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Method of Producing Eggshell-Derived Nanoparticles for Various Applications: A Review

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Abstract: *The utilization of eggshells as an adsorbent material for diverse applications has garnered considerable attention among researchers. The incorporation of eggshell nanoparticles holds promise in enhancing reaction efficiency by virtue of their increased surface area. Consequently, this review focuses on the fabrication of materials at the nanoparticle scale in general, with a specific emphasis on eggshells. Two primary approaches for nanoparticle synthesis, namely top-down and bottom-up methods, exist. Within this review, particular attention is given to the top-down method, which encompasses mechanical milling, sputtering, and laser ablation method. Notably, despite the superior nanoparticle sizes produced through the laser ablation method, most researchers currently opt for ball milling, ultrasound, and microwave irradiation to synthesize eggshell nanoparticles. A comparative analysis of each method's efficacy in preparing eggshell nanoparticles is also presented. In conclusion, eggshell waste exhibits potential as an environmentally valuable material, amenable to various synthesis methods.*

Keywords: Nanoparticles, eggshell, titanium dioxide, nanocomposite

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1. Introduction

The issue of agricultural waste poses significant concerns within the food industry due to its substantial daily consumption and disposal rates. Extensive efforts have been made by researchers to explore alternative applications for this waste and one such discovery pertains to eggshells. Eggs are widely consumed worldwide for domestic and industrial purposes, generating solid waste in the form of eggshells, which are classified as agricultural waste. As only the inner contents of eggs are utilized, the eggshell residue, amounting to several tons per day, is disposed of in landfills, contributing to organic pollution.

Eggshells consist of approximately 2% water and 98% dry matter, comprising primarily ash and crude protein. The composition of the eggshell primarily comprises 94% calcium carbonate ($CaCO_3$), along with 1% calcium phosphate, 1% magnesium carbonate, and 4% other organic

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substances. Functionally, the eggshell serves as an external barrier, effectively impeding the infiltration of microorganisms towards the cell. Additionally, it facilitates the essential process of gas exchange (Waheed et al., 2019).

The calcium content of eggshell powder is comparable to that obtained from commercially available calcium carbonate. Due to the content of $CaCO_3$ in eggshells, researchers nowadays take some initiatives not to waste it and instead use it as a biomaterial. Some studies have shown that the eggshell can be used and utilized for something that has benefits to us like the use of eggshell waste as a catalyst employed in the synthesis of biodiesel (Faridi and Arabhosseini (2018)), hydrogen/syngas, dimethyl carbonate (DMC), bioactive compounds and wastewater treatment (Laca et al., 2017). Besides, Chen et al., (2010) proved that the use of eggshells helps reactions to reach maximum efficiency while reducing cost.

Comprising merely 9% - 12% of the total egg weight, eggshells represent a natural and porous bioceramic material. Notably, they possess the potential to function as adsorbents to treat hydrogen sulfide from wastewater (Habeeb, Yasin, and Danhassan (2014)). The researchers used calcined waste eggshells because they have the highest adsorption capacity.

Eggshells are valuable environmental and economic adsorbents owing to their abundance and ability to remove harmful and dangerous components of all adsorbents. In addition, eggshells have a porous nature that allows them to be employed as an adsorbent, and it is made up of fibrous protein with a large surface area. Jalu et al., 2021 synthesized calcium oxide (CaO) from hen eggshells for the removal of lead (Pb(II)) from aqueous solution. The removal efficiency of around 99.07% was achieved with an initial concentration of 75.46 ppm, pH 6.94, adsorbent dose 0.838g and contact time of 101.97 min.

The application of eggshells extends beyond their role as adsorbent, encompassing their potential utilization as a constitute gel for facilitating the safe transit of probiotics through the stomach. In a recent study by Sahu et al., (2023), it was demonstrated that eggshells contribute surface calcium ions for gel formation, utilize score particles as agents to reinforce gel strength and employ calcite to neutralize the acidic pH of the gastric environment.

Recently, the use of eggshell nanoparticle-sized catalysts has gained much attention due to their high surface area, which increases reaction efficiency. There are various methods to fabricate material at the nanoparticle scale. Therefore, this review focuses on the nanoparticle preparation methods and the attainment of eggshell nanoparticles specifically for photocatalytic application.

