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Recent developments in liquid-phase synthesis and applications of nanomagnesia

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Abstract. Recent developments in the synthesis of nanomagnesia of controlled sizes and shapes that are suitable for various applications are reviewed. Two main methods, based on liquid-phase synthesis, i.e., chemical methods and bio-based methods, are used to synthesize nanomagnesia. Conventionally, nanomagnesia was synthesized by chemical methods such as co-precipitation, sol-gel, combustion method, and so on using different chemical agents and stabilizers which later on become responsible for several biological risks because of the toxicity of used chemicals. Bio-based protocols are growing as another environmental friend method for the synthesis of various nanostructures especially nanomagnesia using biomass, plant extracts, alga, and fungi as a source of precursor material. The ideal method should offer better control of textural properties of nanostructures and decrease the necessity for purification of the synthesized nanoproducts, which sequentially removes the use of large amounts of chemicals and organic solvents and manipulation of products that are unsafe to the environment. Finally, the broad applicability of nanomagnesia in diverse areas is presented. Employment of nanomagnesia reported in several laboratory and industrial fields are valued from the standpoint of the significance of these issues for technological requests, as described in the literature. Nanomagnesia has various applications such as antimicrobial performance, removing pollutants, batteries application, and catalysis.

Keywords: agricultural waste; biological method; magnesium oxide; nanomaterials; wet chemical method

1. Introduction

Due to the unique physicochemical properties of metal oxides nanoparticles, these materials are among the broadest synthesized and used nanomaterials (Diwald and Berger 2022). With the reducing size of particles, the nanoparticles usually demonstrate different properties from the bulk material such as superparamagnetic behavior, catalysis, semiconductivity, and optical properties (Safavi *et al*. 2019, Letti *et al*. 2019, Supraja *et al*. 2019) which have been investigated as certain properties may adjust when the particle size changed in the nanometer scale. This dependence of the essential properties to the particle size is entitled “size effect” in a strict sense (Li *et al*. 2019, Keller *et al*. 2021, Szałaj *et al*. 2019). Due to the ionic property of metal oxides, their chemical and physical characteristics are strongly affected by defects. Among the defects possible, the high percentage of surface atoms (especially edge and vertex atoms) in nanoparticles most drastically affects their properties (Jupille and Thornton 2015). On the other hand, the synthesis of the small particles is assisted due to the general chemical stability of metal oxides. The ionic nature of some metal oxides, particularly magnesia results in the formation of many stable defect sites, containing edges, vertexes, and anion/cation vacancies (Schwab *et al*. 2021, Sterrer *et al*. 2000).

Among the several metal oxides, magnesia is a technologically vital material due to its simple (cubic) crystal structure and absence of d orbital electrons, which persist in way of considering chemical and physical properties (Shand 2006). Bulk magnesia exhibits insulating behavior (optical bandgap = 7.6 eV) and a dielectric constant of 10. These values depend upon the morphology of the nanostructure (Singh *et al*. 2020). Magnesia nanoparticles have found various applications that are considered as especially encouraging within the wide field of Nanoscience, e.g., photo and electroluminescence, spintronics, nanocoatings, insecticides, catalysis, adsorbents, antibacterial and antifungal activities, anti-cancer therapy or biomedical applications (Fernandes *et al*. 2020). The broad range of structures and characteristics, environmentally friendly, and availability of magnesia nanostructures caused the synthesis of this type of materials to be the main goal in various sciences. Accordingly, a great variety of synthetic protocols have been presented for the size and shape-selective synthesis of magnesia nanostructures (Singh *et al*. 2020).

In the current review, mainly, we have highlighted several approaches that are studied to synthesize nano-magnesia. Among these methods, sol-gel and co-precipitation method is frequently considered while bio-based approaches are advantageous and have been given more attention today as a bio-based economy and use of renewable raw material has been developed as eco-friendly

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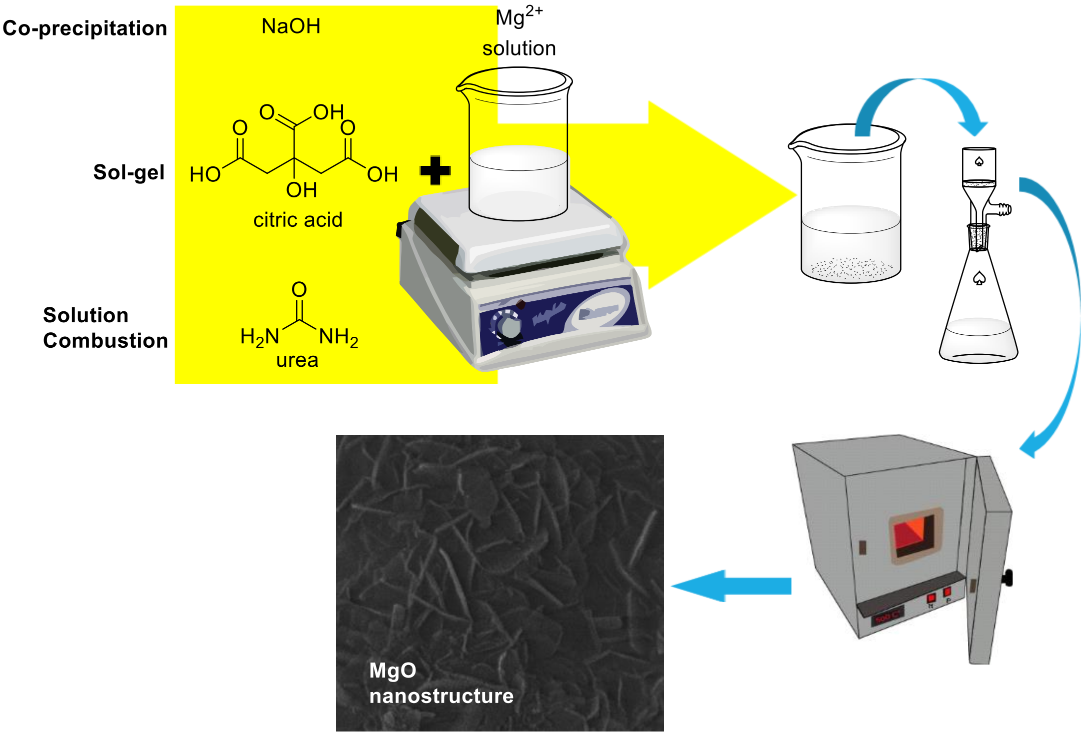


