

## CRITICAL REVIEW

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# Microwave-assisted synthesis of nanomaterials: a green chemistry perspective and sustainability assessment

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This review critically evaluates microwave-assisted synthesis (MAS) as a sustainable approach for nanomaterial fabrication, addressing environmental concerns associated with conventional methods. MAS offers significant advantages through rapid, uniform heating that reduces energy consumption, reaction times, and hazardous waste generation. By systematically comparing energy, reaction efficiency, waste reduction, selectivity, product uniformity and scalability of MAS with conventional techniques, this review provides a comprehensive framework for sustainable nanomaterial production. The review applies green chemistry metrics and sustainability assessment tools to evaluate the environmental performance and industrial viability of various MAS protocols. Moreover, the integration of MAS with eco-friendly precursors, including plant extracts, biomolecules, and ionic liquids, for synthesising three key nanomaterial classes, such as metal nanoparticles, carbon quantum dots (CQDs), and hybrid nanocomposites was discussed in detail. The discussion further extends to practical applications in catalysis, environmental remediation, energy storage, and biomedical technologies, highlighting how MAS-derived nanomaterials address contemporary sustainability challenges. The review concludes by identifying current challenges and future directions for advancing MAS toward industrial-scale implementation, emphasizing its potential to transform nanomaterial manufacturing into a more environmentally responsible process aligned with circular economy principles.

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## Sustainability spotlight

Conventional nanomaterial synthesis methods typically involve excessive energy consumption and toxic chemicals, and generate significant waste. Our review critically examines microwave-assisted synthesis (MAS) as a sustainable alternative that addresses these challenges through rapid, uniform heating mechanisms that substantially reduce energy usage, processing time, and hazardous waste. This work advances sustainability by providing comprehensive green chemistry metrics and assessment frameworks for MAS protocols, particularly for metal nanoparticles, carbon quantum dots, and hybrid nanocomposites with applications in catalysis, remediation, energy storage, and medicine. Our research aligns with UN SDGs 7 (Affordable and Clean Energy), 9 (Industry, Innovation and Infrastructure), and 12 (Responsible Consumption and Production) by promoting energy-efficient manufacturing techniques, cleaner production pathways, and circular economy principles in nanomaterial fabrication.

## 1 Introduction

Nanomaterials have emerged as a ground-breaking field with the potential to revolutionize various industries ranging from healthcare to energy production.<sup>1</sup> Due to the unique properties exhibited by these materials at the nanoscale, the synthesis of nanomaterials has received considerable attention these days.<sup>2</sup> Generally, these properties differ considerably from those of their bulk counterparts<sup>3,4</sup> and hence, these nanomaterials are extremely promising for use in a wide range of applications including catalysis, sensing, biomedical imaging, drug delivery,

environmental remediation and energy storage.<sup>5–7</sup> However, the translation of these promising properties into practical applications has been hampered by synthesis challenges, particularly the environmental and economic costs associated with conventional production methods. The critical question facing the field is whether emerging synthesis techniques can deliver on their promises of sustainability while maintaining the precision and scalability required for industrial applications.

Conventional synthesis approaches for nanomaterials have been extensively criticized for their environmental impact and practical limitations.<sup>2,8,9</sup> These chemical routes typically require high temperatures and pressures, utilize toxic solvents and reducing agents, and generate hazardous by-products that pose renowned risks to human health and ecosystems.<sup>10</sup> A critical examination of the literature reveals that many such

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approaches fail to provide comprehensive life-cycle assessments or quantitative comparisons of environmental impact, making it difficult to accurately assess the true magnitude of these problems. Furthermore, the economic costs of conventional methods are often inadequately reported, limiting our understanding of the trade-offs between environmental sustainability and commercial viability.

Sustainable and eco-friendly synthesis methods are essential to minimize the environmental impact and promote greener approaches in nanomaterial production, thereby addressing the limitations of conventional synthesis techniques.<sup>11</sup> Green and sustainable approaches, such as microwave-assisted synthesis, sonochemical synthesis, plant extract-mediated synthesis, and

biomolecule-assisted synthesis, offer eco-friendly alternatives by reducing energy usage, eliminating harmful reagents, and enabling efficient, scalable production.<sup>1</sup> These methods promote cleaner and safer nanomaterial fabrication and enhance material properties for applications in medicine, energy storage, catalysis, and environmental remediation.<sup>12</sup> The shift towards sustainable synthesis ensures long-term resource efficiency and supports global efforts toward greener technologies and a circular economy.<sup>9</sup> Microwave-assisted synthesis (MAS) as an emerging sustainable technique, promises several advantages over conventional methods.<sup>12,13</sup> MAS is a process whereby chemical reactions are carried out using microwave irradiation to achieve uniform and rapid heating of the reaction mixture<sup>14</sup>. There is a growing global interest in this approach for sustainable nanomaterial fabrication.<sup>15</sup>

Despite growing interest in MAS applications across metallic nanoparticles,<sup>10</sup> metal oxides,<sup>16,17</sup> quantum dots,<sup>18</sup> and carbon-based nanomaterials,<sup>19</sup> the literature exhibits significant methodological inconsistencies that compromise the reliability of performance comparisons. Studies frequently lack standardized characterization protocols, employ different metrics for evaluating synthesis success, and rarely provide direct comparisons with conventional methods under equivalent conditions. Additionally, the claims of reduced reaction times and higher yields are not universally supported across all nanomaterial types, and the energy efficiency comparisons with conventional methods often lack rigorous accounting for total system energy consumption. The assertion that MAS enables “milder conditions” requires contextualization, as microwave heating can create localized hot spots and non-uniform heating profiles that may not represent true process improvements.<sup>20</sup> Furthermore, the environmental benefits of using “benign” solvents in MAS are undermined when the overall process still requires substantial energy input and specialized equipment with limited lifecycle assessments.<sup>21</sup>

Therefore, this review addresses these fundamental gaps by systematically evaluating the evidence supporting MAS as a sustainable nanomaterial synthesis approach. We critically analyze the methodological rigor of existing studies, assess the validity of claimed advantages through comparative analysis, and identify where evidence is strong *versus* where claims may be overstated. Our evaluation framework examines synthesis parameters, characterization methods, and performance metrics across different nanomaterial types while scrutinizing the quality and comparability of reported data. We critically assess green metrics and sustainability claims, examining both the explicit environmental benefits and potential hidden costs of MAS approaches. Finally, we provide a balanced evaluation of MAS scalability challenges and realistic prospects for industrial translation, distinguishing between demonstrated capabilities and speculative projections to guide future research priorities in sustainable nanomaterial synthesis.



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## 2 Fundamental principles of MAS

The underlying principle of microwave-assisted synthesis distinguishes itself through electromagnetic energy delivery within the



0.3–300 GHz spectrum, creating internal heat generation rather than relying on surface-to-core thermal transfer characteristics of traditional methodologies.<sup>22</sup> Conventional heating strategies necessitate sequential energy migration through conductive and convective pathways, inherently producing thermal gradients and extended processing durations.<sup>23</sup> The mechanism involved in MAS of nanomaterials is depicted in Fig. 1. Polar molecules or ions absorb microwave radiation in the reaction mixture in microwave-assisted synthesis. Due to this absorption, localized heating occurs at the molecular scale and allows for breaking of chemical bonds and chemical reactions to begin.<sup>24</sup> This selective heating of reaction components provides a means of controlling reaction conditions, specifically temperature, pressure, and reaction kinetics, with a degree of precision.<sup>25</sup>

Microwave technology promotes simultaneous molecular agitation *via* dipole oscillation and charged particle migration throughout the entire reaction volume. While this internal energy deposition theoretically achieves homogeneous temperature profiles and accelerated kinetics, practical implementation reveals significant challenges.<sup>25</sup> The vessel configuration, reaction scale, and material dielectric characteristics introduce heterogeneous energy absorption patterns that compromise the uniformity assumption, raising concerns about process reproducibility and commercial scalability potential. If uniform heating, a primary claimed advantage of MAS, cannot be reliably achieved, then comparative advantages over conventional methods require reconsideration.

### 3 Nanomaterial synthesis *via* MAS

Continued development of MAS techniques has led to the utility of such methods for rapid and efficient fabrication of

nanomaterials tailored with required properties for numerous applications. They take advantage of the speciality of microwave irradiation to both speed up chemical reactions and produce nanomaterials in controlled environments. Hydrothermal<sup>2,26</sup> and solvothermal<sup>27,28</sup> synthesis are among the diverse MAS methods that use high temperature and pressure to foster the nucleation and growth of nanoparticles in aqueous and organic solvents, respectively. Microwave energy is utilized in various nanomaterial synthesis approaches such as sonochemical synthesis, combustion synthesis, solid-state synthesis, and plasma-assisted synthesis.

Microwave irradiation and ultrasonic waves induced chemical reactions in liquid media, also called 'sonochemical synthesis', and provide rapid synthesis of nanomaterials with improved yield and control over the size and shape of the particles.<sup>29</sup> Microwave-heated combustion synthesis (MHCS) utilizes microwave heating to initiate exothermic reactions between the metal precursor and fuel source to produce nanomaterials with controlled stoichiometry and phase purity at rapid combustion rates.<sup>30</sup> However, MHCS is well-known for its rapid reaction rates, and its scalability and control over byproducts remain underexplored. Microwave irradiated solid state synthesis employs microwave irradiation to trigger the solid-state reactions between solid reactants or precursors for synthesizing various complex nanostructures through solid-state transformations at elevated temperatures.<sup>31</sup> Furthermore, plasma synthesis techniques employ microwave energy to produce and maintain plasma discharges for synthesizing nanomaterials whose sizes, shapes and surface properties are controlled.<sup>32</sup> Plasma synthesis raises sustainability concerns due to its high energy input, which contradicts the green chemistry goals unless renewable energy is employed.

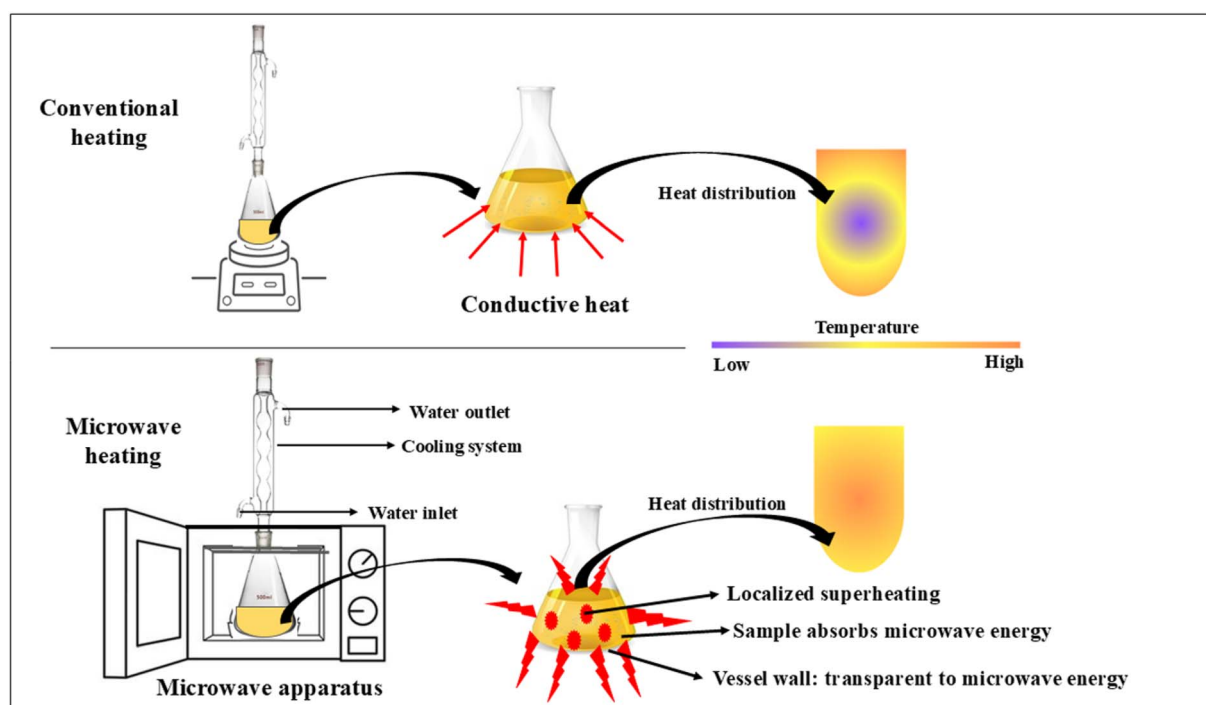


Fig. 1 Schematic representation of the mechanism of conventional and microwave heating.