2. Nanoparticles Preparation Methods

Nanoparticles are solid colloidal particles and are a wide class of materials varying in size from 10 to 1000 nm (1 μm) (Khan et al., 2019; McNamara and Tofail, 2017). The term nanoparticle originated from the Greek word ‘nano’, which means dwarf or small. Nowadays, researchers tend to use nanosized materials, in which their size affects the efficiency of the reaction due to the high surface area. The reduction in particle size to nanosize reveals unique and enhanced properties such as particle size distribution and morphology while the surface-to-volume ratio of nanoparticles is 35%–45% times higher compared to that of larger particles or atoms (Jamkhande et al., 2019).

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Khan et al., 2019 stated that the specific surface area of nanoparticle influences various physicochemical properties of a material, such as its surface area, mechanical strength, optical properties, and chemical reactivity. These properties make nanoparticles unique and usable in various applications such as in drugs and medications, manufacturing and materials, environment, electronics, energy harvesting, and mechanical industries.

Table 1: Top-down methods and bottom-up methods of nanoparticle preparation

No	Top-down method		Bottom-up method	
	Methods	**Examples**	**Methods**	**Examples**
1	Mechanical milling	Ball milling	Solid-state method	- Physical vapor deposition - Chemical vapor deposition
2	Laser ablation		Liquid state synthesis methods	- Sol-gel methods - Chemical reduction - Hydrothermal method - Solvothermal method
3	Sputtering		Gas-phase methods	- Spray pyrolysis - Laser ablation - Flame pyrolysis
4	Electro-explosion	These methods have not been used in the reduction of particle size since 2007	Biological methods	- Bacteria - Fungus - Yeast - Algae - Plant extract
5	Chemical etching		Other methods	- Electrodeposition process - Microwave technique - Supercritical fluid precipitation process - Ultrasound technique

Various methods can be applied for nanoparticle preparation, which is categorized into two main types: top-down method and bottom-up method, as shown in **Table 1**. **Figure 1** shows the difference between these methods is the starting material or precursor in the preparation of nanoparticles. The top-down method is when the bulk material or larger molecule (starting material) is decomposed into smaller units and then converted into nanosized particles, while the bottom-up method is when the atoms or molecules as the starting material are converted into nanosized particles; this method is also referred to as building-up method (Khan et al., 2019; Jamkhande et al., 2019; Kim et al., 2020).

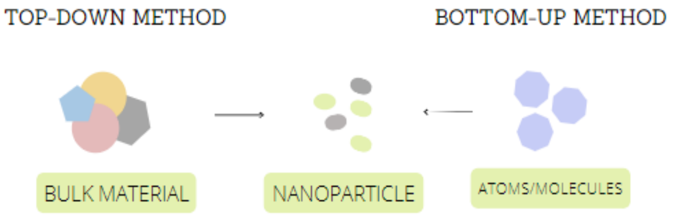

Figure 1: Outline of top-down and bottom-up method

This review focuses on the top-down method for nanoparticle preparation. As discussed earlier, bulk material or larger molecule is converted into a small nanosized particle, and size is reduced via different physical and chemical treatments such as mechanical milling, laser ablation, and sputtering. The top-down method is a suitable technique for the preparation of nanoparticles when the size of starting material or precursor is within the micrometer range (Bello et al., 2015). Top-down methods are easy to perform and applicable to the industrial scale-up but not

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suitable for preparing irregular-shaped and very small-sized particles (Jamkhande et al., 2019; Kim et al., 2020).

2.1 Mechanical milling

The principle of ball milling is size reduction into nanoparticles with a high-energy ball milling process. The reduction in particle size with an increase in milling duration leads to an increase in the surface area. However, the success of mechanical milling is affected by properties of milling powder and process variables such as charge ratio, rate of rotation, brittleness of materials, milling duration, size of the milling balls, and materials of which the balls are made (Ononiwu et al., 2020). Bello et al., (2015) used a ball milling method to prepare coconut shell (CS) powders from bulk CS with 2000 μm initial particle size. The CS powder is less than 37 μm in size and was produced using a hardened steel crusher and a disc grinder. The CS powder was milled for 70 h at 5 h per day using a planetary ball mill with a mixture of ceramic balls of different sizes (5–60 mm). They reported that the maximum and minimum particle sizes are 170.01 and 4.09 nm, respectively.

