milling or grinding can offer an effective way out from this problem, enabling the reactions between solids or solidified reagents in solvent-free conditions. Despite these advances in solvent-free molecular synthesis, solvents remain prevalent for the isolation of products in satisfactorily pure forms. ''Liquid-assisted grinding'' (LAG) has been introduced to provide a potential alternative to minimize the use of solvents in (nano)materials syntheses.2 In contrast to ''dry milling'' mechanochemical processes, LAG may offer advantages such as greater time efficiency, proper usage of materials and energy and can result in the discovery of new or improved reactivity and products [10].

The first part of the chapter is devoted to this subject and discusses various topics, including the theoretical background for Mechanochemistry and relation between milling conditions and phase formation, the course of mechanochemical reactions including amorphization and nucleation phenomena triggered by high-energy milling, the formation of intermediate phases during a reaction, the evolution of the crystallite size and the particularities of the nanocrystalline products and the preparation of nanosized pharmaceutical drugs.

#### 11.2 Theoretical background

Generally, the mechanochemical treatment of solids leads to a positive influence on the solid – liquid kinetics  $[11,12]$ . It has been documented by  $[13]$  that the breaking of bonds in the crystalline lattice of solids brings about a decrease  $(\Delta E^*)$  in the activation energy and an increase in the rate of leaching

$$
\Delta E^* = E - E^* \tag{11.1}
$$

$$
k^* = \text{kerp}\left(\Delta E^* / RT\right) \tag{11.2}
$$

where E is the apparent activation energy of the non-disordered solid,  $E^*$  is the apparent activation energy of the disordered solid k, R and T stand for the rate constant of leaching for the non-disordered solid, (the pre-exponential factor) gas constant and reaction temperature, respectively; k\* is the rate constant of leaching for the disordered solid.

If  $E > E^*$ , then exp ( $\Delta E^* / RT$ ) > 1 and thus it follows from eq. (11.2) that  $k^* > k$ , i.e., the rate of leaching of a disordered solid is greater than that of an ordered mineral.



**Fig. 11.1** Schematic diagrams representing the mutual dependence of physicochemical characteristics and reactivity of mechanically activated solids: k – the rate constant of leaching,  $S_i$  – surface area,  $X$  – structural imperfections,  $E$  – applied energy [14].

Senna have analyzed the effect of surface area and the structural disordering on the leachability of mechanically activated solids [14]. In order to solve the problem, whether the surface area or structural parameters are predominant for the reactivity the rate constant of leaching was divided by the proper surface area and plotted against the applied energy by milling (Fig. 11.1).

For example, if the rate constant of leaching divided by the surface area remains constant with respect to the applied energy, as shown in Fig. 11.1A, then the measured surface area may be the effective surface area and at the same time, the reaction rate is insensitive to structural changes. If, on the other hand, the value k/Si decreases with applied energy, as shown in  $Fig. 11.1B$ , then the surface area is probably not the effective surface area. In the third case where k/Si increases with increasing applied energy, as shown in Fig. 11.1C, the surface area Si, may be again the effective surface area, with an overlapping effect of the structural imperfection as a result of mechanical activation. Alternatively, when k/Si and X vary parallel to each other with E, as shown in Fig. 11.1D, or the value  $k/Si$  is proportional to X, as shown in Fig. 11.1E, it seems more appropriate to accept the chosen Si as an effective surface area [15].

#### 11.3 Mechanochemical reactors

Processing in mechanochemical reactors can be completed in an assortment of ways. The most straightforward is the laboratory mortar and pestle. This hand processing forms can incite countless mechanochemical responses, which do not require the outperforming of a high-vitality hindrance. Ball plants are utilized when higher vitality is included and when the processing time requires hours or even days. Research center vibrators of the Wiggle-Bug type are exceptionally productive in processing small



Fig. 11.2 A ball mill consists of a hollow cylindrical chamber [17].

samples. Exceptionally high-vitality vibrators, for example, fast attritors or treated steel ball plants of high effect are utilized for delayed high vitality processing as in mechanical alloying or amorphization of hard crystalline solids. This area features a portion of the significant utilizations of mechanochemistry in natural blend utilizing mortar and pestle, high speed ball milling (HSBM) process, high intensity grinding (HIG) [16].