Due to their high reaction efficiency, scalability and product quality, these MAS techniques are promising tools for the sustainable synthesis of nanomaterials for a variety of applications in catalysis, electronics, energy storage or biomedical engineering. The blanket assertion that MAS methods inherently ensure product quality and sustainability overlooks the need for lifecycle assessments and standardized metrics to substantiate these claims. Therefore, a more nuanced analysis, considering both the technical advancements and practical trade-offs of MAS, is essential for framing its role in sustainable nanomaterial development.

Utilizing microwave irradiation, researchers can efficiently synthesize a broad range of nanostructures, including metal nanoparticles, metal oxides, carbon-based quantum dots, and nanocomposites.<sup>15,33,34</sup> A detailed discussion about MAS of three major nanomaterials such as metal nanoparticles, carbon quantum dots (CQDs), and nanocomposites is outlined below. Metal and metal oxide nanoparticles are widely studied for their unique electronic, optical, and catalytic properties,<sup>34</sup> while CQDs have attracted growing attention due to their excellent fluorescence, water dispersibility, and low toxicity.<sup>35</sup> Nanocomposites, which integrate two or more nanoscale components, offer synergistic effects that improve structural and functional performance.<sup>36</sup>

### 3.1 Metal nanoparticles

In recent years, the synthesis of metal nanoparticles (MNPs) has attracted considerable attention because of their unique physicochemical properties and applications in various fields.<sup>37</sup> Unique techniques for the synthesis of MNPs, including chemical reduction, sol-gel processing, and hydrothermal methods, have several disadvantages, such as long reaction times, high energy consumption, and the adoption of toxic reducing agents.<sup>38</sup> Microwave-assisted synthesis is widely employed in the laboratory for producing metal nanoparticles with controlled size, shape, and composition.<sup>39</sup> An existing review article on microwave-assisted synthesis of metal nanoparticles (MNPs) discusses the advantages of this method in obtaining high-purity matrices with near-monodispersity and tunable properties.<sup>40</sup>

The MAS approach appreciably improves reaction kinetics, induces effective nucleation, and reduces undesired side reactions, thus rendering it highly appropriate for the manufacture of noble and non-noble metal nanoparticles,<sup>41</sup> along with bimetallic,<sup>42</sup> trimetallic<sup>43</sup> and metal-based nanocomposites.<sup>44</sup> Microwave irradiation offers more control over reaction parameters than traditional heating, cutting processing times from hours to minutes while increasing yield and reproducibility.<sup>45</sup> Additionally, MAS supports green chemistry techniques, which frequently use ionic liquids, plant extracts, or bio-based reducing agents to reduce their negative effects on the environment.<sup>46</sup>

**3.1.1 Noble metal nanoparticles (Ag, Au, Pt, Pd, Rh, and Ir).** The distinctive optical, electronic, and catalytic characteristics of noble metal nanoparticles, such as silver (Ag), gold (Au), platinum (Pt), palladium (Pd), rhodium (Rh), and iridium (Ir),

have led to extensive research into these materials. Compared to traditional methods, MAS improves their size control, crystallinity, and monodispersity.

Silver nanoparticles (AgNPs) are synthesized using a variety of chemical and biochemical techniques.<sup>47</sup> Green synthesis of AgNPs, which uses bio-based precursors for their synthesis, has gained more attention recently as a sustainable approach.<sup>48</sup> Catalysis, sensing, and antimicrobial applications all make extensive use of the synthesized AgNPs.<sup>49</sup> AgNPs with specific morphologies, including spherical particles, triangular nanoplates, nanowires, and dendritic structures, and narrow size distributions can be produced through microwave synthesis.<sup>50</sup> Specific research indicates that the shape of nanoparticles and their bioactivity are greatly influenced by reaction time, precursor concentration<sup>51</sup> and the nature of reducing agents; for example, stress-induced plant extract shows favourable size and antibacterial properties compared to normal plants.<sup>52</sup>

Like AgNPs, gold nanoparticles (AuNPs) are rapidly nucleated and grow when exposed to microwave radiation, producing regulated shapes such as spheres, rods, and hexagonal plates.<sup>53</sup> To ensure sustainable synthesis with high catalytic and plasmonic properties, reducing agents such as ionic liquids and biological extracts have been employed.<sup>54–56</sup>

Palladium (Pd) and platinum (Pt) nanoparticles (NPs) are essential for hydrogen storage, catalysis, and fuel cells. Highly dispersed Pt<sup>57</sup> and Pd<sup>58,59</sup> nanoparticles with superior electrocatalytic performance are produced by microwave-assisted reduction of metal salts using ethylene glycol, polyvinylpyrrolidone (PVP), and ascorbic acid.<sup>40</sup> The synthesis of rhodium (Rh) and iridium (Ir) nanoparticles using a microwave enhances their efficiency in hydrogenation reactions and catalytic applications by producing well-defined nanostructures with regulated oxidation states.<sup>40</sup> MAS ensures sustainable synthesis of these noble metal nanoparticles with superior catalytic activities and may show high qualities in various applications.

**3.1.2 Non-noble metal nanoparticles (Cu, Ni, Co, Fe, Zn, and Mn).** Despite being less expensive, non-noble metal nanoparticles need to be carefully stabilized because of their high oxidation and aggregation susceptibility. Stable nanostructures can be created quickly and under control using MAS techniques. Copper nanoparticles (CuNPs),<sup>60,61</sup> copper nanowires, nanospheres, and core-shell structures can be effectively produced by modifying the reaction conditions.<sup>62</sup> Iron (Fe),<sup>63</sup> cobalt (Co),<sup>64</sup> nickel (Ni),<sup>64,65</sup> zinc oxide (ZnO)<sup>13,66</sup> and manganese dioxide (MnO<sub>2</sub>) nanoparticles (NPs)<sup>5</sup> have been synthesised using MAS and they exhibited a wide range of applications. Rapid nucleation and consistent particle growth are made possible by microwave irradiation, which also increases these particles' properties. By fine-tuning microwave parameters, their crystal structure and surface morphology can be improved.

**3.1.3 Bimetallic and trimetallic nanoparticles.** Compared to their monometallic counterparts, bimetallic and trimetallic nanoparticles have better synergistic qualities.<sup>67</sup> MAS improves heterojunction architectures, alloy formation, and core-shell structures of these nanoparticles<sup>40</sup> through its regulated heating





and consistent energy distribution. In contrast to their monometallic counterparts, bimetallic nanoparticles (BNPs) are nanostructures made of two distinct metal elements that combine the special qualities of both metals to display superior physicochemical properties.<sup>68</sup> Improved catalytic activity, optical behavior, magnetic properties, and stability are the results of the two metals working in concert, which makes BNPs extremely valuable in a variety of applications such as energy storage, sensing, medicine, and catalysis.<sup>69</sup> An effective, quick, and environmentally friendly method for creating BNPs with exact size control, morphology, and composition tuning is MAS.<sup>70,71</sup> Depending on their composition, Ag–Au alloy nanoparticles have adjustable plasmonic and catalytic characteristics.<sup>72</sup> Because of their increased electrocatalytic activity, Pt–Pd<sup>73</sup> and Pd–Au core–shell structures are being investigated extensively for fuel cell applications and catalysis. Microwave-synthesised Cu–Ag bimetallic nanoparticles have demonstrated encouraging improvements in electrical conductivity and antimicrobial activity.<sup>74</sup> Ag–ZnO BNPs were fabricated *via* a microwave strategy for better antibacterial activity.<sup>75</sup> Fig. 2 provides a schematic illustration of how bimetallic Ag–Cu NPs are made from plant extract *via* MW irradiation.

Trimetallic nanoparticles (TNPs) have drawn a lot of interest because, when three distinct metal elements are combined, they produce synergistic effects that improve their physicochemical characteristics when compared to those of their monometallic and bimetallic counterparts.<sup>76</sup> These nanoparticles are ideal for use in energy conversion, environmental remediation, and the biomedical industry because of their exceptional catalytic activity, electronic characteristics, and thermal stability.<sup>77</sup> In methylene blue degradation, Au–Pt–Pd

nanofluid synthesised *via* microwave irradiation shows exceptional stability and catalytic efficiency.<sup>78</sup> TNPs such as AuPtCu,<sup>43</sup> AgRuNi<sup>79</sup> and PtPdCu<sup>57</sup> were also prepared *via* MAS strategies, and they were applicable as an anti-bacterial agent, photocatalysts, and stable catalysts, respectively. Through MAS of trimetallic nanocomposites on CQDs, La/Cu/Zr/CQDs<sup>80</sup> and Ag/Ti/Zn/CQDs<sup>81</sup> have been fabricated and applied for the removal of malachite green dye and solar cells, respectively.

**3.1.4 Metal nanocomposites.** Metal-based nanocomposites improve the mechanical, thermal, and functional properties of carbon, polymer, or ceramic matrices by integrating nanoparticles into them.<sup>65</sup> The unique properties of MAS guarantee that the nanoparticles are uniformly distributed throughout the matrix. High-performance catalysts, sensors, and energy storage nanocomposites are produced when reduced graphene oxide (rGO) is mixed with metal nanoparticles such as rGO–iron oxide,<sup>82</sup> BiOBr/rGO,<sup>83</sup> rGO–Ag, rGO–SnO<sub>2</sub>,<sup>84</sup> Ag–ZnFe<sub>2</sub>O<sub>4</sub>@rGO,<sup>85</sup> and rGO–TiO<sub>2</sub>.<sup>86</sup> Microwave-assisted metal-oxide nanocomposites, like MgFe<sub>2</sub>O<sub>4</sub>–ZnO,<sup>87</sup> chitosan–metal oxide nanocomposite,<sup>88</sup> Co<sub>3</sub>O<sub>4</sub>–graphene sheet,<sup>89</sup> graphene–SnO<sub>2</sub>,<sup>90</sup> LiFePO<sub>4</sub>/C,<sup>28</sup> cellulose/AgCl,<sup>91</sup> graphene–ZnO,<sup>92</sup> NaFePO<sub>4</sub>–C,<sup>93</sup> MoS<sub>2</sub>/PANI,<sup>94</sup> carbon–zirconium-incorporated CeO<sub>2</sub>,<sup>95</sup> g-C<sub>3</sub>N<sub>4</sub>/SnO<sub>2</sub>,<sup>96</sup> CuO/MnO<sub>2</sub>,<sup>5</sup> ZnO/MWCNT,<sup>97</sup> gadolinium/cerium oxide,<sup>98</sup> CdO–ZnO,<sup>99</sup> ZnO/CuO,<sup>100</sup> Sb<sub>2</sub>O<sub>3</sub>–Ag<sup>101</sup> and graphene/CoMoO<sub>4</sub> (ref. 102) exhibit remarkable photocatalytic activity and stability, which makes them perfect for solar energy conversion, dye degradation, antimicrobial application, pollutant degradation, supercapacitors, gas sensors, and lithium and sodium ion batteries. Fig. 3 depicts the antibacterial activity of metal nanocomposites of silver nanoparticles