Fig. 1 Wet chemical synthesis of MgO nanostructures

preferences to tackle environmental problems. Our second goal in this review is the investigation of various applications of nanomagnesia mainly the antimicrobial performance, removal of pollutants, batteries application, and catalysis. These unique characteristics of nanomagnesia will lead scientists and engineers to further explore its uses in several areas.

2. Wet chemical methods

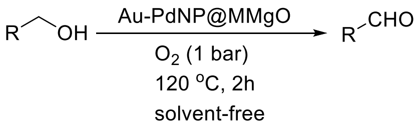
Wet chemical approach, known as solution processing, illustrates a convenient, adaptable, and efficient method for the preparation of materials with outstanding control of their chemical and physical, and textural properties. It will be of special significance when synthesizing nanostructures, which need exact control and tunability of their properties, especially size, shape, and surface chemistry that is conducive to attaining the requested effectiveness (Baig *et al*. 2021). Outstanding improvements in the wet chemical method for the synthesis of nanoparticles have been attained in the last decades. however, this method still needs development compared with other processes such as sputtering, chemical vapor deposition, atomic layer deposition, and plasma which despite being normally high-cost, are believed more efficient in the synthesis of superior nanomaterials, mainly for nanoelectronic applications (Ijaz *et al*. 2020).

Nevertheless, solution processing-based protocols have conventionally been considered as an unclean process, since it frequently does not offer appropriate quality and pureness in comparison with vacuum-based deposition methods (Gaspera *et al*. 2021). This impression about wet chemical method is gradually being modified because outstanding successes have been described in the preparation and use of nanomaterials by wet chemistry approach. Numerous papers have been published proving the effectiveness of wet chemical approach comparable with those achieved with vacuum-based depositions. Co-precipitation method, sol-gel method, and Solution Combustion method are the main methods that are reported in the synthesis of MgO nanostructures (Fig. 1).

2.1 Co-precipitation

A lot of the classic methods for the synthesis of metal or metal oxide nanoparticles were attained by the co-precipitation of partially soluble salts from aqueous solutions followed by calcination to produce oxides (Bhagyaraj *et al*. 2018). Co-precipitation processes include the simultaneous steps of nucleation, nucleus growth, Ostwald ripening, and/or agglomeration. Processes for the preparation of oxides can usually be divided into two types: those that resulted in an oxide straightly and those that produce precipitate (usually hydroxide) that must be calcined or annealed to form nano-metal oxide. In either case, monodispersed oxide nanoparticles may require a stabilizer to avoid aggregation of the particles. In methods where calcination of the precipitate is needed, some aggregation will be inescapable (Cushing *et al*. 2004). Any process parameters affecting the reaction progress, such as the pH of the precipitation mixture, the addition rate of precipitating agent (hydroxides or carbonates), temperature, and stirring rate, should be investigated to control the size, shape, and particle size distribution of the product.

The growth of metal oxide in the water includes



R	cat. (mol%)	coversion (%)	aldehyde selectivity (%)	TOF (mol/h.mol)
Ph	0.356	21.4	96.3	1501
n-C_7H_{15}	0.429	1.36	100	78.9

Scheme 1 Au-PdNP@MMgO catalyzed aerobic oxidation of alcohols

hydrolysis and condensation processes and can be studied as the three fundamental steps of initiation, propagation, and termination. Initiation: A hydroxylated metal complex is the precursor of the condensation process. Hydroxylation of the metal cation can be started by an acid-base reaction. In the water, the pH of the solution plays a vital role in tuning the hydrolysis ratio of metal precursors. Propagation: A condensation process between hydroxylated metal complexes starts to proceed and results in oxygenated bridges between cations. The process may take place by dissociative substitution, associative substitution, or direct displacement. It is assumed that the pH of the solution will be extremely dependent on the dynamics of the reaction mixture. Termination: The condensation process will be terminated if the hydroxide ligand of the hydroxylated metal complexes drops its nucleophilic character speaking of polycation. Hydroxylated metal complexes are bridged by OH ligands in the condensation process and initially form $M(OH)_2$ that can be unstable and automatically dehydrated by internal oxolation to produce the hydrated oxide. Nucleation step is based on the condensation process of the hydroxylated metal complexes as described above. Growth of the nuclei is through the addition of the hydroxylated metal complexes on the current metal oxide nuclei and continues until the primary oxide nanoparticles are produced.

Many nanomagnesia samples are synthesized by calcination of hydroxide co-precipitation products. Alshammari *et al.* (2017) prepared mesoporous magnesia (MMgO) with 80 m^2/g BET surface area and 8 nm average pore diameter by precipitating $MgCl_2$ solution with NH_4OH at 100 °C followed by aging, drying, and calcining at 400 °C. This magnesium oxide nanostructure was demonstrated to be sufficient for absorbing and stabilizing gold-palladium nanoparticles (Au-PdNP) by treatment with $PdCl_2$ and $HAuCl_4$ followed by reducing with $NaBH_4$ to form catalyst Au-PdNP@MMgO. This catalyst displayed high selectivity in the aerobic oxidation of benzyl alcohol and 1-octanol to corresponding aldehyde (Scheme 1).