Krishna and Patel (2019) prepared CS powders by using a wet-stirred media mill, where they used surfactant to lower the surface tension and improve wetting of the feed sample dispersed in an aqueous solution. They stated that the amount of surfactant should be enough to provide wetting of raw samples and prevent agglomeration with enough surface coverage. The rate of particle breakage would be lowered if the surfactant is used in excess due to increased viscosity, hindering mobilization and stabilization. The milling was carried out with different feed concentrations (2, 4, and 6 g), different concentrations of the sodium salt of polyacrylic acid (PAA–Na) (3%, 5%, and 7%). and different surfactants (5 wt % surfactant concentration) such as Polysorbate 80 and PAA–Na. The feed of 4 g coconut shell powder produced nanoparticles with the smallest size; 5% of PAA–Na concentration produced 180.4 nm at 120 min; and 5 wt % of Polysorbate 80 produced CS powder with a size of 191 nm. The morphology of CS particles is spherical.

Chen et al., (2019) used the solvothermal method as a pre-preparation of $Cu(InGa)Se_2$ (CIGS) granules before transferring them to a ball milling system. They mentioned that several parameters affect the sizes of the nanoparticles, especially the amount of poly(ethylenimine) (PEI), chemical dispersant mixed with CIGS granulates, as well as the conditions of the ball milling process such as the size of zirconium (Zr) bead, milling speed, and time duration. Ten grams of CIGS granulates were dispersed in 0.2 g of PEI to avoid particle aggregation. They produced 40–60 nm of CIGS particle sizes by using 0.05 mm Zr beads and 3 h milling at 2000 rpm.

2.2 Sputtering

Sputtering is a physical vapor deposition (PVD) method via sputtering with a beam of inert gas ions (Jamkhande et al., 2019). **Figure 2** explains the process of sputtering where this process is carried out by attacking the surface of target substances with gaseous ions (usually Ar+) under high voltage acceleration to form nanoparticles. Low pressure is employed in the sputtering process to keep the target distance as the average size becomes independent of the substrate temperature (Abdelrahman, 2015). Low temperature prevents the growth of the film and leads to condensation of the deposited material in the form of nanoparticles (Verma et al., 2018). Various techniques of sputtering can be used for the preparation of nanoparticles such as magnetron sputtering, reactive sputtering, ion-beam sputtering, and others.

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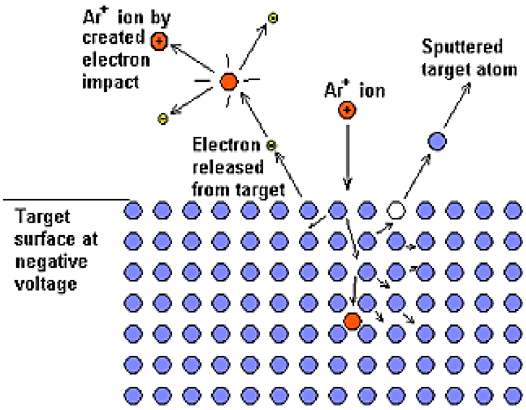

Figure 2: The process of sputtering (Abdelrahman, 2015)

Magudapathy et al., (2017) prepared three samples of copper (Cu) and noted them as large (L), medium (M), and small (S) with sizes 7×7, 5×5, and 3×3 mm, respectively using the ion beam-induced sputtering method. A 0.5 mm copper (Cu) foil was placed at the center of a silver (Ag) foil. These foils were irradiated by an Ar+ ion beam with a fixed dose of 1×10^{17} cm^{-2}. The amount of Cu and Ag atoms sputtered was managed by adjusting the region of Cu foil only. This process was carried out in a high vacuum chamber (~10−7 mbar) using Ar+ ion irradiation. The particle sizes of the Ag/Cu samples were determined by X-ray diffraction (XRD), where samples S, M, and L are 17, 23, and 32 nm, respectively. Their result showed that such method and conditions can change solid metals to powder particles where the particle size produced is directly proportional to the cutting size of solid matter.