#### *11.3.1 Ball milling*

This mechanical method of production of nanomaterials works on the principle of controlled impact. The size reduction is achieved through the impact caused when the balls drop from the top of the chamber containing the source material.

A ball mill consists of a hollow cylindrical chamber (Fig. 11.2) which rotates about a horizontal axis, and the chamber is partially filled with small balls made of steel, tungsten carbide, zirconia, agate, alumina, or silicon nitride having diameter generally 10 mm. The inner surface area of the chamber is lined with an abrasion-resistant material like manganese, steel, or rubber. The magnet, placed outside the chamber, provides the pulling force to the grinding material, and by changing the magnetic force, the milling energy can be varied as desired. The ball milling process is carried out for up to approximately 150 h to obtain uniform-sized fine powder (Fig. 11.3).

In high-energy ball milling, vacuum or a specific gaseous atmosphere is maintained inside the chamber. High-energy mills are classified into attrition ball mills, planetary ball mills, vibrating ball mills, and low-energy tumbling mills. In high-energy ball milling, formation of ceramic nano-reinforcement by in situ reaction is possible.

It is an inexpensive and easy process which enables industrial scale productivity. As grinding is done in a closed chamber, dust, or contamination from the surroundings is avoided. This technique can be used to prepare dry as well as wet nano powders. Composition of the grinding material can be varied as desired. Even though this method has several advantages, there are some disadvantages. The major disadvantage is that the shape of the produced nanoparticles is not regular. Moreover, energy consumption is relatively high, which reduces the production efficiency. This technique is suitable for the fabrication of several nanocomposites, which include Co- and Cu-based nanomaterials, Ni-NiO nanocomposites, and nanocomposites of Ti, C [18].



**Fig. 11.3** Pictorial representation of different types of mechanochemically synthesized (nano) materials [25].

#### 11.4 Mechanochemical synthesis of metal nano-particles

Mechanochemical reduction could be an effective route for the synthesis of metal nanoparticles (generally noble metals) with improved structural and catalytic properties. Mechanochemically prepared  $Ag/Al<sub>2</sub>O<sub>3</sub>$  nanomaterials have been prepared and tested in the selective catalytic reduction (SCR) of NOx using hydrocarbons in the presence and absence of hydrogen [19,20].

The catalyst exhibited a remarkable increase in activity at lower reaction temperatures as compared to traditional catalysts prepared via standard wet impregnation methods. The enhanced activity of the mechanochemically synthesized catalyst was related to surface modifications that provided an increased affinity towards hydrocarbons relative to water, consequently reducing the activation barrier for the reduction of NOx.

The straightforward two-step process involved a dry mixing of a precursor metal salt (e.g., a metal acetate) with carbon nanotubes (single- or multiwalled) followed by heating in an inert atmosphere in the absence of any solvent, reducing agent or electric current. The proposed methodology can be, in principle, scalable to multigram quantities and generally applicable to various other carbon substrates (e.g., carbon nanofibers, expanded graphite and carbon black) and many metal salts (e.g., Ag, Au, Co, Ni and Pd acetates) [21].

Generally, mechanochemical synthesis has attracted considerable scientific and technical interest in recent years as a consequence of the unique nanostructures and properties developed by this process. High-energy milling can be used to induce a wide variety of solid-solid and even solid-liquid reactions [22,23]. A chemical reaction is believed to occur at the interfaces of the nanometer sized grains that are continually regenerated during milling. As a consequence, reactions, that would normally require high temperatures to occur, due to separation of the reacting phases by the product phases, can occur at lower temperatures in a ball mill. An important feature of the mechanochemical synthesis is the rapid refinement of the particle microstructure, i.e. grain size or crystallite size, during milling. While powder particle sizes generally decrease only to the micrometer level, a nanometer grain size can develop within each particle [23].

Farit Kh. Urakaev 2012. Have studied the mechanochemical synthesis of nanoparticles by a dilution method. The results showed that studying the kinetics of mechanochemical processes can be recommended to find the specific features for the numerical estimation of the mass transfer coefficient in a mechanochemical reactor [24].