In a similar way, 20 nm magnesia nanoparticles were produced by the co-precipitation method using dropwise addition of sodium hydroxide solution to Magnesium sulfate solution followed by calcination at 500 °C (Dawood *et al.* 2018). This heterogeneous catalyst was then employed in the transesterification of triacylglycerols from yellow oleander seeds oil to methyl esters with maximum conversion (93.1%).

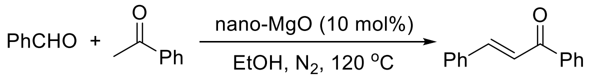
The co-precipitation of Mg^{2+} as carbonates followed by calcination and decomposition is a classic approach for the synthesis of crystalline magnesia nanostructures. As an example, magnesia nanohexagonal sheets were precipitated by the addition of Na_2CO_3 to an aqueous solution of $Mg(NO_3)_2$ followed by calcination (Azzam *et al.* 2018). Calcining at 600 °C yielded MgO nanohexagon with a diameter in the range of 150-300 nm and pore size ranging from 1.9 nm to 65.6 nm. Antibacterial activity of synthesized nanohexagonal sheets against Gram-positive (Staphylococcus aureus) and -negative (*Escherichia coli* and *Pseudomonas aeruginosa*) bacteria was improved at proposed optimum conditions. The reactions with cell wall proteins resulted in decreased permeability, however high release rates of ROS from MgO nanohexagon made damage to DNA and cellular components and finally caused bacteria death. As a result, this protocol may offer a progressive approach for wastewater cleanliness that can be used for watering.

Imani and Safaei (2019) prepared magnesia nanoparticles of 21 nm in size by co-precipitation of $Mg(NO_3)_2$, NaOH, and their antibacterial performance against *Escherichia coli* and *Staphylococcus aureus* was investigated by colony-forming unit and disk diffusion methods. Authors have reported that nanoparticles can obviously decrease the number of bacteria and can be used as a suitable alternative to frequently used antibiotic compounds to prevent drug resistance among pathogens.

The antibacterial performance of 27 nm-sized similar nanoparticles, synthesized by the same method and precursors, against two representative bacteria *Bacillus sp* and *Escherichia coli* (Maji *et al.* 2020). The observations showed that $6 \mu g.ml^{-1}$ dose is enough for the complete inhibition of Bacillus sp. whereas it is $7.5 \mu g.ml^{-1}$ for E. coli. The oxygen vacancy in nano-sized MgO is adsorbed at the bacterial cell wall and the membrane and forms superoxide radicals through Interaction by the cell wall and kills bacteria.

The nanocomposite of carbon nanotube, graphite, and MgO nanoparticles synthesized by $Mg(NO_3)_2$ and NaOH was prepared by Asgari *et al.* (2020), and its catalytic performance was studied in the ozonation (degradation) of diazinon in aqueous solutions. Very recently, El-Shamy (2021) described the preparation of a novel nanocomposite based on carbon dots and MgO nanoparticles and its application in Schottky device-based- H_2S gas sensing.

In materials science and nanotechnology, it is very problematic to control particle size and morphology. A clever solution for this problem provided by nanochemists is a variant of the templating method known as the surfactant-mediated pathway (Phan and Nguyen 2017).



nano-MgO morphology	surface area (m^2/g)	basicity (mmol/g)	time (h)	yield (%)
Nanoflakes	48.2	0.73	12	76
Interconnected nanocapsules	61.2	0.86	12	83
Hexagonal nanostructures	74.3	1.68	12	95
Irregulars nano particles	49.6	1.32	12	72
Nanocapsules	56.2	0.92	12	81
bulk MgO	_	_	24	21

Scheme 2 Catalytic activity of nano-magnesia materials in chalcone synthesis

Noteworthy, in the case of metal oxide nanoparticles that need to be calcined in the final stage of synthesis, the surfactant will be decomposed (so-called sacrificial template). De Silva *et al.* (2017a) prepared 151-200 nm MgO nanoparticles by the addition of methyl methacrylate monomer and $MgCl_2$ solution to a solution of sodium carbonate and sodium persulfate. Simultaneous polymerization and co-precipitation led to magnesium carbonate/poly (methyl methacrylate) composite that was calcined at 600 °C to produce MgO nanoparticles. The authors continued their studies on the fabrication of chitosan/MgO nanocomposite films by the solution casting method in various nanoparticle concentrations. The addition of magnesia nanoparticles into chitosan led to improvement in tensile stress, thermal stability, and flame retardancy. This nanocomposite film with improved characteristics can probably be employed as a packaging material to save the quality of food products.

The same group continued their studies on the preparation of 45 nm-sized poly(acrylic acid)-templated MgO nanoparticles by $MgCl_2$, NaOH as a co-precipitator agent, acrylic acid as a monomer, and $K_2S_2O_8$ as an initiator (De Silva *et al.* 2017b). MgO nanoparticles were used in the preparation of alginate-based nanocomposite scaffolds by the electrospinning method. Due to the highest tensile strength and elastic modulus of alginate/MgO nanocomposite, the authors suggested that this material is a suitable candidate for extracellular matrix in tissue engineering applications.

Siriwardane *et al.* in 2017 and Myneni *et al.* in 2019 prepared MgO nanoparticles with sizes 50 nm and 20 nm, respectively, by $Mg(NO_3)_2.6H_2O$ and hydroxide in the presence of PVP. Activated carbon-based nanocomposites of these nanoparticles prepared by the impregnation method have been employed in H_2S (Induni *et al.* 2017) and methylene blue (Myneni *et al.* 2019) removal.

