REVIEW

Nickel oxide nanoparticle synthesis and photocatalytic applications: evolution from conventional methods to novel microfuidic approaches

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Abstract

Nickel oxide (NiO) nanoparticles are essential to developing a wide range of important industrial products, examples of which include electrodes, catalysts, and sensors, leading to diverse applications from electrochemical detection to energy storage and environmental remediation. NiO nanoparticles exhibit higher reaction selectivity under solar-driven conditions. Thus, they are good candidates for photocatalysts, which can generate strong oxidizing and reducing agents for photodegradation of organic pollutants and other target molecules under normal temperature and pressure conditions, giving rise to versatile applications for energy and environmental remediation. The conventional strategies of NiO nanoparticle synthesis can be broadly categorized into three themes: solid-phase method, liquid-phase method, and vapor-phase method. Recently, microfuidic reactors hold great promise for nanomaterial synthesis due to the thermal homogeneity across the reactor and rapid heat transfer ensured by the large ratio of surface area to volume. The exquisite control over the size, structure and composition of the droplet by microfuidic emulsifcation technology outperforms the traditional microemulsion method. Herein, we present an overview of the latest advances in fabrication of NiO nanoparticles using diferent approaches including both conventional methods and microfuidic methods, and focus on the fundamentals of each formation process with the main advantages and disadvantages discussed. This review also provides comparative overview of infuence of synthesis conditions on size and morphologies of NiO nanoparticles. We also summarized the development of NiO-based photocatalysts in environmental applications. The perspectives for future research are also discussed. It can be envisioned that success in microfuidic method will continue to inspire novel approaches to drive the rapid evolution of the NiO synthesis technologies in future.

Keywords Nickel oxide · Nanomaterials · Microfuidics · Photocatalytic applications

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1 Introduction

Nanoparticles are ubiquitous in our daily life and are of great interest in several areas, forming the basis for an astounding array of applications of technological and scientifc importance. Nanoparticles can be broadly categorized as those with one of its characteristic lengths in the dimension range between 1 and 100 nm. They can be used as nano-building blocks of more sophisticated nanocomposites. The particles with size reduction to nanometer-scale will possess the properties deviated from their bulk ones signifcantly because of the large specifc surface area and unique quantum phenomenon (Greenham et al. [1997;](#page-15-0) Yang et al. [2019](#page-18-0); Wang et al. [2021](#page-18-1); Santhi et al. [2004](#page-18-2); Stickler et al. [2021](#page-18-3)), thus possessing remarkably superior advantages in chemical, photology, thermology, and magnetism areas. With the rapid development of nanotechnology, nanoparticles have been widely used in medicine (Kung et al. [2020](#page-16-0)), biology (Restaino and White [2019;](#page-17-0) Furtado et al. [2018\)](#page-15-1), electrical engineering (Jayathilaka et al. [2019](#page-16-1)), sensing (El-Shamy [2021](#page-15-2)), energy (Ma et al. [2020](#page-17-1)) and environmental applications (Peng [2002\)](#page-17-2). Therefore, nanoparticles have attracted great interest in recent years and remain a hot research topic in the near future.

The metal-oxide nanoparticle is one of the most important sub-classes of nanoparticles. The past decade has witnessed an explosion in the development of metal-oxide nanoparticles with controlled compositions, sizes, shapes, and structures for industrial applications. For example, titanium dioxide $(TiO₂)$ nanoparticles can remove a range of organic species via photodegradation under UV irradiation, thus it has been widely utilized in environmental pollution mitigation for water purifcation and air pollution treatment. One can find many excellent review papers on $TiO₂$ (Palmas et al. [2021;](#page-17-3) Hasan and Rana [2021;](#page-16-2) Jaji et al. [2020](#page-16-3)). NiO is also a burgeoning metallic oxide because it is naturally abundant and environmentally friendly with high thermal and chemical stability (Ghosh et al. [2016\)](#page-15-3). It can be applied as a combustion catalyst (Liu et al. [2017](#page-17-4)), anode interfacial layer in solar cells (Irwin et al. [2008](#page-16-4)), anode material in batteries (Li et al. [2021](#page-16-5)), gas sensors (Yang et al. [2021](#page-18-4)), and magnetic materials (Kumar and Das [2021](#page-16-6)). NiO has face center cubic crystal structure and ferromagnetic properties with a Neel temperature of 525 K (Rinaldi-montes et al. [2016a](#page-17-5); [b\)](#page-17-6). It is a p-type semiconductor with a wide bandgap of 3.6–4.0 eV and possesses peculiar magnetic and electric behavior depending on the particle size (Khatri and Rana [2020;](#page-16-7) Pooyandeh et al. [2021;](#page-17-7) Mohseni Meybodi et al. [2012](#page-17-8)). The unique chemical and physical characteristics render NiO particularly suitable for photocatalytic applications.

Diferent synthesis methods of NiO electrode material for supercapacitor have been reviewed (Kate et al. [2018](#page-16-8)),

the methods for NiO nanostructure synthesis and characterization of material properties have been reviewed (Bonomo [2018](#page-15-4)), and the overview has been provided for the synthesis and applications of nickel nanoparticles in size range of 1–100 nm based on solvothermal, physical, and chemical approaches (Jaji et al. [2020](#page-16-3)).