Verma et al., (2020) used a direct current reactive magnetron sputtering technique for the preparation of copper oxide (CuO) nanoparticles at various working pressures (10, 20, 30, 40, and 50 mTorr). The particle sizes of CuO nanoparticles at 10, 20, 30, 40, and 50 mTorr of working pressure are 6, 8, 9, 11, and 13 nm, respectively. They concluded that the working pressure at 10 mTorr produced smaller nanoparticles as the increasing working pressure leads to decreasing mean free path where the atoms tend to agglomerate and slow down the growth of particles. In 2020, Verma et al. modified the reactive magnetron sputtering technique to synthesize tungsten (VI) oxide (WO_3) nanoparticles in a custom-designed vacuum chamber of 12-inch diameter. They varied the heating temperature of the furnace at 400, 500, 600, and 700 °C for 2 h. The particle sizes produced at 400, 500, 600, and 700°C are 28, 35, 41, and 48 nm, respectively. At 400 °C, smaller nanoparticles were produced since the increasing temperature affects the combining of grain boundaries and smaller nanoparticles into nearby larger nanoparticles.

2.3 Laser ablation

Laser ablation is the process where a laser beam is used to reduce the particle size to a nano level and ablate the target. A laser beam is applied to the target in a solvent to form nanoparticles, which remain in the liquid that surrounds the target and produces a colloidal solution (Jamkhande et al., 2019). Wenguang Cheng et al., (2020) found that the shape and size of nanoparticles can be controlled by suitable parameters such as the wavelength of the laser beam, laser energy, and laser width for target ablation. Chen et al., (2020) prepared zinc oxide (ZnO) nanoparticles using metallic zinc powders (24 μm) as the raw material. Zinc powders (30 mg) were put into a beaker that contained 30 mL deionized water and continuously stirred to prevent the zinc powders from sinking. A pulsed laser beam from a frequency-doubled Q-switched Nd:YAG laser was focused by a cylindrical convex lens with a focal length of 30

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mm. The lens was placed 22 mm away from the beaker and the laser energy was set at 50, 80, 110, and 140 mJ/pulse. The particle size observed by transmission electron microscopy (TEM) was 10–50 nm. This result proved that the percentage reduction of the size is around 99%.

Tan et al., (2019) successfully prepared gold (Au), silver (Ag), copper (Cu), aluminum (Al), and nickel (Ni) nanoparticles with 0.6, 0.6, 0.1, 1.04, and 0.6 mm of thickness, respectively via laser ablation method. The metals were put respectively in a glass vessel with 3 mL of distilled water. The laser was operated with two different wavelengths for each metal, which were 1064 nm (infrared laser) and 532 nm (green laser). A focusing lens with a focal length of 100 mm and laser energies of 350, 550, 750, and 950 mJ were used to ablate the metals. As listed in **Table 2**, the smallest particle sizes were achieved for Au, Ni, Al, and Cu by using a green laser, while Ag was an infrared laser. The differences of Au, Ag, and Al particle sizes by using infrared red and green laser were too big compared to Cu and Ni.

Table 2: The results of particle size (Tan et al., (2019))

Type of nanoparticles	**Average diameter size (nm)**	
	1064 nm (Infrared laser)	**532 nm (Green laser)**
Au	12	5
Ag	19	32
Cu	15	14
Al	22	7
Ni	6	5

Rocha-Mendoza et al., (2018) successfully prepared ZnO nanoparticles by using a Zn disk of 2.54×0.375 cm and acetone as a liquid medium. A convex lens of 13 cm focal length was used with nanosecond laser pulses (1064 nm) to ablate the Zn target. The colloidal solution was evaporated to obtain ZnO nanoparticles. The particle size of ZnO nanoparticle is 5–8 nm while the morphology of ZnO nanoparticle is spheroidal. **Figure 3** shows the SEM image and the morphology of ZnO nanoparticle.

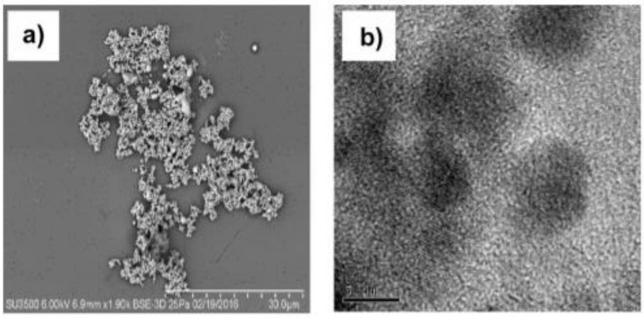

Figure 3: (a) SEM image of ZnO nanoparticles on a glass slide and (b) the morphology of ZnO nanoparticles (Rocha-Mendoza et al., 2018)

3. Methods of producing eggshell nanoparticles

As stated previously, eggshell is a domestic waste that has benefits toward the development of science in the whole world. For example, eggshell powder can be used to accelerate the degradation of dye wastewater because of its high absorption capacity. Therefore, the preparation of eggshell nanoparticles using various techniques needs to be considered as one of the important aspects to get the optimum absorption capacity by increasing the surface area. Many researchers have tried various techniques to get the desired nanoparticles size.