In 2019, Pourrahim *et al.* used solid waste from the ductile iron industry, with high content of MgO, for the synthesis of nanoporous magnesia. Different surfactant mixtures, including sodium dodecyl sulfate (SDS) cetyltrimethylammonium bromide (CTAB), and Triton X-100 (TX100) were mixed with the magnesium nitrate solution followed by precipitation with ammonium hydroxide and calcination. The porosimetry data showed that the mixture of SDS and TX100, 1:1 contribute effectively to the synthesis of nanoporous magnesia with the appropriate average pore size, 16 nm. Magnesia nanostructure was found to be efficient material for the removal of reactive dye. Also, various nanostructured MgO can be prepared by treatment of ammonium hydroxide and Mg(II) solution in the absence of surfactants (Li *et al.* 2022).

In recent years, ionic liquids (ILs) were progressively employed and studied as a solvent, template, and stabilizing agent for the synthesis of nanoparticles (Wegner and Janiak, 2017). Low interface energy in ILs makes them promising solvents for stabilizing nanoparticles. ILs contain anions and cations, which can act as electrostatic stabilizers. The IL ions or IL self-aggregation are adsorbed to the nanoparticle surface by electrostatic forces. Also, imidazolium-based IL coordinated with nanoparticles via π–π interaction and help as a stabilizing agent to prevent the aggregations of nanoparticles (Vasanthakumar *et al.* 2018). On the other hand 1,3-dialkyl imidazolium-based, ILs effectively absorb microwave radiations and thus are used as solvents and co-solvents in microwaves-assisted synthesis (Hoffmann *et al.* 2003).

In this regard, Jadhav *et al.* (2016) described the use of *N*-methyl imidazolium and 3-methyl pyridinium halides in the microwave-assisted synthesis of various MgO nanostructures by co-precipitation of magnesium acetate and sodium hydroxide. Several forms of nanomagnesia such as nanoflakes, interconnected nanoparticles, hexagonal nanoparticles, and nanocapsules were successfully synthesized by this method with specific surface area, pore volume, and average pore diameter in the range of 48-74 m^2/g, 0.49-0.91 cm^3/g, and 24-36 nm, respectively. Interestingly, based on temperature-programmed desorption of CO_2, the basicity of these nanomaterials is in the range from 0.73 to 1.68 mmol/g. To recognize the performance of the basicity of the magnesia nanomaterials, all materials were tested as a catalyst in the Claisen-Schmidt process to synthesize chalcone (Jadhav *et al*. 2018, Scheme 2). In a comparative study, the hexagonal nanostructure that has the largest surface area and basicity showed the highest yield in chalcone synthesis.

In addition to ionic liquids, other green solvents such as ethanol, acetic acid, and ethylene glycol have been used in the co-precipitation method for the synthesis of MgO nanoparticles with a size smaller than 50 nm (Prado *et al*. 2020).

2.2 Sol-gel

Sol-gel technique is classified among well-known synthetic methods to produce desired metal oxide micro and nanostructures (Parashar *et al*. 2020). Technically, it can be better defined more widely as covering the synthesis of metal oxide materials from solution-state precursors. This technique has superior control over the textural properties and chemical stability of the nanomaterials. Principally, the sol-gel process can be explained by five basic steps; hydrolysis, polycondensation, aging, drying, and calcination (Danks *et al*. 2016). Citric acid is one of the most frequently used chemicals in the sol-gel process known as the citrate sol-gel method. In a typical way, a metal salt solution is mixed with this readily available and cheap triprotic acid followed by heating to make a viscous solution or gel.

In 2017, Hikku *et al*. described the synthesis of 19 nm-sized porous magnesia nanoparticles by dropwise addition of aqueous citric acid solution to magnesium nitrate solution followed by adjusting the pH of this mixture to 10 and boiling. The calcination of obtained powder at 800 °C for 1h resulted in 19 nm-sized MgO nanoparticles that display antibacterial activity towards *Escherichia coli*, and *Bacillus badius*. Also, these nanoparticles possess the self-cleaning capability and removal of methyl violet dye during exposure to sunlight.

Synthesis of magnesia nanostructures can be also carried out through the non-aqueous sol-gel method. In the non-aqueous sol-gel protocol, the process carries out in an organic solvent under the exclusion of H_2O (Niederberger and Pinna 2009). Selvi and Das (2016) prepared magnesium oxide nanoparticles by sol-gel reaction between KOH and $MgCl_2$ in toluene under reflux conditions followed by washing and calcination. Tests carried out in batch mode showed that the degradation of cefdinir by magnesia nanoparticles coated on Candida sp. SMN04 was more competent than the individual systems. Also, the catalytic activity of these nanoparticles was tested in the ozone degradation of phenol by a radical mechanism (Wang *et al*. 2017). Hydroxyl radicals were produced on the surface of magnesia and consequently, nanoparticles were able to catalyze ozone into hydroxyl radicals and phenol oxidation.

Further modification of the non-aqueous sol-gel method led to the development of the synthesis of magnesia nanoparticles in ethanol using a complexing agent such as benzene-1,2-dicarboxylic acid (Deepa and Rajendran 2018) and oxalic acid (Durgalakshmi *et al*. 2019) in the presence of Magnesium acetate tetrahydrate.

Interestingly, the addition of water to an ethanolic solution of magnesium methoxide, $Mg(OCH_3)_2$ followed by aging and calcination resulted in 8 to 20 nm-sized magnesia nanoparticles (Castillo *et al*. 2019). Due to the antibacterial activity of resulting nanoparticles against both Gram-negative and Gram-positive bacteria, these nanomaterials were used to protect several 18th-century papers from microbial colonization. TEM images have proved that cell membrane leakage and cell death resulted from oxidative stress.