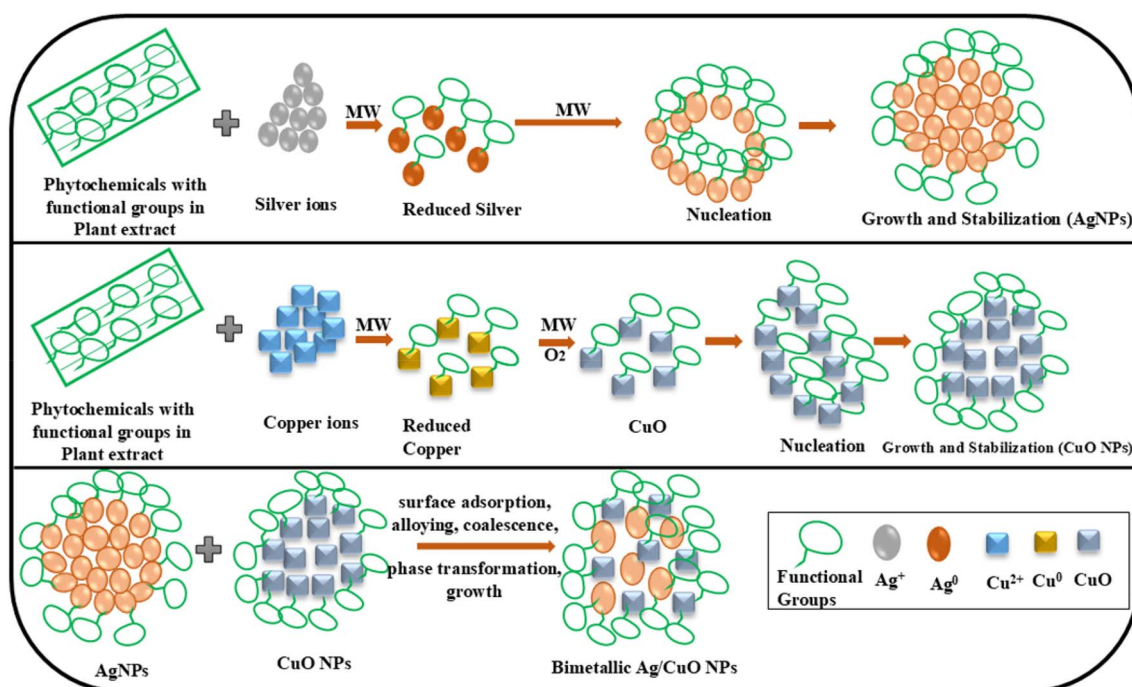


Fig. 2 Schematic representation of the formation of bimetallic Ag–Cu NPs from plant extract.



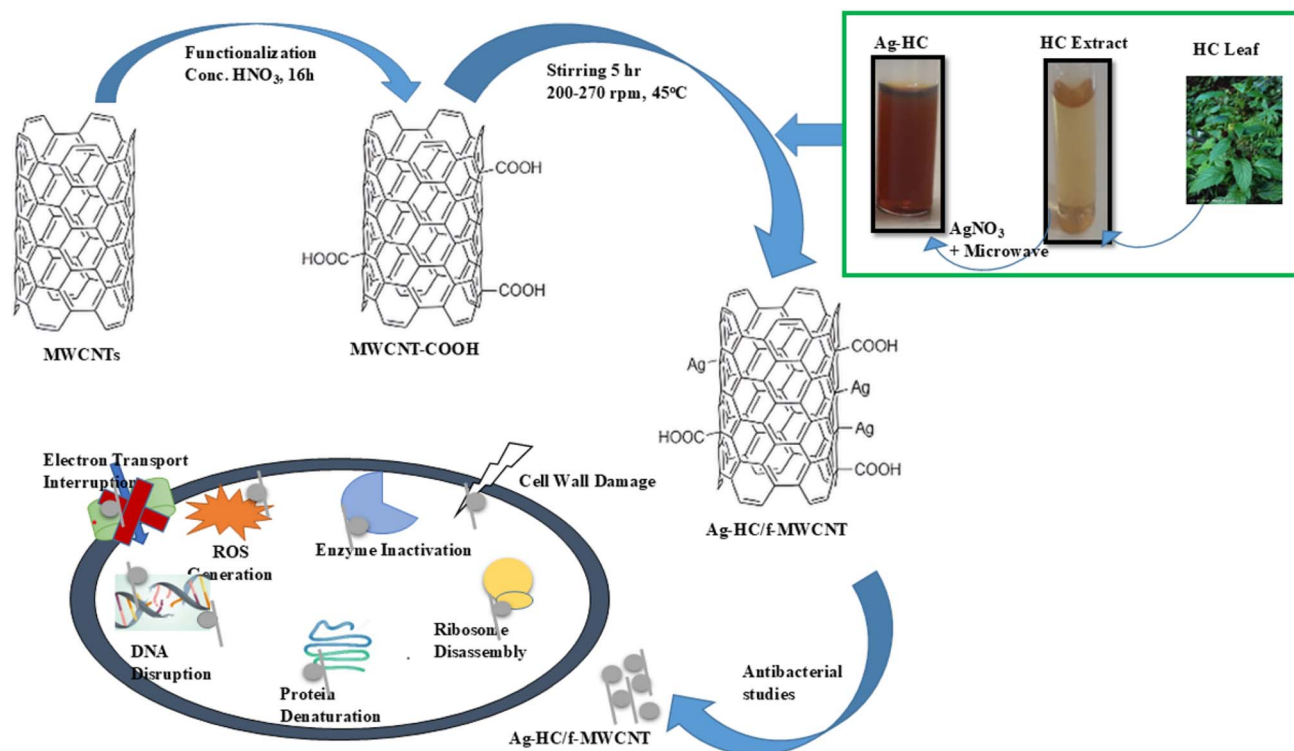


Fig. 3 Schematic representation of the antibacterial activity of Ag-HC/f-MWCNT. Reproduced with permission from ref. 44. Copyright, 2023, Springer.

decorated on functionalized MWCNTs synthesised *via* the microwave method.<sup>44</sup>

Microwave-assisted synthesis has undeniably transformed the landscape of metal nanoparticle fabrication by enabling faster, more energy-efficient, and environmentally conscious routes compared to conventional techniques. While the advantages of MAS, such as reduced reaction time, enhanced monodispersity, and shape control, are highlighted for noble and non-noble metals, the sustainability implications of the precursors used (e.g., ethylene glycol<sup>60</sup> and polyvinylpyrrolidone<sup>40</sup>) and the post-synthesis processing steps are not thoroughly assessed.

Moreover, the increasing complexity from mono- to tri-metallic systems introduces new challenges in phase control, alloying behavior, and surface segregation, yet these are seldom critically discussed in the literature. Assertions regarding “synergistic effects” in bimetallic and trimetallic nanoparticles often lack quantitative validation or mechanistic insights. Likewise, while metal nanocomposites are described as having enhanced performance due to uniform dispersion, the scalability of such uniformity, especially in solid supports like graphene or cellulose, remains a significant hurdle. A more nuanced evaluation is warranted, especially in linking synthesis parameters (microwave power, irradiation time, and solvent type) with property tuning and sustainability metrics. Without standardized benchmarking across studies, the real comparative advantage of MAS remains difficult to quantify, particularly when transitioning from laboratory-scale demonstrations to industrial-scale applications.

### 3.2 Carbon quantum dots

Nearly spherical metal oxide particles comprising carbon atoms known as carbon quantum dots (CQDs) have gained a noticeable amount of attention due to their ease of functionalization and high biocompatibility.<sup>103</sup> CQDs are guaranteed to reach their goals through microwave heating as it allows for direct contact instead of surface contact with solvents and other precursors, turning them into dielectrics.<sup>104</sup> CQDs are useful for many applications,<sup>105</sup> summarized in Table 1, and the following items are commonly needed for their synthesis. Carbon precursors: they include glucose, citric acid or even agricultural waste such as banana peels.<sup>106</sup> Reaction medium: solvents like ethylene glycol, water and ionic liquids, which are capable of soaking up energy from the microwave, are used. Reaction conditions: the desired mixture is commonly exposed to a level of microwave radiation between 500 and 900 W for a time span between 2 and 15 minutes. Wrap-around doping with heteroatoms (like phosphorus, nitrogen and sulfur) boosts the optical performance and photoluminescence of CQDs, ensuring a photoluminescence quantity of 98 percent.<sup>107</sup> Functionalization also enhances the binding of CQDs with target molecular species, making their potential application better.<sup>108</sup>

In MAS of CQDs, a bottom-up approach is followed where small organic precursors are thermally decomposed, polymerized, and carbonized to yield nanoscale carbon quantum dots, which is depicted in Fig. 4.<sup>109</sup> Polar molecules of the precursor solution can absorb electromagnetic energy when subjected to microwave irradiation, resulting in broad and uniform heating through dipole rotation and ionic conduction.<sup>110</sup> This heating



Table 1 CQDs formed via microwave irradiation and their application

Name of CQDs	Precursors/materials used	Microwave specification	Application	Reference
Fluorescence CQDs	Roasted chickpeas	350 W	Detection of Fe <sup>3+</sup> ions	112
N, S co-doped CQDs	Citric acid and 4-aminobenzenesulfonic acid	800 W, 5 min	Fe <sup>3+</sup> and hydroquinone detection	107
Amphoteric fluorescence CQDs	Sugarcane bagasse (SB) and citric acid	750 W, 135 min	Chromium adsorption	113
Fluorescence CQDs	Succinic acid (SA) and tris (2-aminoethyl)amine (TAEA)	700 W, 5 min	Multi-color fluorescence imaging of cellular media	114
CQDs	<i>Hibiscus rosa-sinensis</i>	700 W, 1 min	Antibacterial, anti-inflammatory, and wound healing	26
CQD nanocomposite	Anchoring carbon quantum dots onto TiO <sub>2</sub>	—	CO <sub>2</sub> photoreduction	115
N, S doped CQDs	Cyclodextrin, melamine, and thiourea	700 W, 5 min	Sensor for silver(I) ions	116
Fluorescent carbon quantum dots	Mexican mint extract	1200W	Fe <sup>3+</sup> detection and bio-imaging	117
CQDs	Arabica coffee grounds	3–7 min	Detection of Fe <sup>3+</sup> , Pb <sup>2+</sup> , and Cr <sup>3+</sup>	18
CQDs	Raw cashew gum	800 W, 30–40 min	—	118
Lysine-based CQDs	Lysine	800 W	Luminescent and biocompatible	119
CQDs	L-Cystine	800 W, 30–90 s	—	120
CQDs	Acidic linter waste	400 W, 5 min	Fluorescence cancer imaging	121
N-CQDs	Ammonia solution of xylan	200 W, 10 min	Tetracycline detection	109
CQDs	Citric acid (monohydrate) and urea	700 W, 150–300 s	—	110
N-CQDs	Glucose water solution with ammonia hydroxide	100 W and 200 W, 60 s	Catalytic	122
CQDs	Glucose	500 W, 7–11 min	Photocatalytic	123
CDs@MIPs	Citric acid	750 W, 2 min 30 s	Tetracycline detection	124
CQDs	Lignocellulosic residues	100 °C for 120 min	—	125
CQDs	A4/B2 polyamidation monomer sets	—	Cu <sup>2+</sup> detection	111
CQDs	<i>Ziziphus Mauritiana</i> (ZMS) biomass	—	Detection of NH <sub>3</sub>	126
CQDs	Fenugreek seeds	500 W	To grow fluorescent protein crystals	127
S, N-CQDs	Cabbage and onion	700 W, 4 min	Fluorescence sensor for nitazoxanide and hemoglobin	128
CQDs	Citric acid	700 W, 3 min	Degradation of rhodamine-B	129
CQDs	Bamboo tar	180 °C, 15 min	Detection of 2,4,6-trinitrophenol	130
Co-NS-CQDs	Citric acid and methionine	1.5 min	Detection of monosodium glutamate	131
CQDs	Citric acid	800 W, 5 min	Cellular imaging	132
Fe-CQDs	Citric acid monohydrate and urea	750 W, 150 s	Detection of diclofenac sodium	133
CQDs	L-Arginine	3 min	Detection of Fe <sup>3+</sup> ions	134
N,S-CDs	Citric acid and thiosemicarbazide	1 min	Sensor for copper (Cu <sup>2+</sup> ) and etidronate disodium (ETD)	135
CQDs	<i>Calotropis gigantea</i>	2 min	Bioimaging	136
CQDs	Polyethylene glycol	500 W, 60 min	—	137
CQDs	EDTA, sodium thiosulfate, and urea	2 min	Sensor for ascorbic acid and riboflavin	35
CQDs	Cellulose and carboxymethyl cellulose	750 W, 135 min	Chromium absorption	138
CQDs	Quince fruit	30 min	Cell imaging and As <sup>3+</sup> determination	139
CQDs/Ag nanocomposite	Glucose and natural orange juice	—	Colorimetric sensor for losartan potassium	140
CQDs@PAFP	Starch	700 W, 10 min	Removal of uranium	141