C.-F. Zhou et al,. 2009 studied the synthesis of polyaniline nanofibers, by a simple solid-state mechanochemical reaction and template-free method. Another study by Venkatesha Narayanaswamy et al., 2019 regarding the synthesis of Graphene  $o$ xide-Fe<sub>3</sub>O<sub>4</sub> nanocomposites by a mechanochemical method to yield extremely heterogeneous particles. The nanocomposites milled for deferent extents of time have shown deferential behavior for magneto thermic heating. The magnetic composites synthesized by the ball milled method were able to retain the functional groups of graphene oxide [25].

From supported metal nanoparticles, composite nanomaterials to metal oxide nanoparticles and metal organic frameworks (MOFs) including covalent organic frameworks (COF). Examples of relevant materials include zinc-based ZIF-8 structures (BIT-11), Cu  $(INA)_2$  and Cu-containing HKUST-1 as well as supported noble metals on aluminosilicates, and graphene [26].

#### 11.5 Mechanochemical synthesis of complex ceramic oxides

In general, complex ceramic oxides exhibit a variety of properties, making them attractive for a diversity of different applications. The main effects associated with each direct mechanochemical synthesis (regarding the formation of the final product at once at some stage in milling) and the mechanochemical activation-based synthesis (relating to excessive-electricity milling, used to increase the reactivity of the powders, which is

accompanied through an annealing step to set off the formation of the very last product) of complex oxides with a variety of properties is investigated [27].

Ferroelectric, antiferroelectric, piezoelectric, relaxor and multiferroic oxides as well as oxides with magnetic, semiconducting and catalytic properties are highlighted, in particular the influence of mechanochemical activation on both the phase formation during subsequent annealing and the preparation of the final ceramics, the influence of milling conditions, such as humidity, and the effect of the hydration state of the reagents on the course of the mechanochemical reaction as well as the issue of contamination during milling [27].

In metallic–ceramic composite powder formation, the preparation of metals and alloys by reducing their salts or oxides with more reactive metals is commonly known as metallothermic reduction. This reaction is expressed in general by the equation:

$$
M_A X + M_B > M_A + M_B X \tag{11.3}
$$

where a metal  $M_A$  is reduced by a more reactive metal  $M_B$  (reductant) to the pure metal  $M_A$ .  $M_A X$  and  $M_B X$  are oxides, chlorides, sulphides and other salts.

Metallothermic reactions are characterized by a large negative free-energy change and therefore they are thermodynamically feasible at room temperature [27].

Mechanochemical synthesis has recently received a lot of interest for processing ceramic powders, opening up better approaches for creating mechanically significant oxides with complex compositions one of the most significant research regions is the investigation of the mechanisms and kinetics of mechanochemical reactions, which structure the basis of the further advancement of mechanochemical synthesis [19,21,27].

#### 11.6 Mechanisms and kinetics of mechanochemical reactions

Based on the theory of glancing collision of rigid bodies, the theoretical calculation of t–P–T conditions and the kinetics of mechanochemical processes are possible for the reactors that are intended to perform different physicochemical processes during mechanical treatment of solids. According to the calculations, the 'physicochemical' effect of mechanochemical reactors is due to short-time impulses of pressure  $(\Delta P = \sigma \sim 1010-1011$  dyn cm−2) with shift, and temperature  $\Delta T(x, t)$ . The highest temperature impulse  $\Delta T \sim 103$  K are caused by the 'dry friction' phenomenon.

Typical spatial and time parameters of the impact–friction interaction of the particles with a size R  $\sim$  10−4 cm are as follows: localization region,  $\Delta x \sim 10-6$  cm; time, Δt ∼ 10−8 s. the effect of short-time contact fusion of particles treated in various can play a key role in the mechanism of activation and chemical reactions for wide range of mechanochemical processes.

The spatial and time characteristics of the fused zone are such that quenching of non-equilibrium defects and intermediate products of chemical reactions occurs; solidification of the fused zone near the contact point results in the formation of a 'nanocrystal or Nano amorphous state'. The calculation models and the kinetic equations permit quantitative estimates of rate constants to be performed for any particular processes of mechanical activation and chemical transformation of the materials in ball mills [28].

The main merits of applying mechanochemistry to various functional materials are charge transfer across a boundary of dissimilar solids with simultaneous polarization, size reduction and homogenization without external heating. This could prompt development in organic syntheses starting from the solid state, involving solvent-free and subsequently green chemical processes. Affordable metals could be used instead of noble metals in catalysis process since the mechanical stress applied brings excited state. Mechanochemical processes is expected to be applied further in different fields including biocompatible complexes and pharmaceutics [29,30].