Prado *et al*. (2020) prepared mesoporous magnesium oxide with 171 m^2/g BET surface area and 26 nm average pore diameter via a sol-gel method where $Mg(OAc)_2$ was added slowly to aqueous CTAB solution at pH=11.5 followed by aging, washing, and calcination at 500 °C. Along this line, it was shown that MgO nanoparticles can be synthesized using low-cost and naturally existing bittern, and dolomite (Gunathilake *et al*. 2020). Bittern solution (as a first Mg source) was treated with HNO_3 and then barium chloride to remove sulfate ions. CTAB and dolomite (secondary Mg source) were then slowly added to the reaction. The produced gel was separated dried and calcined at 450 °C to obtain final plate-like MgO nanoparticles. Graphene oxide-supported MgO nanoparticles were examined for carbon dioxide sorption at several temperatures. The Immobilization of MgO nanoparticles on graphene oxide enhances the number of hydroxyl groups and is thus accountable for higher carbon dioxide chemisorption via hydrogen carbonate and bidentate carbonate formation.

2.3 Combustion synthesis

Solution Combustion Synthesis (SCS) is an energy and time-effectiveness, low-cost, and simple process, which enables an efficient synthesis of metal oxide nanoparticles (Varma *et al*. 2016). This method includes a self-sustained reaction in a homogeneous solution of various oxidizers (e.g., nitrate salts) and fuels (such as citric acid, glycine, and urea). Essentially, SCS contains three key stages, 1) mixture formation (solution of metal salt and organic fuel in water), 2) gel network formation (complexation between metal ions and fuel) 3) combustion of the gel. Due to the essential rapid and uncontrollable combustion procedure, the common problem encountered in combustion synthesis is the controllability over phases, size, and textural properties of the products (Deganello and Tyagi 2018). Large quantities of gaseous byproducts generated in SCS make a considerable expansion of the solid product which leads to porosity or dispersed particles.

Madzokerea and Karthigeyan (2017) reported on the synthesis of 12-17 nm-sized agglomerated MgO nano-particles by Magnesium nitrate as an oxidizer and glycine as the fuel. This nanomaterial could efficiently remove Cu(II) ions from copper (II) chloride solution. A similar experimental process was used for the magnesium nitrate-urea solution and yielded MgO with nanoparticle morphology. Owing to the high sorption capacity of produced MgO nanoparticles, they can successfully remove humic acid, fluoride (Oladoja *et al*. 2017), and methyl orange (Yan *et al*. 2020). Also, SCS-based nano MgO filler/polymer nanocomposite can be used as a thermally

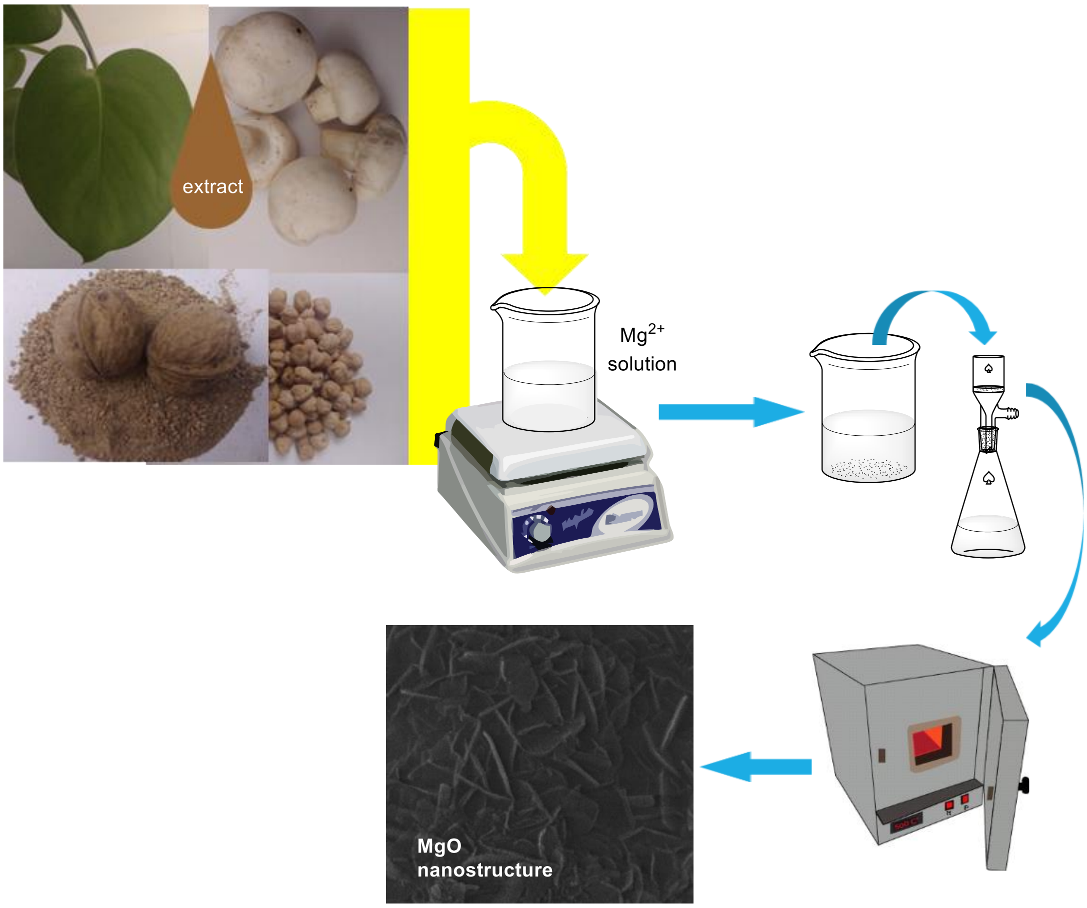


Fig. 2 biological synthesis of MgO nanostructures

stable electrolyte for lithium-ion batteries (Masoud *et al*. 2018). Ma *et al*. (2018) used magnesium chloride and acetate in combination with hexamethylenetetramine (HMTA) as fuel to prepare nanoflowers and nanoparticles. Also, MgO nanowires were attained by SCS using manganese acetate-HMTA mixtures, initiated in heated furnaces at 450 ◦C. The adsorption capacity of magnesium oxide nanowires for removing arsenate and phosphate was tested by the authors.