Name of CQDs	Precursors/materials used	Microwave specification	Application	Reference
N@CQDs	<i>Eruca sativa</i> leaves	700 W, 4 min	Estimation of modafinil	142
CQDs	Orange peels	—	Photosensitizers of TiO <sub>2</sub>	108
N, S-CDs	Sucrose, urea, and thiourea	4 min	Natamycin determination	143
N-CQDs	Orange juice and urea	10 min	Determination of palbociclib	144
CQDs	Crude glycerol residue		Sensing Fe <sup>3+</sup> and Cu <sup>2+</sup> and the photocatalysis of Victoria Blue B dye	145
S-CQDs	Reduced glutathione and L-cysteine	2 min	Detection of Co <sup>2+</sup> and Ni <sup>2+</sup>	146
CQDs	Citric acid monohydrate and urea	—	Detection of tartrazine	147
NS-CQDs	Citric acid, urea, and <i>p</i> -aminobenzenesulfonic acid	800 W, 3–5 min	Detection of Fe <sup>3+</sup> ions and hydroquinone (HQ)	148
N-CQDs	Succinic acid and gallic acid	5 min	Detection of methyl orange in saffron	149
Cu-CQDs	Ammonium citrate and CuCl <sub>2</sub> ·2H <sub>2</sub> O	Programmed temperature control	Histidine detection	150
CQDs	<i>Araucaria heterophylla</i> gum extract	4 min	Drug detection	151
NS-CQDs	Citric acid and L-cysteine	1 min	Determination of ascorbic acid	152
CQDs	Ascorbic acid, vitamin C, EDTA, and polyethylene glycol	1 h	Dopamine detection	153
N-CQDs	Citric acid and urea	15 min	White-light emissive diodes	154
CQDs	Empty fruit bunch	1 min	Detection of Cu <sup>2+</sup>	155
NP@CQDs	EDTA and diammonium hydrogen phosphate	3 min	Determination of glutathione	156
ZnPc-CQDs	Zinc(II) tetra-amino-phthalocyanine (ZnPc) and citric acid	700 W, 2 min	Antibacterial	157

yield, purity, and lifecycle impacts. Moreover, although surface functionalization and heteroatom doping (*e.g.*, N, S, and P) are reported to enhance photoluminescence and sensing capabilities, there is limited mechanistic understanding of how these modifications influence CQD behavior across applications. The diversity of precursor materials, irradiation conditions, and inconsistent reporting standards hamper scalability and reproducibility. A more structured approach, incorporating mechanistic insights, lifecycle analysis, and standardized characterization, would significantly strengthen the scientific foundation of CQD synthesis *via* MAS and its viability for real-world sustainable applications.

The emergence of multifunctional nanocomposites has led to a rapid development within polymer matrices owing to the improved properties they possess.<sup>158</sup> Such composite materials are being synthesized at a fast pace in laboratories around the world for numerous applications. MAS is well known for producing nanocomposites with controlled architecture and enhanced performance.<sup>159</sup> One such example is the development of epoxy/graphene nanocomposites using MAS (Fig. 5a). The resulting graphene layers, dispersed within the epoxy matrix, were characterized using transmission electron



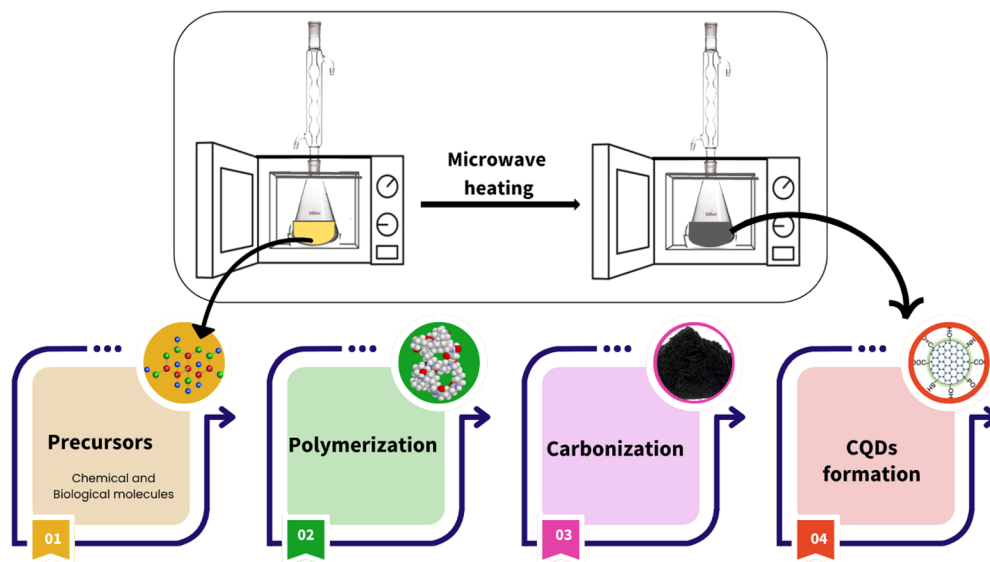


Fig. 4 Schematic representation of the process involved in CQD formation using microwave irradiation.

microscopy (TEM) and atomic force microscopy (AFM). The AFM analysis indicated that the graphene platelets were predominantly monolayer with an average thickness of  $4.17 \pm 0.63$  nm and lateral dimensions ranging between 1.00 and 4.77  $\mu\text{m}$  (Fig. 5c), confirming successful exfoliation and nanoscale uniformity.<sup>160</sup>

In another instance, MAS was employed to synthesize  $\text{Cu}_3\text{N}$  supported on graphdiyne (GDY) (Fig. 5b), where GDY served as a two-dimensional support material. TEM and AFM imaging (Fig. 5d and e) revealed the formation of thin GDY nanosheets with a diameter of approximately 15  $\mu\text{m}$  and a thickness of 4.73 nm, illustrating the structural precision achievable

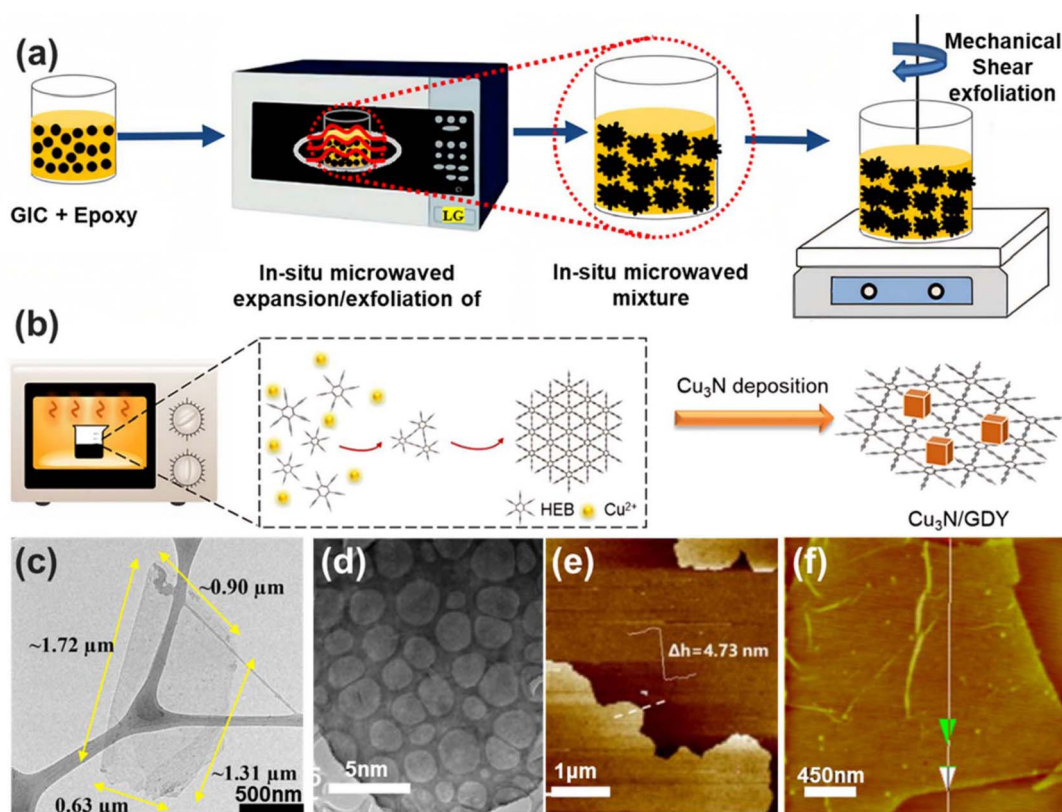


Fig. 5 (a) Schematic for preparation of epoxy/graphene nanocomposites, (b) schematic illustration of the synthesis of  $\text{Cu}_3\text{N}/\text{GDY}$ , (c) TEM images of graphene platelets, (d) TEM and (e) AFM images of GDY, and (f) typical AFM height images of MW-rGO. Reproduced with permission from ref. 160. Copyright, 2024, RSC.



Table 2 Microwave-assisted fabrication of nanocomposites and their application

Nanocomposites	Application	Reference
Co <sub>3</sub> O <sub>4</sub> -graphene sheet	Lithium-ion batteries	89
ZnO/MWCNT	Alcohol-sensing	97
rGO-ZnO/CuO	Photocatalytic degradation	165
MoS <sub>2</sub> /graphene	Hydrodesulfurization	166
Graphene nanosheets-gold	Electrochemical response	167
Cellulose/AgCl	Antimicrobial	91
NiCoFe <sub>2</sub> O <sub>4</sub> anchored polymer	Asymmetric supercapacitor	64
Fe <sub>2</sub> O <sub>3</sub> /Ag	Photocatalyst, antibacterial and anticancer	168
Cellulose/calcium silicate	Biomass application	169
Graphene nanosheets-zinc sulphide	Optical and photocatalytic	19
Carbon-coated Si nanocomposites	Anode for lithium-ion batteries	170
Ag/ZnO/graphene	Photocatalytic activity	20
Guar gum grafted sodium acrylate/cloisite	Absorbent	171
N-rich carbon quantum dots/dual-phase TiO <sub>2</sub>	CO <sub>2</sub> photoreduction	115
Pt/graphene	Hydrogen peroxide sensor	172
CuO/Cu <sub>2</sub> O-ZnO mixed metal oxide	Antibacterial and photocatalytic degradation	173
rGO/titania	Adsorbent	174
SnO <sub>2</sub> /ZnO	Photocatalytic, antimicrobial and electrochemical	175
MnCo <sub>2</sub> O <sub>4</sub> /Co <sub>3</sub> O <sub>4</sub>	Photocatalytic	176
Cellulose-silver	Antimicrobial	177
Indium sulfide/indium oxide/gold	Photocatalytic hydrogen generation	178
NiCo <sub>2</sub> O <sub>4</sub> -graphene oxide	Supercapacitors	179
Cellulose/zinc-sulfate-calcium-phosphate (ZSCAP)	Biomedical applications	180
Cobalt-polyoxometalate@carbon black	Oxygen reduction reaction	158
Graphene/CoMoO <sub>4</sub>	Supercapacitors	102
Perovskite hydroxide-derived Co <sub>3</sub> O <sub>4</sub> /SnO <sub>2</sub> /reduced graphene oxide	Hybrid supercapacitor devices	181
SnO <sub>2</sub> -graphite	Li-ion batteries	182
Polyimides chemically cross-linked with functionalized carbon nanotubes	Aerospace applications	183
BiOBr/RGO	Photocatalytic activity	83
Ni@NSiC	Electrocatalysis of glucose	184
AgGO	Antibacterial	185
Poly( <i>ortho</i> -phenylenediamine-co-aniline) and functionalised carbon nanotubes	Supercapacitors	186
Graphene oxide	Remediation of toxic metal ions	187
CuO/MnO <sub>2</sub>	Supercapacitors	5
BiNbO <sub>4</sub> and silver	Photocatalytic	45
Carbon nitride/CuO	Catalytic activity	188
Graphene-SnO <sub>2</sub>	Gas sensors	90
Mn <sub>3</sub> O <sub>4</sub> nanoparticles@reduced graphene oxide	Supercapacitors	17
BiOBr/graphene	Photocatalytic activity	189
In <sub>2</sub> S <sub>3</sub> /mesoporous TiO <sub>2</sub>	Photocatalytic H <sub>2</sub> evolution	190
Co-Fe <sub>2</sub> O <sub>3</sub> @NiO core-shell	Photoelectrochemical	191
CdS/TiO <sub>2</sub>	Larvicidal activity	192
ZnO nanoparticle/poly(vinyl alcohol)	Antimicrobial activity	193
Sb <sub>2</sub> O <sub>3</sub> -Ag	Catalytic degradation of <i>p</i> -nitrophenol	101
MoS <sub>2</sub> /PANI	Electrochemical	94
MWCNT/polystyrene	Dielectric performance	194
Ag-ZnFe <sub>2</sub> O <sub>4</sub> @rGO	Removal of organic dyes	85
NiAl-LDH/rGO	Electrodes	195
Fe <sub>2</sub> O <sub>3</sub> /g-C <sub>3</sub> N <sub>4</sub>	Photocatalytic activity	196
CoFe <sub>2</sub> O <sub>4</sub> @TiO <sub>2</sub> @rGO	Sonophotocatalytic degradation of tetracycline	197
PANI-SiO <sub>2</sub>	1,4-Dioxane sensor	198
MgFe <sub>2</sub> O <sub>4</sub> -ZnO	Photocatalyst	87
g-C <sub>3</sub> N <sub>4</sub> /SnO <sub>2</sub>	Solar water-splitting	96
Titanium dioxide decorated graphene	Photodegradation of organic dyes	86
Ag/ZnO-TiO <sub>2</sub>	Photocatalytic degradation of rhodamine B	199
Chitosan/ZnO	Dye removal	6
Graphene/polypyrrole	Supercapacitors	161
Gadolinium/cerium oxide	High-performance supercapacitors	98
CuO/TiO <sub>2</sub>	Humidity sensor application	200
TiO <sub>2</sub> /graphene	Photocatalytic activity for methylene blue degradation	201
Silver nanoparticles and chitosan	Removal of reactive blue-19	202