Seyedia et al.,2015 clarified that Nano-size particles of less than 100 nm in diameter are currently attracting more attention for the wide range of new application in many fields and industries because of some effects like small particle dimension, high surface to volume ratio and quantum confinement, [31–33] Mechanochemical processing is a new method involving the mechanical activation of solid-state chemical reactions displacement during ball milling which has been widely researched in the recent years and were used to synthesize of metallic, oxide and sulfide nanoparticles, [31,32]. The other competitor methods that have been developed to synthesize nanoparticles include sol-gel, vapor phase condensation, sputtering, wet chemical precipitation and hydrothermal synthesis.

The main advantage of mechanochemical synthesis is that in this method the process carried out in solid state and agglomeration did not occur in comparison to the above methods. In addition, the control of the overall particle size distribution in this method is easier, Yang et al. (2004). During milling fracture, deformation and welding of the reactants take place. Chemical reactions occur at the interface of reactants, consequently the chemical reactions that require high temperature will occur at low temperature without any external applied heat,[ 31, 35,36]. Mechanochemical synthesis was done by displacement reaction according to the following reaction:

$$
AxC + yB = xA = ByC
$$
 (11.4)

where AxC and B are the reactants, A is favorable product and ByC is by-product of the reaction. By choosing suitable conditions such as milling parameters, suitable BPR and the stoichiometric ratio of starting materials, mechanochemical processing can be used to synthesize nanocrystalline particles. After the milling process, the nanoparticles will be surrounded with by-product materials which are dispersed within this soluble salt matrix, selective removal of the matrix phase must be done by washing the resulting powder with appropriate solvents.

After that nanoparticles will be formed as small as 5 nm, [34,36]. Nowadays fabrication of iron oxide nanoparticles has attracted a lot of attention due to their unique properties and industrial capability.

#### 11.7 Aldol condensation

The reaction of two carbonyl compounds, one as a nucleophile (in the form of an enol or enolate derivative) with another acting as an electrophile, gives rise to a b-hydroxycarbonyl compound. This on dehydration produces an a,b-unsaturated carbonyl compound which is commonly referred to as the aldol condensation reaction. These reactions are utilized to underivative ketones and aldehydes with simple acid or base catalysis, the reaction is usually carried out using preformed enolates which serve both to increase the driving force of the reaction and to ensure that the desired chemoselectivity is achieved [30].

#### *11.7.1 Reformatsky reaction*

Toda et al. demonstrated that Reformat sky reactions proceed efficiently in the absence of solvent. The solvent-free reaction was carried out by mixing aldehyde or ketone, a-bromo ester and Zn-NH4C1 in an agate mortar and pestle while keeping the mixture at room temperature for several hours. Treatment of the aromatic aldehydes with ethyl bromoacetate and Zn-NH<sub>4</sub>C1 gave the corresponding Reformatsky reaction products in very high yield (80–94 percent) (Fig. 11.4).146 The reaction yield was found to be much better than that obtained by the reaction in dry benzene–ether solution (61–44 percent); the additional advantage of solvent-free Reformatsky reaction is that it does not require the use of an anhydrous solvent [30].



**Fig. 11.4** Synthesis of b-hydroxy ester [30].

#### *11.7.2 Luche reactions*

Toda and coworkers also found the Luche reactions proceed efficiently in the absence of solvent, although Grignard reactions under similar conditions are not very efficient and give more reduction product than the normal carbonyl addition product. The solvent-free Luche reactions can be carried out by a very simple procedure and give very high yield in comparison to the reaction under conventional reaction conditions using solvent [30,35–36].

#### 11.8 Mechanochemical synthesis of carbon nanotubes

In a more recent report, carbon nanotubes were also utilized as support for Pd NPs using a similar solvent less dry milling method in the absence of any reducing agent or electric current  $[25]$ . 16 Pd NPs (1–3 nm size) could be uniformly dispersed on the support and the mechanochemically prepared Nano catalyst exhibited excellent activities in Suzuki cross coupling reactions with high turnover numbers (TON) and turnover frequencies (TOF). Pd NPs prepared by thermal annealing (300 °C) formed larger NPs due to agglomeration as compared to Pd NPs prepared at room temperature, thereby leading to reduced catalytic activities. A similar mechanosynthesis technique can be utilized to prepare functionalized ultra-small Au NPs in gram scale using a bottom-up approach [17]. The size of AuNPs could be controlled by varying the ligand/precursor ratio, milling time and/or the nature of the ligand [25].