3. Biosynthetic methods

Green chemistry is a modern field of laboratory and industrial chemistry that deals with the design of products and synthetic methods that decrease or eliminate the use and production of hazardous chemicals. Due to the harmful toxic contaminants which could be found in the chemical protocols for the synthesis of nanomaterials, bio-based synthesis of nanostructures using plant and animal parts has progressively attracted some attention (Omran 2020). These methods (Fig. 2) usually apply microorganisms including bacteria, fungi, and algae, and plants biomass or extracts (Malarkodi *et al*. 2013, Sur 2014, Baláž *et al*. 2019).

In this class of methods, biological compounds cover the surface of nanoparticles during green synthesis (Javed *et al*. 2020). Consequently, this auxiliary coat improves the biological characters of produced nanoparticles compared to the ones synthesized by chemical methods. Also, the presence of a biological layer avoids the contamination of nanoparticles with harmful by-products. However, one of the main reasons why biosynthetic methods have stayed at laboratory-scale, up to now, is that the method includes a set of interconnected factors (pH, the biological reducing/precipitation agent composition and concentration, temperature, metal salt/precursor concentration, rate of agitation, process time). These affect the properties of the produced nanoparticles. So, the optimization of a bio-based technique is a challenging goal that needs more investigation. Moreover, the participation of organisms makes the procedures hard to standardize and repeat on a larger scale, as the internal media of organisms varies from one species to another and between individuals from the same species (Miu and Dinischiotu 2022).

3.1 Plant extract-mediated method

A growing area in green nanoscience, the so-called phytonanotechnology, is the green synthesis of nanostructures by the extracts of diverse plant tissues such as leaves, fruit, roots, seeds, peels, and flowers (Bao *et al*. 2021). Due to the uncertainty of precise phytochemical components in extract, the true mechanism of extract-mediated synthesis of nanoparticles is yet unclear to some extent. But these extracts play well the role of reductants, capping agents, and stabilizers because of the existence of

Table 1 Recent papers on leaf extract mediated synthesis of MgO nanoparticles and their applications

Plant name	Nanoparticle Size (nm)	Application	References
Bauhinia purpurea	11	antimicrobial applications	Das *et al*. 2018
Crantz	36.7	---	Essien *et al*. 2020
Sesbania bispinosa	80–90	effect on growth parameters and chlorophyll content in Long Bean Plant	Elakkiya *et al*. 2020
Achyranthes aspera	7-13[a]	antimicrobial applications	Pavithra *et al*. 2020
Amaranthus tricolor, *Amaranthus blitum* and *Andrographis paniculata*	20-44	cytotoxicity	Jeevanandam *et al*. 2020b
Moringa oleifera	13-35	capacitor applications	Venkatachalam *et al*. 2021
Green tea	80	antimicrobial applications	Khan *et al*. 2021
Moringa Oleifera	40-70	antimicrobial applications	Amrulloh *et al*. 2021

[a] average crystalline size of MgO nanosheets

proteins containing amino groups and alkaloids, flavones, and anthracenes with carbonyl, hydroxyl, and ether groups (Singh *et al*. 2018). Due to the sensitivity of phytochemicals to temperature, the drying step in plant extraction is very important. Freeze drying is described to be the best technique to dry. Also, the dried part will be ground to increase the surface area. The extraction process is done using suitable polar or nonpolar solvents. In addition to water, organic solvents such as ethanol or methanol frequently are used for the extraction. (Thomas *et al*. 2020).

Ghashang *et al*. (2016) utilized carboxylic acid-rich extract of *Rosmarinus officinalis* leaves and $MgCl_2$ which yielded 73 nm-sized MgO nanoparticles with sizes between 20–140 nm. These nanoparticles are applied as catalysts for the synthesis of thiochromeno[4,3-b]pyran and thiopyrano [4,3-b]pyran derivatives.

In 2017 magnesia nanoparticles were synthesized by Jeevanandam *et al*. using three various leaf extracts (*Amaranthus tricolor*, *Amaranthus blitium*, and *Andrographis paniculata*) and the effects of reaction factors including extract concentration, magnesium salt loading, temperature, and process time on the size and shape the MgO nanoparticles were studied. FTIR investigations exposed that the degradation of functional groups of extract components is accountable for the production of magnesia nanoparticles. It is interesting that the functional group transformations are different, by varying metal salt. According to the TEM images, this method can be used for tuning the size of MgO nanoparticles in the range of 18 to 80 nm (frequently spherical and hexagonal) where leaf extract compositions play an important role in size controlling. Jeevanandam *et al*. (2018) continued their studies on the synthesis of 6-8 nm width-sized MgO nanorods using *Eucalyptus globulus* aqueous leaf extract. Due to the large surface area, nanorods demonstrate the noteworthy biological activity by comparison with spherical nanoparticles. Based on FT-IR spectroscopy, eucalyptol, triterpenoids, polyphenols, and flavonoids in extracts and enol to keto transformation were proved to be accountable for nanoparticle formation. Authors have continued their investigations on the synthesis of magnesia nanoparticle size of 48 nm using *Calotropis gigantea* leaves extracts (Hii *et al*. 2018). Nanoencapsulation of magnesia nanoparticles by Eudragit L100 (Poly(methacrylic acid-co-methyl methacrylate) was found to synthesize 53.37nm-sized particles at higher stability. also indicated that Eudragit L-encapsulated magnesia nanoparticles had higher drug loading.