through microwave processing.<sup>160</sup> These nanocomposites demonstrate enhanced surface area and nanoscale features, making them promising candidates for catalytic and energy storage applications.

Microwave-assisted synthesised polymer nanocomposites, like graphene/polypyrrole,<sup>161</sup> PTFE/C,<sup>162</sup> NiCoFe<sub>2</sub>O<sub>4</sub> anchored polymer,<sup>64</sup> hemin-graphene/poly(3,4-ethylene-dioxythiophene)<sup>163</sup> and poly( $\epsilon$ -caprolactone)/montmorillonite,<sup>164</sup> have improved conductivity, mechanical strength, and antimicrobial activity, which makes them valuable for flexible electronics and biomedical applications. The following Table 2 summarizes the different nanocomposites derived *via* microwave irradiation and their application.

The rapid advancement of MAS in fabricating multifunctional nanocomposites underscores its role as a powerful tool in sustainable material design. However, much of the current literature tends to present MAS-derived composites as universally superior without fully interrogating the scalability, reproducibility, or long-term stability of these systems. While MAS can yield nanocomposites with enhanced electronic, mechanical, or catalytic properties, there is often limited discussion on how these improvements quantitatively compare to composites synthesized *via* conventional routes. Furthermore, the diverse range of materials (*e.g.*, polymers, metal oxides, and carbon-based supports) and hybrid structures reported, though impressive, lacks standardization in synthesis conditions and performance benchmarking, making it difficult to assess their broader applicability. The role of interfacial interactions, dispersion uniformity, and matrix compatibility in determining composite performance is also underexplored, especially when natural or bio-derived materials are used. A more critical and comparative analysis of reaction parameters, material

interfaces, and environmental impact, particularly for composites intended for biomedical, energy, or environmental applications, is essential for establishing MAS as a truly sustainable nanocomposite fabrication strategy.

## 4 Comparative analysis with other sustainable methods

To critically evaluate the position of MAS within the broader landscape of sustainable synthesis, it is essential to compare its performance with other established sustainable methods including sonication, electrochemical synthesis, and enzymatic approaches. Table 3 summarizes a comparative analysis of different sustainable methods available for the synthesis of nanomaterials. MAS operates through dielectric heating mechanisms that provide rapid and uniform heating, typically achieving superior reaction rates with most reactions completing within minutes to hours.<sup>12</sup> In contrast, sonochemical methods rely on acoustic cavitation to create localized high-energy zones, often requiring longer time frames but excel in operational simplicity and cost-effectiveness.<sup>203</sup> While MAS demonstrates significant advantages through its selective heating mechanism and broad substrate compatibility, sonication offers better scalability for industrial applications through continuous flow systems that can be more readily implemented than large-scale microwave reactors. Electrochemical synthesis presents a fundamentally different sustainable approach through electron transfer mechanisms, offering precise control over reaction conditions that MAS cannot match.<sup>204</sup> While MAS provides rapid heating and acceleration of thermal processes, electrochemical methods enable selective redox transformations under mild conditions without chemical

**Table 3** Summary of different sustainable methods applicable for nanomaterial fabrication

Parameter	Microwave-assisted	Sonication/ultrasonic	Electrochemical	Enzymatic
Activation mechanism	Dielectric heating through electromagnetic radiation	Acoustic cavitation through ultrasonic waves (>20 kHz)	Electron transfer through electrical current	Biocatalytic conversion through enzymes
Reaction time	Significantly reduced (minutes to hours)	Shortened compared to conventional methods	Variable, depends on the current density	Generally longer but mild conditions
Energy efficiency	High efficiency with selective heating	Moderate to high efficiency	High efficiency, electricity-driven	Very high efficiency under mild conditions
Reaction temperature	Mild to moderate temperatures	Ambient to moderate temperatures	Ambient to moderate temperatures	Ambient to mild temperatures (30–80 °C)
Selectivity	High selectivity and yields	Enhanced selectivity through cavitation	High selectivity with controlled potential	Excellent selectivity and stereoselectivity
Environmental impact	Green method, reduced solvent use	Environmentally friendly, minimal waste	Green alternative when using renewable electricity	Highly sustainable, biodegradable catalysts
Scale-up potential	Moderate to good scalability	Good scalability	Excellent scalability	Limited scalability, cost considerations
Equipment cost	Moderate initial investment	Low to moderate cost	Moderate to high cost	Low equipment cost, high enzyme cost
Solvent requirements	Reduced solvent use, water-compatible	Compatible with various solvents	Often aqueous or low-toxicity solvents	Aqueous systems preferred
Product purity	High purity, fewer side products	Good purity with controlled conditions	High purity with selective reactions	Excellent purity, minimal purification
Substrate scope	Wide range of substrates	Broad substrate compatibility	Limited by electrochemical properties	Limited by enzyme specificity
Operational simplicity	Simple operation	Simple operation	Moderate complexity	Moderate complexity



oxidants or reductants, achieving high atom economy and eliminating chemical waste. However, electrochemical synthesis faces limitations in substrate scope, being restricted to compounds with suitable electrochemical properties, whereas MAS demonstrates broader applicability across various chemical transformations. The scalability of electrochemical methods often surpasses MAS for industrial applications, though MAS maintains advantages in reaction versatility and processing speed across diverse chemical classes.

Enzymatic synthesis represents the pinnacle of selectivity in sustainable chemistry, operating under the mildest conditions with exceptional stereoselectivity and minimal side reactions using biodegradable, renewable catalysts.<sup>205</sup> While MAS achieves rapid reaction rates through thermal activation, enzymatic methods accomplish transformations at ambient temperatures with precision that MAS may struggle to achieve under rapid heating conditions. The sustainability comparison heavily favors enzymatic methods due to their mild aqueous conditions and renewable nature,<sup>206</sup> though MAS demonstrates significant advantages in reaction speed and substrate versatility. The cost analysis reveals complexity: MAS requires moderate initial equipment investment, while enzymatic methods face ongoing costs related to enzyme procurement and replacement, with substrate scope limitations restricted by specific enzyme–substrate interactions, contrasting sharply with MAS's broad applicability.

The comparative analysis reveals that no single sustainable method dominates across all parameters, with MAS excelling in rapid processing and broad substrate compatibility<sup>207</sup> while facing limitations in uniform heating for large-scale reactions and requirements for microwave-transparent materials. The integration of multiple sustainable methods presents promising opportunities, with hybrid approaches combining MAS with sonication for enhanced mixing or with enzymatic steps for improved selectivity, potentially addressing individual method limitations. The critical evaluation indicates that MAS represents a significant advancement in green chemistry, but its position within the sustainable synthesis landscape is complementary rather than dominant, with true value emerging when applied appropriately to reactions where rapid heating and broad substrate compatibility are priorities, while recognizing situations where alternative sustainable methods may provide superior performance.

## 5 Green metrics and sustainability assessment

In alignment with the green chemistry principles, a set of rules meant to support environmentally friendly and financially viable chemical operations, MAS provides a cleaner, more sustainable pathway for the synthesis of nanomaterials by

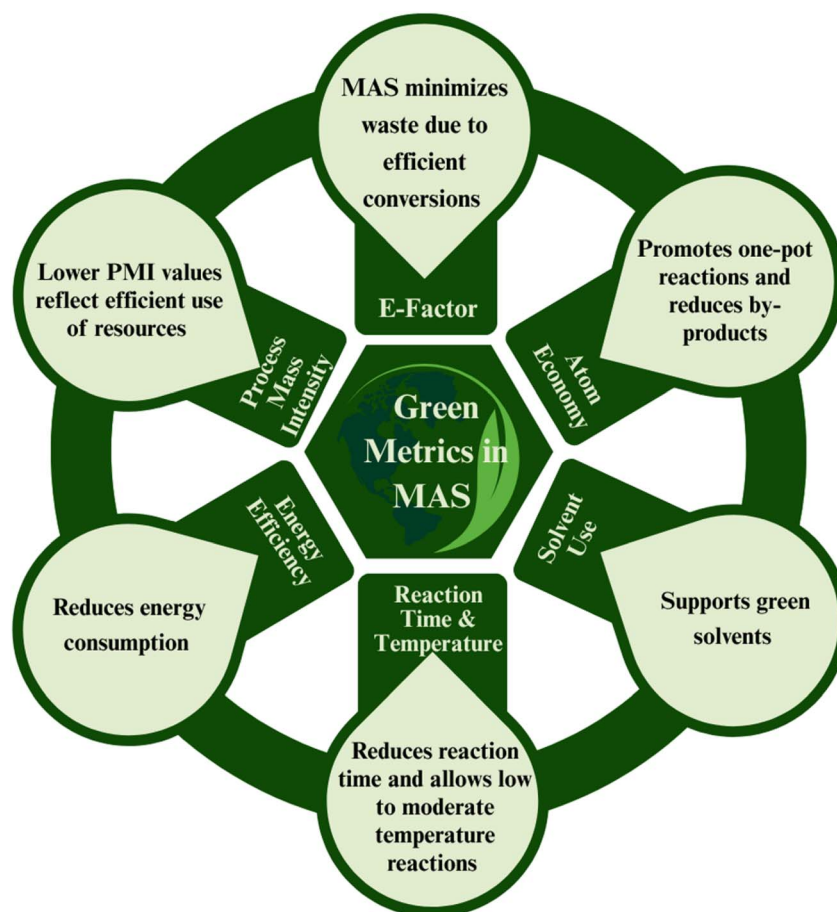


Fig. 6 Green metrics involved in MAS.





minimizing waste, lowering reaction temperatures, and enhancing atom economy.<sup>13,15,208</sup> Prevention of waste: employing improved control over reaction parameters in MAS minimizes the generation of undesired by-products, thus lowering waste at the source. Atom economy: a crucial component of atom-economical processes, MAS frequently encourages high-yield reactions with fewer steps, optimizing the incorporation of all starting materials into the finished product.<sup>207</sup> Energy efficiency: MAS usually requires lower temperatures and shorter durations, resulting in significant energy savings, because of the quick internal heating made possible by microwaves.<sup>34</sup> Use of safer solvents and auxiliaries: to promote safer laboratory and industrial procedures, many MAS protocols make use of safe solvents like ethanol or water, and in certain situations, they completely do away with the need for solvents.<sup>52</sup> Design for degradation: MAS makes it easier to synthesize materials that are more environmentally friendly throughout their life cycle when combined with biodegradable precursors like plant extracts or renewable feedstocks.<sup>145</sup> MAS is a sustainable chemical manufacturing model in addition to being an effective synthetic tool. It has the potential to address urgent global issues such as energy consumption, pollution, and sustainable development in the field of nanomaterials because it is in line with important green chemistry principles.