Along these lines, a simple and scalable method for the preparation of supported gold, palladium and gold–palladium bimetallic catalysts via the physical mixing of the acetate salts of metals followed by a simple heat treatment was also recently described.

#### 11.9 Mechanochemical dry synthesis of nanocrystalline semiconductors

Nanocrystalline semiconductors, also called quantum dots, appear to be interesting objects for studying basically novel properties of matter. In general, reducing the size of semiconductors down to the nanometer length scale is expected to increase the energy gap of the semiconductor structure and, consequently, to give rise to shorter wavelengths optical emission spectra relative to that of bulk semiconductors [1].

Tatykaev et al., have successfully prepared nanoparticles of silver chloride dispersed within a soluble salt matrix by means of powders grinding in a mortar  $(z = z2 = 3:64)$  and in rotating laboratory mill  $(z = z1 = 7:22)$  via reaction NH<sub>4</sub>Cl + A  $gNO<sub>3</sub> + zNH<sub>4</sub>NO<sub>3</sub> = (z + 1)NH<sub>4</sub> NO<sub>3</sub> + AgCl.$  The synthesis of silver chloride by soft mechanochemical reaction, revealed by XRD,and that the sizes (L) of synthesized particles are  $L(z2) = 132$  nm,  $L(z1) = 151$  nm. The SEM examination of the reaction products suggests the formation of silver chloride particles with wide ranges of size distribution, commonly less than 200 nm. Thermal analysis allowed identification of the optimal temperature range to yield nanoparticles of a desired phase [37].

#### 11.10 Mechanochemistry in the preparation of nanosized pharmaceutical drugs

During KOFST project at the College of Pharmacy of Pusan National University, Baláž et al. clarified that "Mechanochemical preparation of nano – sized pharmaceutical drugs for the application in cancer therapy". The obtained results are good starting point for the future research activities. Arsenic sulphides belong to the very progressive compounds in pharmaceutical applications. In medicine, there is a great need for an effective treatment of cancer, particularly haematological cancer including leukaemia and lymphomas. Further, there is a great need for orally active anticancer agents. Drugs with arsenic sulphides as active components are good candidates for such demands [15].

#### 11.11 Conclusions

Mechanochemistry has made significant progress during the recent decade. The accessible hypothetical, experimental and applied results are outlined in this chapter. The distinguished hallmarks of mechanochemistry include: impacting reactivity by creating interphases (particularly in composite and multi/phase systems), abandons in solids and by presence of relaxation phenomena, making great crystalized cores of nanoparticles with disordered near surface shell regions, performing simple, dry, time-convenient one-step syntheses, preparing nanomaterials with properties set in advance, scaling up to industrial production, and empowering work under environmentally friendly and essentially waste-free conditions.

In general, there is still a long way to go in mechanochemistry. Mostly the clarification of the mechanism of the reactions and industrial application of mechanochemistry in nanotechnology are the issues. However, there is no doubt that mechanochemistry should be incorporated in textbooks of not only solid-state, but additionally other branches of chemistry. A similar idea was proposed by Wilhelm Ostwald (The Nobel Prize in Chemistry 1909) long time ago.

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### Handbook of **Greener Synthesis of Nanomaterials** and Compounds **VOLUME 1**

## **Fundamental Principles and Methods**

## Edited by Boris I. Kharisov and Oxana V. Kharissova

Modern techniques to produce nanoparticles, nanomaterials, and nanocomposites are based on approaches that frequently involve high costs, inefficiencies, and negative environmental impacts. As such, there has been a real drive to develop and apply approaches that are more efficient and benign. The Handbook of Greener Synthesis of Nanomaterials and Compounds provides a comprehensive review of developments in this field, combining foundational green and nano-chemistry with the key information researchers need to assess, select and apply the most appropriate green synthesis approaches to their own work.

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