Antibacterial activity of *Pisidium guvajava* and *Aloe vera* leaf extract-mediated MgO nanoparticles evaluated against E. coli and S. aureus using the disc diffusion method by Umaralikhan and Jaffar in 2018. The antibacterial activities of magnesia nanoparticles were relatively similar for both bacterial species. Also, Aloe Vera-mediated MgO nanoparticles were used for improvement in seedling growth of Vigna Radiata and Cajanus Cajan (Rani *et al*. 2020). The seed germination data showed that the existence of these MgO particles enhanced the seed germination rate and seedling altitude.

In 2018, Kaur *et al*. published a paper in which they described the green synthesis of MgO nanoparticles using *aloe vera* extract as well as the solution-combustion technique. The SEM and TEM images revealed the formation of the 25 nm-sized fibrous-shaped nanoparticles for the combustion method and 15 nm-sized spherical nanoparticles for the biological method. These nanoparticles were tested in sensing liquefied petroleum gas in 400–1000 ppm concentration at temperatures. The relationship between synthesis method and sensitivity towards LPG was investigated and it was found that biosynthesized MgO is recommended for detecting trace-level gases at room temperature to 300 ^{o}C (Thirupathi *et al*. 2018).

Solanum trilobatum leaf extract was applied to synthesize MgO nanoparticles with an average size of 30 nm (Narendhran *et al*. 2019). It was reported that mixing *S. trilobatum* leaf extract and $Mg(NO_3)_2$ solution followed by calcination of precipitate resulted in MgO nanoparticles. Also, it shows significant in vitro antibacterial activity against *E. coli* and *B. subtilis*.

Hexagonal 43 nm-sized magnesia nanoparticles were prepared by *Amaranthus tricolor* leaf extract and the effect of pH (3-11) on their shape and morphology was studied (Jeevanandam *et al*. 2020a). The protonation of magnesia

Table 1 Recent papers on leaf extract mediated synthesis of MgO nanoparticles and their applications

Plant name	Nanoparticle Size (nm)	Application	References
Swertia Chirayaita	whole plant	<20	antimicrobial applications
Clitoria ternatea	whole plant	50-400	in vitro antioxidant activity
Artemisia abrotanum	whole plant	10	removal of dye
Saussurea costus	root	30	cytotoxicity and antimicrobial applications
Pterocarpus marsupium	heartwood	<20	biomedical applications
Opuntia monacantha	whole plant	12-18 [a]	antimicrobial applications

[a] Thicknessof MgO nanosheets

nanoparticles in an acidic medium was proposed to act as a capping agent and stabilizer to adjust the morphology of the nano-magnesia from spherical to hexagonal.

In a different study, visible light-mediated synthesis of magnesia nanoparticles by *Amaranthus tricolor* leaf extracts without the addition of any chemical precipitator, has been investigated (Siaw *et al*. 2020). In this report, researchers looked at the effect of the reaction time, the precursor concentration, and light intensity on the size of MgO nanoparticles. Based on The proposed mechanism for this process absorption of light energy by phytochemicals such as carotenoids and xanthophyll resulted in the reduction of their long carbon chains into smaller compounds. The oxide ions from the reduced phytochemicals can be bonded with Mg^{2+} ions to form magnesia. Today, scientists show more interest in leaf extract-mediated synthesis of magnesia nanoparticles as described in Table 1.

In addition to leaves, extracts from other parts of plants can also be used to synthesize magnesium oxide nanoparticles. Nano rice-like MgO with an average width of 50~70 nm (Maruthai *et al*. 2018) and nano cauliflower-like MgO (Jayapriya *et al*. 2020) were synthesized by *Musa paradisiaca* bract extract. The catalytic efficiency of these MgO nanostructures with Ag and ZnO has been tested in the removal of methyl orange, methylene blue, and O-nitrophenol.

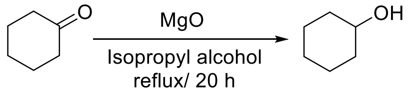
Rosemary Floral extract-mediated MgO nano-flowers considerably prevented bacterial growth, biofilm formation, and motility of Xanthomonas oryzae pv. oryzae is one of the most common agents of disease in rice (Abdallah *et al*. 2019). Verma *et al*. (2020) have performed a comparative study using *Calotropis gigantean* floral extract-mediated MgO nanoparticles (F-MgONPs) and commercial MgO nano-particles (C-MgONPs) in cellular toxicity analysis. The results revealed the molecular nanotoxicity of C-MgONPs and supported the usage of F-MgONPs as an encouraging alternative to C-MgONPs for higher bio-compatibility in both clinical as well as environmental fields. The authors reported the considerable toxicity of C-MgONPs in the water environment as a result of time-consuming accumulation, and their widespread use posing a serious risk to the ecosystem. Whereas Biocompatible F-MgONPs in optimized concentration can be a powerful solution to the threat. Taking into account the concerns related to toxicity and ecological pollution the authors proposed the adoption and application of the green approach in nanoparticle synthesis. Table 2 provides recent reports on the synthesis of magnesia nanoparticles by using extracts of various parts of plants and their applications in different areas.

Also, it should be noted that in addition to plants, the white mushroom aqueous extract has been used too for the synthesis of 15-20 nm-sized MgO nanoparticles (Jhansi *et al*. 2017). The ability of algae to adsorb and reduce transition metal ions makes them one of the best candidates for the biological synthesis of nanoparticles (Oscar *et al*. 2016). Furthermore, algae are partly easy to manipulate in addition to numerous other benefits such as synthesis at low temperatures with higher energy saving, less toxicity, and ecological risk. Zaky *et al*. (2018) have reported that phlorotannins present in the brown seaweed (Sargassum-muticum) extract are responsible for the synthesis of 17–30 nm-sized MgO nanoparticles from Magnesium acetate solution. Anand *et al*. (2020) investigated the synthesis of 12 nm-sized magnesia nanoparticles using an extract of the marine brown alga, *Turbinaria ornata*, and the effect of nanoparticles on seed germination of *Vigna radiata*. Treatment with MgO nanoparticles indicated a considerably high germination rate and seedling growth in comparison with conventional hydropriming. Also, MgO nanoparticles were prepared by co-precipitation method using NaOH and extract of a marine macroalga (red seaweed) *Kappaphycus alvarezii* (Pachiyappan *et al*. 2020).