To evaluate the environmental and process efficiency of MAS methods, green metrics serve as critical tools.<sup>209,210</sup> Numerous quantitative green metrics have been developed to objectively evaluate MAS's environmental sustainability. These metrics are used to assess how well a chemical process conforms to the green chemistry principles, especially concerning energy consumption, waste generation, and resource efficiency.<sup>211</sup> The benefits of MAS over traditional synthesis methods are demonstrated by evaluating it using these standardized parameters.

Fig. 6 illustrates a schematic overview of the key green metrics, highlighting their role in quantifying the eco-friendliness of MAS processes. These metrics help in comparing MAS with conventional synthetic strategies and

support the optimization of greener routes. Table 4 summarizes these green metrics, providing definitions and the specific advantages offered by MAS over conventional methods. It demonstrates how MAS routes often achieve lower waste generation, higher material utilization, and superior energy performance, thereby supporting sustainable development in nanomaterials synthesis.

### 5.1 E-factor (environmental factor)

The E-factor is a widely accepted metric used to quantify the amount of waste generated per unit of product.<sup>210</sup> It is calculated using the formula:

$$\text{E-factor} = \frac{\text{total waste generated}}{\text{amount of product obtained}}$$

The E-factor in MAS is substantially lower than that in traditional techniques.<sup>211</sup> This is mostly because fewer solvents are used, fewer side products are produced, and the reaction pathways are shorter.<sup>212</sup> For example, the microwave approach showed an E-factor reduction of more than 60% in comparison to conventional thermal methods in a comparative study involving the synthesis of ZnO nanoparticles.<sup>213</sup> A smaller environmental impact and streamlined post-synthesis purification procedures are the direct results of this reduction.

### 5.2 Atom economy (%)

Atom economy is a concept describing how much of the reactants are used for the final product and is given by,<sup>210</sup>

$$\text{Atom economy (\%)} = \frac{\text{molecular weight of desired product}}{\text{total molecular weight of all reactants}} \times 100$$

MAS usually promotes high atom economy as most reactions are conducted in one-pot and solvent-free, minimizing excess

**Table 4** Summary of green metrics and their relevance in MAS

Green metric	Relevance in MAS	Example/observation
E-factor (environmental factor)	MAS minimizes waste due to efficient conversions and minimal use of solvents	Carbon nitride-supported silver nanoparticles: E-factor is 0.27 (ref. 211)
Atom economy (%)	Promotes one-pot reactions and reduces the formation of by-products	MAS often results in higher atom economy due to fewer side reactions. Example: CQDs from fenugreek seeds <sup>127</sup>
Process mass intensity (PMI)	Lower PMI values reflect efficient use of resources and reduced waste	MAS offers lower PMI due to shorter reaction and minimal solvent use
Energy efficiency	MAS significantly reduces energy consumption due to volumetric heating and shorter reaction times	MAS of TiO <sub>2</sub> showed ~40% lower energy consumption vs. the sol-gel method
Reaction time	MAS reduces reaction time from hours to minutes in most cases	Metal nanoparticle synthesis is often completed within 5–30 minutes under MAS <sup>15</sup>
Solvent use	MAS supports green solvents like water and ethanol, or even solvent-free synthesis	Silver and gold nanocatalysts from <i>Aerva lanata</i> extract under MAS with water as the solvent <sup>55</sup>
Temperature profile	MAS allows low to moderate temperature reactions with precise control, avoiding thermal degradation	<i>Hylocereus costaricensis</i> -mediated Ag/ZnO-NPs synthesis was performed under mild MAS conditions <sup>75</sup>



reagent and the generation of unwanted by-products.<sup>212</sup> Thus, a higher ratio of starting materials becomes part of the end nanomaterials, both improving sustainability and economic efficiency in the process.

### 5.3 Process mass intensity (PMI)

Process mass intensity (PMI) is another valuable metric used to determine the material efficacy of a synthesis route and is given by the relation,<sup>210</sup>

$$\text{PMI} = \frac{\text{total mass of all inputs}}{\text{mass of isolated product}}$$

MAS typically produces a lower PMI than traditional methods. This is attributable to the low solvent volume, short reaction time, and efficient heating methods that enable quicker and more complete reactions.<sup>34</sup> An effective PMI value demonstrates a tight process with minimal waste of materials, which can help develop nanomaterials more sustainably on a large scale.

### 5.4 Energy efficiency

The most noteworthy benefit of MAS is its improved energy efficiency. Microwave systems enable the fast and specific heating of reaction mixtures, minimizing the energy losses characteristic of bulk heating systems.<sup>23</sup> Interestingly, MAS reportedly consumes 80% less energy compared to conventional hydrothermal synthesis.<sup>40</sup> This is mainly because the energy delivered is directly coupled to the reactants, as opposed to heating the entirety of the vessel or the surrounding medium. Additionally, one major factor that keeps MAS as a pioneering energy-efficient synthesis method is the accurate control of microwave power, preventing overheating and wastage of energy.<sup>22</sup>

### 5.5 Solvent and reagent aspects

One of the main advantages of MAS is that it can easily be used with green solvents and natural reagents, which means that MAS is itself an inherently sustainable method.<sup>212</sup> Compared with traditional synthesis processes that typically require toxic solvents or harmful surfactants, MAS allows the use of eco-friendly and renewable materials without loss of process efficiency or product quality.<sup>46,128,169</sup> Water is likely the most common solvent amongst all solvents used for MAS because of its high dielectric constant, which enables it to interact strongly with microwave radiation.<sup>214</sup> This allows the microwave energy to be absorbed very effectively, allowing for very rapid and uniform heating. In addition, water is non-toxic, non-flammable, easily obtainable, and compatible with many biological precursors, making it an ideal medium for eco-friendly synthesis.<sup>215,216</sup> MAS in aqueous media has proven to be an effective strategy for producing nanocrystalline three-dimensional metal oxides and gold nanoparticles.<sup>217</sup> This approach not only enhances the structural and morphological control of the materials but also improves their functional performance in various applications.

Organic salts known as ionic liquids have low melting points and exhibit novel solvent and thermal stability properties.<sup>218</sup> One area where MAS using ionic liquids as reaction media has several advantages is that it improves the solubility of reactants, improves mass transfer and reduces energy consumption.<sup>219</sup> Additionally, ionic liquids can act as a solvent and stabilizing agent, and no additional toxic solvents or surfactants are necessary. Nanoparticles such as MgO,<sup>46</sup> ZnO,<sup>220</sup> ZnS quantum dots<sup>221</sup> and carbon dots<sup>222</sup> have been prepared *via* this approach in the presence of the respective ionic liquids, showing enhanced applications in various fields. The green nanomaterial synthesis using MAS in ionic liquids has been an attractive approach because ionic liquids can produce nanomaterials with tailored properties and high purity.<sup>218,219</sup>

Another frequently employed green solvent in MAS is ethanol, which has moderate polarity and a relatively high boiling point.<sup>223</sup> Being renewable and biodegradable, ethanol from biomass is consistent with the green chemistry principles. In particular, the solvating power of the ionic liquids toward various kinds of metal precursors and the capability to work with various plant-derived extracts make them suitable for the nanomaterial or nanocomposite synthesis.<sup>217</sup>

An exciting advance in the field of sustainable nanomaterial synthesis, including MAS is using plant extracts, which act as reducing agents, stabilizers, and capping agents.<sup>224</sup> The extracts are bioactive in nature and contain species like flavonoids, terpenoids, tannins, polyphenols and alkaloids.<sup>75</sup> They serve multiple functions, so there is no need to use toxic reagents and synthetic surfactants that are used to manipulate nanoparticle morphology and stability. Apart from environmental safety, the incorporation of plant extracts as reagents in MAS often endows the synthesized nanomaterials with additional functional properties. Examples include increasing the antioxidant activity of the nanoparticle surface by using phytochemical residues, improving biological interactions and enhancing their dispersion stability in aqueous systems.<sup>41</sup> This paves the way for applications in biomedicine, food packaging and environmental remediation. The thoughtful selection of solvents and reagents in MAS contributes significantly to making the process more sustainable. By incorporating renewable, non-toxic, and multifunctional materials, MAS not only reduces the environmental impact but also improves the performance and applicability of the resulting nanomaterials.

### 5.6 Lifecycle assessment (LCA) and environmental impact

Lifecycle assessment (LCA) is an analytical approach used to determine the environmental impacts of a process, product, or technology from beginning to end-including raw material sourcing, production, usage, and end-of-life treatment.<sup>225</sup> In MAS, LCA is valuable for measuring factors such as energy usage, emissions, waste output, and overall resource efficiency.<sup>226</sup> The assessment typically involves four key stages:<sup>226</sup> (i) defining the goal and scope, which sets the purpose and boundaries of the analysis; (ii) inventory analysis, where input and output data (like materials, energy, and waste) are gathered; (iii) impact assessment, in which environmental consequences



such as the carbon footprint or toxicity are evaluated; and (iv) interpretation, where findings are analyzed to identify sustainable improvements and guide greener decision-making. Software tools such as SimaPro,<sup>225</sup> GaBi,<sup>227</sup> and OpenLCA,<sup>228,229</sup> allow for detailed modelling, data analysis, and impact assessment based on international standards. These tools help ensure standardized, transparent, and repeatable environmental assessments aligned with ISO 14040 and ISO 14044 guidelines.<sup>230</sup> These tools enable users to evaluate the environmental impacts of products or processes throughout their life cycle.

LCA of ZnO nanostructures synthesized using a MAS technique with varying microwave power (110 W to 710 W) and annealing temperature (90 °C to 220 °C) revealed that increasing microwave power or synthesis temperature led to enhanced material output with lower environmental impact, and the footprint reduced by 27% (power) and 41% (temperature).<sup>231</sup> Silver nanoparticles (AgNPs), synthesized using MAS with glucose as the reducing agent and food-grade corn starch as the stabilizer, were evaluated using LCA in GaBi 6.0 software.<sup>227</sup> The analysis revealed that the primary environmental burdens arose from silver metal production and electricity consumption. In contrast, glucose and starch contributed negligible impacts, affirming the environmental benefits of using biogenic, food-safe reagents.

A LCA study compared the environmental impacts of six bottom-up synthesis methods for carbon dots (CDs), focusing on hydrothermal and MAS using citric acid and urea.<sup>232</sup> The results revealed that electricity use dominated the impacts in hydrothermal methods, while citric acid was the major contributor in MAS. When adjusted for quantum yield, MAS using both citric acid and urea emerged as a more sustainable option for high-performance CD production.<sup>232</sup> Fernandes *et al.* conducted a LCA study comparing high-yield and standard bottom-up methods for fabricating carbon dots, using MAS.<sup>233</sup> Their findings revealed that certain standard methods, notably MAS, exhibited lower environmental impacts compared to some high-yield conventional techniques. These studies suggest that MAS offers a more sustainable approach for carbon dot production.

Nanocomposites of cerium oxide, copper oxide, and zinc oxide (CeO<sub>2</sub>-CuO-ZnO; CeCZ) were developed through MAS across a range of 300 to 800 W.<sup>234</sup> An LCA based on power consumption indicated that utilizing lower microwave energy notably decreases greenhouse gas emissions and lessens the environmental footprint. The findings further suggest that incorporating renewable energy into the process can enhance its sustainability. This study demonstrates the effectiveness of MAS as a green and energy-saving technique for creating advanced photocatalysts aimed at environmental cleanup. A review of J. Munuera *et al.* explores multiple sustainable approaches for graphene synthesis, with a particular focus on MAS due to its energy efficiency and lower environmental burden compared to conventional methods.<sup>228</sup> It underscores the role of LCA in evaluating the ecological impacts of these production techniques, offering a comprehensive view of their sustainability.