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3.1 Ball milling

Foroutan et al., (2019) successfully prepared eggshell nanoparticles with size 70 nm by using the ball milling method. They dried the eggshell in a microwave to remove moisture at 105 °C for 2 h and then crushed it by using a small ball mill. Their results only mentioned the final size of the eggshell produced. They did not mention the time interval for the ball milling process and the initial size; therefore, the percentage of size reduction cannot be calculated.

3.2 Ball milling and sintering

Puspitasari et al., (2019) prepared eggshell nanoparticles by ball milling method, followed by sintering. Sintering is a process of compacting and forming solid mass by heat without melting it. It is usually used for metals, ceramics, and other materials. They prepared a mixture that consisted of 300 g eggshells, 5 zirconia balls, and 30 mL acetone as a lubricant. The mixture was ball-milled with a varying time of 1, 5, and 10 h. After the ball milling process, the sample was dried in a microwave at 110 °C for 1 h to remove moisture in the sample. Then, it was crushed by using a crushed ball to reduce the size of the eggshell powder. Lastly, all the samples were sintered for 1 h.

As listed in **Table 3**, the non-sintered and sintered eggshell particle sizes ball-milled for 1 and 10 h showed almost the same results. However, 5 h of ball milling duration time influenced the reduction of particle size for both non-sintered and sintered conditions. Therefore, 5 h is the critical level (threshold) because at 10 h of ball milling, the particle size increased for non-sintered and sintered conditions as proved by scanning emission microscopy (SEM) images shown in **Figure 4**. It is interesting to note that the effect of the sintering process could reduce the particle size of the eggshell. Besides, the tricalcium phosphate compound completely turned into hydroxyapatite (HA) during the sintering process.

Table 3: Results for ball mill and sintering of eggshell (Puspitasari et al., (2019))

Time for ball milling	Particle size (nm)	
	Non-sintered	Sintered
1 h	59.61	52.18
5 h	51.18	46.37
10 h	59.58	52.14

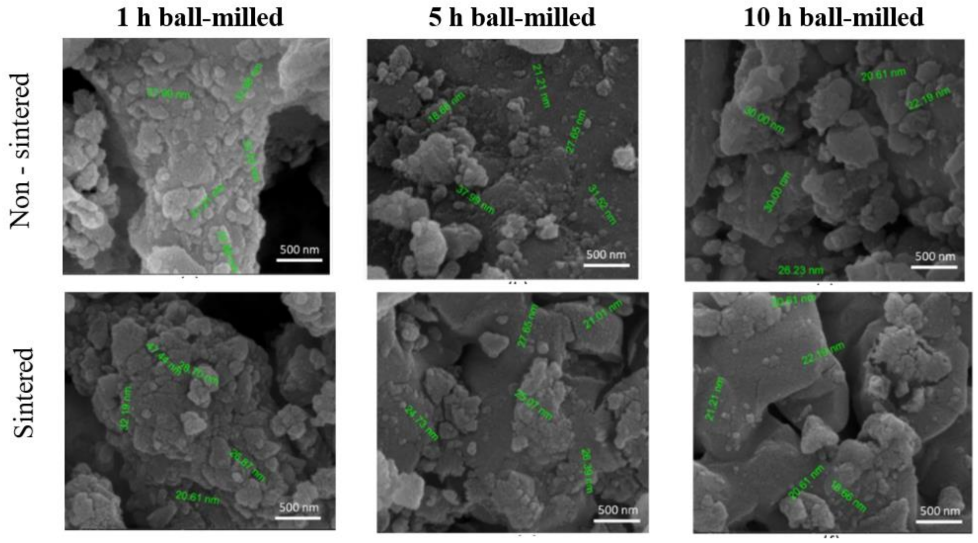

Figure 4: SEM images of eggshell for different durations of ball milling, and non-sintered and sintered conditions (Puspitasari et al., (2019))

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Singh et al., (2017) used nanoeggshell supported with TiO_2 to degrade the mixture of methylene blue and rhodamine 6G in an aqueous solution. In their study, they prepared nanosized eggshells by grinding and calcination method. Unfortunately, they did not mention the final size of the TiO_2 support on eggshell.