3.2 Biomass-mediated method

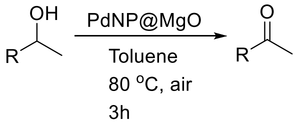
Biomass has distinctive properties which offer unique advantages, containing easy access and availability, renewability, biocompatibility, and biodegradability. Various biomass Such as fungi, plants, and agricultural wastes have been used to produce nanostructures for various applications (Omran 2020). Fungi are generally presented as a favored and appropriate green nano-factory for the preparation of nanoparticles (Guilger-Casagrande and Lima 2019). The fungal system-based methods in the synthesis of nanomaterials are commonly known as psychosynthesis. In this line, some fungal species have been studied to synthesize MgO nanoparticles.

Saied and co-workers (2021) prepared 8.0-38.0 nm-sized magnesia nanoparticles by treatment of $Mg(NO_3)_2$ and biomass filtrate of fungal strain Aspergillus terreus S1 at 25-40 ^{o}C to form $Mg(OH)_2$ followed by calcination. In a similar way, Aspergillus niger strain F1 (Fouda *et al*. 2021) and Erysiphe cichoracearum (Fathy and Mahfouz 2021)



surface area of MgO (m^2/g)	yield(%)
212	85
195	88
95	52
78	43

Scheme 3 MgO-catalyzed Meerwein-Ponndorf-Verley process



R	cat. (mol%)	yield(%)
Ph	0.25	88
n-hexyl	0.5	90

Scheme 4 PdNP@MgO catalyzed aerobic oxidation of alcohols

were used in the synthesis of MgO nanoparticles. The performance of these nanoparticles was tested in transition metal ion removal and inhibiting the growth of pathogenic microbes such as Escherichia coli.

Plant biomass can be considered as a potential candidate in the synthesis of metal oxide nanoparticles. In 2018, Kumar and co-workers prepared 20-25 nm magnesia nano-flakes by maxing Vetiver plant powder as a fuel and $Mg(NO_3)_2$ followed by calcination in a muffle furnace. Based on cyclic voltammetric data nano-flakes can be applied as electrode material. A similar technique was employed for the synthesis of MgO nanoparticles by *Cicer arietinum* powder as a fuel (Kumar *et al.* 2019).

Established on a bio-based economy, agricultural waste biomass such as rice husk, walnut shell, and fruit peel has progressively been considered as a low-cost renewable resource (as fuel) for the synthesis of nanomaterials (Zamani *et al.* 2019a). For example, Kumar *et al.* (2018) reported the synthesis of 15-20 nm-sized MgO nano-powders using banana peel powder as fuel. MgO nano-powders show photocatalytic activity under sunlight for rhodamine-B and Malachite green. It was reported that photocatalytic activity is sensitively dependent on the oxygen vacancies in MgO nanoparticles (Gurylev 2021).

Also in our group, after that, we synthesized copper (Zamani *et al.* 2018a) ceria (Zamani *et al.* 2018b), and alumina nanoparticles (Zamani *et al.* 2019b) using walnut shell, we focused on the walnut shell-assisted preparation of high surface area nanomagnesia (Zamani *et al.* 2019c). Walnut shell powder was combined with an aqueous solution of $Mg(NO_3)_2$ in different weight ratios followed by evaporation of water, and calcination. Nanostructured magnesia has BET surface area of up to 79 m^2/g. Hydrothermal treatment of these MgO nanoparticles and annealing unexpectedly enhance surface area up to 212 m^2/g. To evaluate the catalytic performance of MgO nanomaterials as solid catalysts we focused on Meerwein-Ponndorf-Verley process (Scheme 3). It is of interest that magnesium oxides with the highest surface area revealed maximum yields. Also, MgO nanoparticles were evaluated as the support of palladium nanoparticles (PdNP@MgO) in the aerobic oxidation of benzylic and aliphatic alcohols under air without the use of an exogenous base (Scheme 4). A similar technique was employed to prepare MgO nanoparticles by using sugarcane bagasse, rice husk (Guo *et al.* 2020), and olive pomace (Dakroury *et al.* 2020) as abundantly available agro-waste. Carbon dioxide and metal ion adsorption behaviors of MgO nanoparticles were investigated.

4. Conclusions

The present article emphasizes the various protocols for the synthesis of nanostructured magnesia and their potential application in antibacterial activities, dye and metal ion removal, catalysis, and Electro-Chemical Applications. An attempt is been made to classify a variety of chemical and biological protocols and raw materials such as co-precipitators, fuels, plants, and fungi under green synthesis. The analysis made here presents microscopic and textural details of types of size and morphology carried out of magnesia nanostructures. From the references cited in the article, it is understood that the method of synthesis, precipitators, fuels, surfactants, and solvents play an important role to define the improved properties of MgO nanomaterials prepared by wet chemical or biological protocols. Therefore, the aim of this article efforts to comprehend the development of chemical and biological synthesis for preparing efficient magnesia nanoparticles for future environmental, medical, and industrial applications. A wide range of amorphous nanoparticles as well as hexagonal nanoparticles are synthesized by various chemical and biological methods, but as can be seen, magnesia nanoplates and nanotablets can be synthesized mainly by biological methods, especially by using plant leaf extracts. But more studies are needed to determine the relationship between the synthesis methods of magnesium oxide nanoparticles and their morphology, as well as their physical and chemical properties and finally their application.

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