LCA indicates significant environmental benefits of MAS over conventional synthesis methods. Moreover, MAS processes are often solvent-free or use minimal solvents, resulting in reduced

wastewater generation and a lower burden of effluent treatment. Furthermore, in closed-vessel microwave systems, chemicals are contained, and the risk to the user from chemical exposure and release of volatile organic compounds (VOCs) is reduced.<sup>218</sup> Thus, MAS is efficient, clean, and meets the regulatory mandates for safe and environmentally friendly manufacturing processes.

## 5.7 Circular chemistry and resource efficiency

MAS aligns well with the tenets of circular chemistry, a framework that seeks to limit the use of resources, waste, and use waste as feedstock for useful products.<sup>209</sup> MAS advocates for this model in multiple ways. MAS can efficiently utilize plant-derived precursors, such as fruit peels,<sup>81</sup> leaf extracts,<sup>26</sup> and agricultural waste.<sup>235</sup> These biomaterials, which are usually high in reducing agents and stabilizing agents, are good substitutes for toxic chemical reagents. MAS enables the transformation of biomass waste into valuable nanomaterials.<sup>18,121,236</sup> Various metal and metal oxide nanoparticles particularly silver,<sup>237</sup> gold,<sup>238</sup> zinc oxide,<sup>75</sup> and copper oxide,<sup>239</sup> have been synthesized using plant extracts of *Aloe vera*, *Moringa oleifera*, and *Azadirachta indica* under microwave irradiation, and exhibit astounding antibacterial, catalytic, and optical activities. Some other materials, which can potentially become agro-wastes, are not only collected but also converted into functional materials, e.g., carbon quantum dots (CQDs) and doped metal oxides have been successfully synthesized from sugarcane bagasse,<sup>113</sup> citrus peels,<sup>108</sup> and other biogenic residues.

The reduced use of organic solvents and progress of reactions contained in sealed vessels lowers emissions and avoids cross-contamination, thus contributing to the novel trend of circular economy, and green engineering principles.<sup>15,214</sup> The industrial capacity of MAS is becoming more and more understood, particularly due to its capacity to manufacture high-quality nanomaterials at a low environmental cost. Case studies show the scalability of MAS and its implementation in sustainable manufacturing. Carbon nanospheres, synthesised using banana peel extract under microwave conditions, were applied for selective and specific sensing of Cu<sup>2+</sup> ions and the amino acid tryptophan in aqueous solutions.<sup>240</sup> The use of agricultural waste as a precursor and a capping agent eliminated toxic chemicals. Photoluminescent carbon dots obtained from palm frond biomass wastes *via* MAS was demonstrated to be an exceptional sensor for lead(II) ions.<sup>236</sup> Such examples reinforce the relevance of MAS not only in laboratory-scale research but also in industrial applications where eco-efficiency, performance, and cost-effectiveness are equally critical. The adoption of MAS supports the development of nanotechnologies that are not just advanced in function, but also responsible in practice-fostering a future where innovation and environmental stewardship go hand in hand.

## 6 Applications of microwave-assisted nanomaterials

Due to their unique properties at the nanoscale, nanomaterials have gained considerable interest in multiple fields due to their



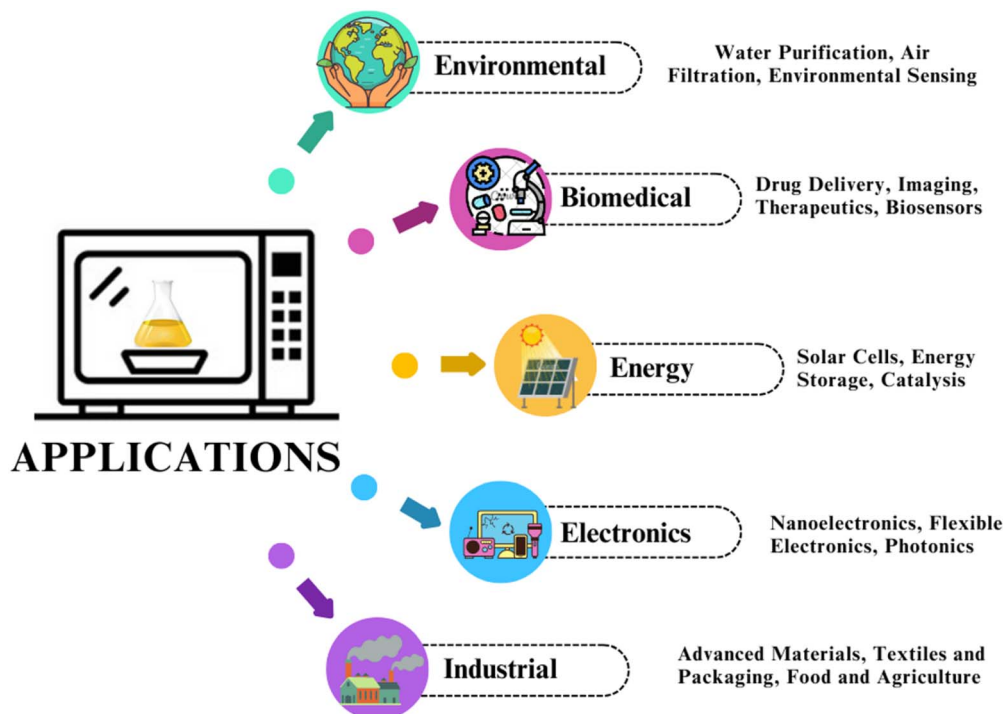


Fig. 7 Applications of microwave-assisted fabricated nanomaterials.

potential to transform industries and address key societal challenges. The advantages of MAS include rapid synthesis kinetics, scalability and ability to produce nanomaterials with high purity and uniformity for electronics, catalysis and biomedical engineering applications (Fig. 7). These nanomaterials are proved to be versatile solutions, possessing improved performance and new functionalities for biomedical applications, environmental remediation and energy storage.<sup>26</sup> Moreover, they are important in the drive towards electronics, optoelectronics, industrial applications of miniaturization, better power efficiency and multifunctionality.

### 6.1 Biomedical applications

Because of their unique and versatile properties, nanomaterials have revolutionized biomedical research and healthcare.<sup>2</sup> Some key applications include drug delivery, imaging, therapeutics and biosensors. Encapsulating drugs, peptides or nucleic acids in nanoparticles enables the targeting of these to specific tissues or cells.<sup>79</sup> Controlled release and enhanced therapeutic efficacy are realized by surface modification to minimize side effects.<sup>241</sup> As contrast agents for a variety of imaging modalities, including magnetic resonance imaging (MRI),<sup>242</sup> bio-imaging,<sup>136</sup> cell-imaging<sup>107</sup> or fluorescence imaging,<sup>114,121</sup> nanomaterials, such as quantum dots, iron oxide nanoparticles or gold nanoparticles, are used. The cellular and chemical properties of nanomaterials are being exploited to design nanosystems for use in therapeutic drug monitoring,<sup>142</sup> antibacterial therapy,<sup>157</sup> photodynamic therapy,<sup>27</sup> cancer therapy,<sup>144</sup> infections, and other diseases.<sup>79</sup> Targeted delivery systems increase treatment efficacy and decrease systemic toxicity. Biosensors based on

nanomaterials provide fast and sensitive biomolecule, pathogen, and disease marker detection for diagnosis.<sup>243</sup> Nanoparticles are functionalized with disease targets for signal transduction acceleration and sensor enhancement.<sup>135,172,198</sup>

Microwave-assisted synthesis has accelerated the development of nanomaterials tailored for biomedical applications, offering rapid, energy-efficient fabrication of highly uniform and functionalized nanoparticles. However, while MAS-generated nanomaterials have shown promise in drug delivery, imaging, therapeutics, and biosensing,<sup>12</sup> much of the existing literature tends to emphasize performance outcomes without adequately addressing long-term biocompatibility, *in vivo* stability, or toxicological implications. For instance, although quantum dots and metal-based nanoparticles synthesized *via* MAS exhibit superior imaging and therapeutic properties, concerns about their degradation, bioaccumulation, and clearance from biological systems remain largely underexplored. Moreover, the surface functionalization of nanoparticles, while improving targeting and controlled release, is often not correlated with reproducibility or consistency across batches, an essential factor for clinical translation. Biosensors based on MAS nanomaterials have demonstrated heightened sensitivity and specificity, yet few studies address their real-world diagnostic reliability under complex biological conditions. There is also a gap in comparing MAS-derived nanomaterials with those synthesized through traditional methods in terms of cost-effectiveness, lifecycle analysis, and regulatory compliance for medical use. Therefore, a critical focus on bridging the gap between laboratory performance and clinical feasibility, backed by standardized testing protocols, is





necessary to validate MAS as a sustainable and scalable route for biomedical nanomaterial development.

## 6.2 Environmental remediation

Utilizing nanomaterials makes for innovative solutions to environmental remediation and pollution control through pollutant removal, detection and monitoring.<sup>2</sup> Key applications include water purification, air filtration and environmental sensing.<sup>126,145,238</sup> For the removal of heavy metals,<sup>113,244</sup> organic pollutants,<sup>80,107</sup> and pathogens from water sources, nanomaterials, like graphene oxide, carbon nanotubes, and metal oxide nanoparticles, are employed in adsorption, filtration and photocatalysis.<sup>1</sup> Particulate matter, volatile organic compounds, and harmful gases are captured from indoor and outdoor air by nanomaterial-based filters and membranes that improve indoor and outdoor air quality and decrease health risks. Real-time sensors and detectors based on nanomaterials make it possible to monitor pollutants, greenhouse gases, and environmental contaminants in air, water and soil for early warning systems and to develop environmental management strategies.<sup>145,148</sup>

Microwave-assisted synthesis of nanomaterials for environmental remediation holds immense promise due to the ability to rapidly produce high-surface-area materials with tailored physicochemical properties for pollutant capture and degradation. However, despite the growing number of studies highlighting their efficacy in water purification, air filtration, and environmental sensing, many claims remain largely performance-based, lacking comprehensive evaluation of environmental safety, reusability, and post-treatment disposal. While materials like graphene oxide, carbon nanotubes, and metal oxides synthesized *via* MAS demonstrate efficient removal of heavy metals and organic pollutants, there is insufficient focus on their regeneration potential and cost-effectiveness in real-world applications. Future work should critically address the long-term sustainability, risk assessment, and policy implications of deploying MAS-derived nanomaterials in environmental technologies.

## 6.3 Energy applications

Renewable energy technologies require and benefit from the use of nanomaterials to advance their impact. Major applications include solar cells, energy storage, and catalysis.<sup>12</sup> Next generation solar cells use nanomaterials like quantum dots, perovskites, and nanowires, which increase light absorption, charge transport, and device performance capabilities of solar cells.<sup>3,236,245,246</sup> Also, nanostructured materials permit the fabrication of lightweight, flexible, and low-cost photovoltaic devices.<sup>14</sup> To improve energy density, cycling stability and charge-discharge rates, nanomaterials, including carbon nanotubes, graphene and metal oxides are used in lithium ion batteries, supercapacitors, and fuel cells.<sup>12,247</sup> This leads to energy storage and electrochemical performance enhancement of the electrodes and electrolytes *via* nanostructuring.<sup>64,181</sup> Because of their efficiency, nanomaterials are indeed excellent catalysts to conduct chemical reactions in energy conversion

and energy storage processes, including hydrogen production, synthesis of fuels, and pollutant degradation.<sup>14,82,101,248</sup> Enriched nanostructures and active sites enhance the catalytic activity and selectivity.

While MAS-derived nanomaterials show promise in improving light absorption and energy storage performance, real-world testing under variable conditions and lifecycle assessments are rarely addressed. Furthermore, the long-term stability and degradation behavior of these materials in operational environments remain critical gaps that hinder their commercialization. A more application-oriented and techno-economic evaluation is needed to validate MAS as a truly transformative tool in the renewable energy sector.