In recent study, Eskikaya et al., (2022) used ball milling and sintering methods to get eggshell-CaO nanocrystals from chicken eggshells for Safranin and Red 180 decolorization. They found that 1g/L of eggshell-CaO sintered at 900^{o}C achieved 100% and 97.90% of Safranin and RR180 dyes, respectively in 6.80 for Safranin and 6.60 for RR180 of the original pH solution.

3.3 Ball Milling and Ultrasound

Besides sintering, the ultrasound method after the ball milling process has been employed by Villarreal-Lucio and Rivera-Armenta (2018). The main purpose of their study was to use sonofragmentation method to reduce the particle size of the eggshell. Sonofragmentation is a process where ultrasound is used to radiate the particles in water (this process is usually done in water), which causes pressure and compression in water molecules. After the pressure has passed the required capacity, the sample breaks down and creates smaller particles.

In their experiment, eggshell was ground using a blender and soaked in acetone for 2 h. After that, the sample particle size was reduced by using a high-energy ball mill for 10 h using distilled water as a lubricant. Lastly, the sample was further reduced by using a UP200Ht Hielscher ultrasonic horn with 26 kHz frequency and wave amplitude of 100%. The pulse was set at 100% for a continuous operation. The particle size of the sample produced is 350 nm. Liew et al., (2015) also used ultrasonification method as an additional way to reduce the particle size of chicken and duck eggshell. Both eggshells were first washed and fully dried to remove any impurities. They successfully prepared eggshell nanoparticles with a size of 300 nm.

3.4 Micronizer and microwave

Hatim and Ahmad (2013) used micronizer and microwave to create nanosized hydroxyapatite (HA) powder, which can be obtained from eggshell, for medical purposes. Micronizer is a type of ball mill that grinds materials down to sizes of 1 to 10 µm. They crushed the eggshell and calcined it in an air atmosphere at 900 °C to transform calcium carbonate into calcium oxide. After that, phosphoric acid was slowly added to calcium oxide under constant stirring to form hydroxyapatite. The HA powder was then ground by using a domestic blender for 1 min. The HA powder was then ground by using a micronizer. Right after the grinding, a microwave was used to irradiate the HA powder on high power for 30 and 60 min. Irradiation for 30 min resulted in a smaller size compared to 60 min, which was 0.1563 and 2.9763 µm, respectively.

3.5 Comparison of data based on literature

As listed in **Table 4**, ball milling followed by the sintering method produced the smallest eggshell nanoparticles compared to other methods. However, this comparison is possibly not accurate because the initial particle size of starting materials was not stated; hence, the percentage of size reduction cannot be calculated. There may be a possibility that the starting material is already small enough before the ball milling process, which makes the percentage of size reduction low. For the ultrasound method, it is possible that the frequency is not high enough to create smaller particles. Based on these results, it can be concluded that until now, the ball milling process is the first step to reduce the size of the eggshell particle, followed by other additional methods to obtain the smallest particle size.

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Table 4: Comparison of the eggshell particle size achieved from various methods.

Method	Eggshell particle size (nm)	References
30 min microwave irradiation	1563	Hatim et al., 2013
10 h ball milling followed by ultrasound	350	Villarreal-Lucio and Arment, 2018
Ultrasound	300	Liew et al., 2015
Ball milling	70	Foroutan et al., 2019
5 h ball milling followed by 1 h sintering	46.37	Puspitasari et al., 2019

4. Conclusion

This paper provides a comprehensive review of the various techniques employed for synthesizing nanoparticles utilizing eggshell as the primary material. Among the methods discussed, laser ablation emerges as a highly promising approach for nanoparticles generation due to its exceptional ability to produce suspended nanoparticles. Moreover, it enables the creation of nanoparticles within the range of 5 nm, surpassing the capabilities of alternative top-down methodologies. Notably, despite ongoing research, the production of eggshell nanoparticles remains an area that has not been extensively explored. As such, this review paper serves as a valuable resource for individuals seeking to gain profound insights into the preparation of eggshell nanoparticles, offering guidance and direction in this field of study.

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