## 6.4 Electronics and optoelectronics

Modern electronic devices and optoelectronic technologies are dependent on and require nanomaterials in order to facilitate miniaturization, high performance, and new functionalities. Major applications include nanoelectronics, flexible electronics, and photonics.<sup>3</sup> To achieve high speed operation, low power consumption and improved electronic properties, MAS based nanomaterials, such as carbon nanotubes, graphene and semiconductor nanoparticles, are used in transistors, supercapacitors, sensors and memory devices.<sup>2</sup> The development of flexible displays, wearable sensors, and electronic textiles is enabled by nanomaterial-based flexible substrates, conductive inks and transparent electrodes.<sup>98,200</sup> As lightweight, bendable and stretchable electronic devices, nanocomposites and thin-film technologies are being developed. Optical devices based on nanomaterials, such as plasmonic nanoparticles, quantum dots, and photonic crystals, are used in photonic circuits, LEDs, and display technology with applications in telecommunications and sensing.<sup>4,27,103</sup> With nanostructured materials, precise control of light-matter interactions and optical properties is achieved (Fig. 8).<sup>249</sup>

However, despite promising laboratory outcomes in flexible electronics, memory devices, and photonic applications, the practical integration of MAS-derived nanomaterials into large-scale device fabrication remains a major hurdle. Challenges such as batch-to-batch consistency, substrate compatibility, and thermal stability under operational conditions are often overlooked. Moreover, while nanostructured materials enable improved optical and electronic properties, their long-term reliability, particularly in flexible and wearable formats, is not rigorously evaluated. Future research must bridge the gap between material innovation and real-world device engineering by focusing on durability, manufacturability, and cost-benefit analyses.

## 6.5 Industrial applications

Nanomaterials are used in many industrial sectors to improve material performance, and enable new product properties and functionalities. Major applications include advanced materials, textiles, packaging, food and agriculture.<sup>2</sup> Microwave-based nanomaterials that include nanocomposites, nanocoatings and nanoporous materials are used to strengthen mechanical



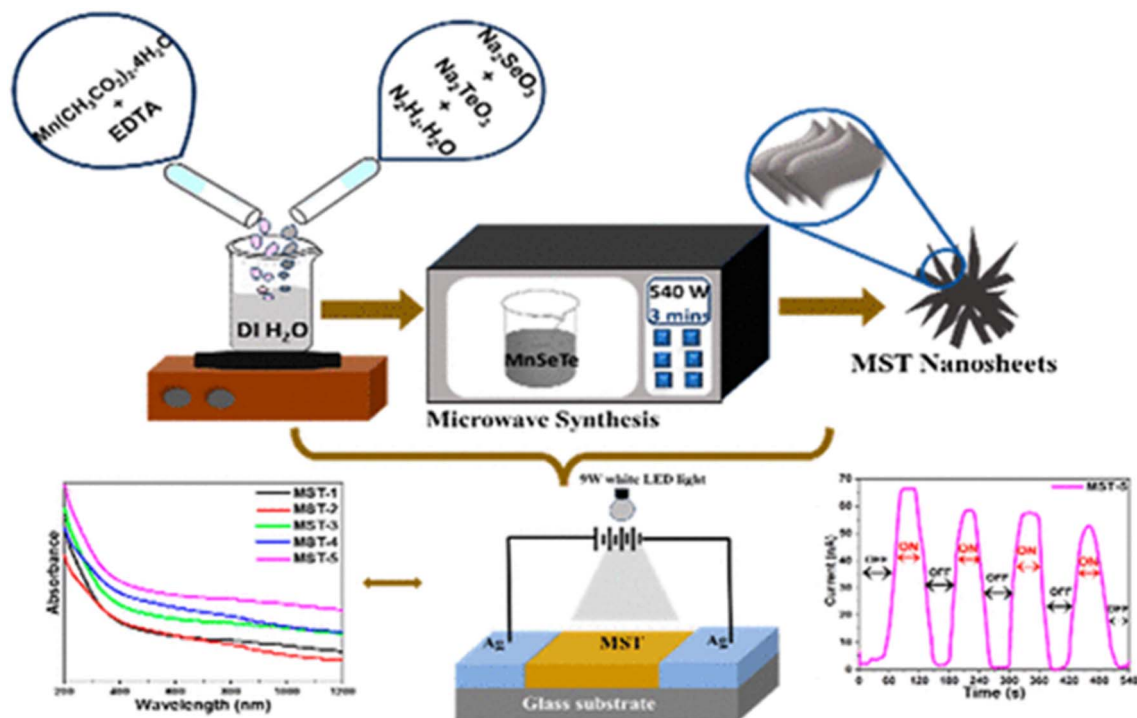


Fig. 8 Schematic representation of MAS of MnSeTe nanocomposite flowers for optoelectronic and photoresponse applications. Reproduced with permission from ref. 249. Copyright, 2024, ACS.

strength, thermal stability and chemical resistance for automotive, aerospace and construction industries.<sup>3</sup> Lightweight, durable and multifunctional materials for structural components and coatings can be developed using nanostructured materials. Textiles, clothing, and packaging materials use

coatings, films and fibers containing nanomaterials to resist stains, be antimicrobial (Fig. 9)<sup>250</sup> and to serve as barrier materials for moisture, gases and UV radiation.<sup>251</sup> Nanostructured materials increase durability, comfort, and functionality of textiles and packaging products.<sup>88,252</sup> In food

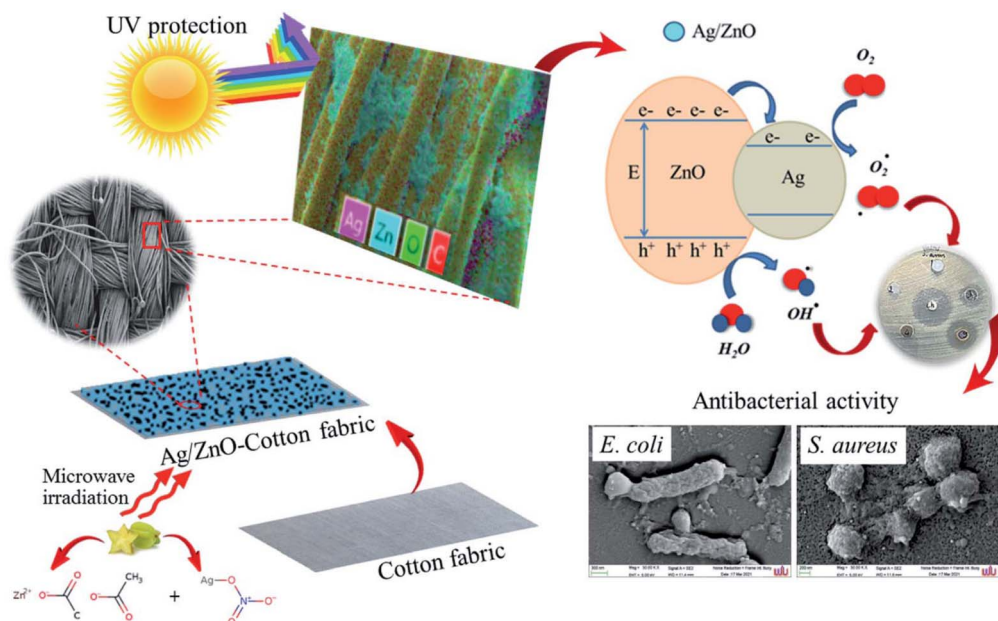


Fig. 9 Proposed mechanism of the cotton fabrics treated with the Ag/ZnO nanocomposite for antibacterial activity. Reproduced with permission from ref. 250. Copyright, 2022, RSC.



packaging, processing, and storage, MAS based nanomaterials are employed to enhance the safety, preservation, and quality of food.<sup>2,35,147</sup> Controlled release of antimicrobial agents, antioxidants and nutrients by nanoemulsions, nanocapsules, and nanosensors reduces food spoilage and contamination. In many instances, the applications in these materials represent just a fraction of the potential of nanomaterials to address societal challenges, where transformative technologies are critical to progress and to improve quality of life across numerous sectors.

While applications in textiles, packaging, and construction have leveraged MAS-derived nanocomposites and coatings for added durability, antimicrobial activity, and environmental resistance, many studies lack long-term performance evaluations under real industrial conditions. Moreover, the regulatory and safety implications of incorporating nanomaterials, particularly in food packaging and consumer goods, are often underreported or insufficiently addressed. The controlled release of bioactive agents in food systems, while conceptually valuable, still faces hurdles related to migration, degradation, and human health impacts. For MAS to truly support transformative industrial innovation, future research must critically integrate lifecycle assessment, regulatory compliance, and real-world performance data to validate both safety and sustainability claims.

## 7 Challenges and future prospects

Despite these advantages, MAS still faces several critical limitations that hinder its widespread adoption, particularly at the industrial scale. One major challenge is the difficulty in scaling up the process without compromising product uniformity or energy efficiency. In laboratory-scale setups, the small reaction volumes ensure uniform microwave absorption, but scaling to larger volumes often results in uneven heating and local hot spots, which can lead to variability in particle size, shape, and crystallinity. Furthermore, many published studies report successful synthesis under highly controlled lab conditions, but often lack discussions on reproducibility, batch-to-batch consistency, or adaptability to continuous production models. This disconnect between academic success and industrial feasibility must be addressed through engineering innovations in reactor design and process standardization.

Another limitation is the incomplete mechanistic understanding of microwave–material interactions. Unlike conventional heating, where temperature and reaction pathways are more predictable, MAS involves complex electromagnetic interactions that are not yet fully understood. This knowledge gap hinders the rational design of synthesis protocols and limits control over fine-tuned nanostructures. Moreover, while natural and bio-based precursors are widely used, their compositional variability, due to seasonality, geography, and storage conditions, poses a challenge for reproducibility. Therefore, future research should emphasize reaction mechanism elucidation, standardization of green feedstocks, and the development of robust synthesis protocols that can adapt to feedstock variability.

Looking ahead, the future of MAS depends on strategic, interdisciplinary efforts. The development of scalable, energy-efficient, and automated microwave reactors tailored for nanomaterial synthesis is crucial. Integration of artificial intelligence (AI) and machine learning (ML) tools can revolutionize MAS by enabling predictive modelling, real-time process control, and adaptive optimization based on feedback from reaction parameters. Additionally, the environmental claims of MAS need to be backed by rigorous life cycle assessments (LCA) and toxicological evaluations to ensure long-term sustainability and safety. Cross-sector collaboration between chemists, biologists, materials scientists, engineers, environmental experts, and industry leaders is essential for transitioning MAS from a promising laboratory technique to a widely adopted industrial technology.

## 8 Conclusion

Microwave-assisted synthesis (MAS) stands out as a breakthrough approach in the field of sustainable nanotechnology, offering a rapid, energy-conscious, and eco-friendly method for creating nanomaterials with controlled properties. This review has explored the core principles that govern MAS, its strong alignment with green chemistry, and its broad utility in the synthesis of metallic nanoparticles, carbon quantum dots (CQDs), and nanocomposites. The distinctive mode of microwave heating—characterized by uniform, internal, and selective energy delivery—enables faster reactions, improved yields, and reduced processing times under gentler conditions when compared to conventional techniques. A particularly noteworthy feature of MAS is its compatibility with green synthesis approaches. Utilizing natural and renewable precursors such as plant extracts, biodegradable materials, and bio-based reagents not only decreases the environmental footprint but can also impart valuable functional traits to the synthesized nanomaterials such as improved biocompatibility, catalytic activity, or targeted responsiveness. The materials produced through MAS have demonstrated great potential across various fields, including catalysis, drug delivery, environmental remediation, biosensing, and renewable energy systems. Considering its sustainable and eco-friendly properties, MAS has a considerable advantage over other sustainable methods and conventional methods, and is capable of driving the future of sustainable nanomaterial manufacturing.

## Author contributions

Sajini T.: conceptualization, writing – original draft, supervision. Jebin Joseph: literature review, writing – review and editing.

## Conflicts of interest

The authors declare no conflict of interest in this paper.



## Data availability

The data supporting the findings of this study are available from the corresponding author upon reasonable request.

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