Microfuidics has emerged as a promising tool in nanomaterial formation, and it is also of importance to assess the application of microfuidics for NiO nanoparticle synthesis, which has not yet been sufficiently reviewed. This motivates us to undertake the review study in a more systematical fashion. The content of the review paper is organized as the following: in the frst section, the synthetic approaches for NiO nanoparticles are systematically surveyed, including solidphase method, liquid-phase method such as direct precipitation method, homogeneous precipitation method, sol–gel method, hydrothermal method, microemulsion method, organic complex precursor method, polymer-network gel method and biosynthesis method, as well as vapor-phase method. In the subsequent section, the principles and applications of microfuidics in fabricating NiO nanoparticles are reviewed. The representative applications using NiO nanoparticles for environmental remediation via photocatalytic approaches are also reviewed and discussed, considering the ever-increasing demands of NiO in renewable and sustainable applications. The summary of NiO synthesis methods and future development perspectives is fnally presented.

2 Conventional synthesis methods

The synthesis methods of nanoparticles can be divided into two major types: top-down and bottom-up. The former refers to crushing the bulky material into small-size substances by mechanical techniques, while the latter refers to building the nanoparticles via a chemical process. NiO nanoparticle formation process mainly relies on the bottom-up method through chemical reactions, obeying the classical nucleation theory, which involves three stages: nucleation, growth, and aging (Köhler et al. [2013](#page-16-9)). Adjusting the temperature of heat treatment, changing the ratio of water or pre-dehydration before the calcination can efficiently avoid nanoparticle aggregation during the formation process (Zhang [2014\)](#page-19-0). The NiO nanoparticle synthesis methods can be classifed into the following three categories according to the variations in the reaction media and environment. All the approaches have been intensively investigated to synthesize NiO nanomaterials with tailored composition, size, shape, and crystalline structure.

2.1 Solid‑phase method

The solid-phase method is a traditional and long-standing way of fabricating nanomaterials; it has advantages of low cost, solvent-free, high selectivity, and can be applied in industrial production at a relatively mild reaction environment. However, it has drawbacks such as limited accuracy, relatively low efficiency, and difficulty in controlling the particle characteristic properties (Zhang and Qiu [2009](#page-19-1)). Solid powders are normally used as precursors or reactants. Once homogeneously mixed, the input energy is required to initiate the reaction. Therefore, the high reaction temperature is usually indispensable for the solid-phase method. The microwave reaction system can be applied in solid-phase method, microwave can uniformly radiate to each part of reactants, it can cause the rotation, vibration, and swing of particles, therefore, increasing the efficiency of particles collision, and shortening the reaction time. As shown in Scheme [1,](#page-2-0) the solid reactants are grinded into powders, then mixed through vibration or rotation, and the heat treatment via calcination is applied to facilitate the pyrolytic reaction before the nanoparticles can be synthesized.

Xia et al. [\(2015](#page-18-5)) used nickel nitrate and oxalic acid as reactants with heat treatment at the temperature of 400 °C for 4 h. Wang et al. (2005) used Ni $(OAc)_{2}$ ⁻⁴H₂O and Tween 80 as the precursor, grinded them into the powder, and then applied the heat treatment by the calcination at 400 °C for 2 h and drying the samples for 4 h under 80 °C. NiO nanoparticles with a well mesoporous structure were synthesized, as shown in Fig. [1](#page-2-1). Hosny ([2011](#page-16-10)) produced 8-nm nanoparticles through a semi-solid-phase reaction with a 700 °C decomposition process.

2.2 Liquid‑phase method

The liquid-phase method can be classifed into several subtypes, such as direct precipitation method, homogeneous precipitation method, sol–gel method, hydrothermal

Fig. 1 The TEM of NiO nanoparticle samples synthesized by solidphase method, reproduced with permission (Wang et al. [2005\)](#page-18-6) from Elsevier

method, microemulsion method, organic complex precursor method, biosynthesis method and polymer-network gel method, according to the diferent liquid media that have been used. The liquid-phase method mostly uses metallic sault solution to separate the metal element into ions. The heat treatment, hydrolysis, or other processes can be applied to obtain the material precipitation or crystal, and dehydration will turn the precipitation into the target powders (Zhang and Qiu [2009](#page-19-1)). The NiO nanoparticles fabricated by the liquid-phase method normally feature with relatively uniform distribution of particle size, thus high monodispersity; thus, this method has been most widely used nowadays, owing to the controllable reaction conditions (Karatutlu et al. [2018\)](#page-16-11). The representative samples fabricated by each type of liquid-phase method are shown in Fig. [2](#page-3-0).

Scheme 1 The scheme of the solid-phase method using calcination

Fig. 2 A Scanning electron micrographs (SEM) images of powders synthesized by direct precipitation method. Reproduced with permission (Bahadur et al. [2008](#page-15-5)) from Elsevier. **B** TEM image of NiO nanoparticles synthesized by homogeneous precipitation. Reproduced with permission (Deng and Chen [2004](#page-15-6)) from Elsevier. **C** SEM images of sol–gel product. Reproduced by permission (Thota and Kumar [2007](#page-18-7)) from Elsevier. **D** SEM (a, b, c) and TEM (d, e, f) images of particles synthesized by the hydrothermal method. Reproduced with permis-

sion (Cao et al. [2020\)](#page-15-7) from Elsevier. **E** TEM image of NiO nanoparticles synthesized by the microemulsion method. Reproduced with permission (Han et al. [2004\)](#page-16-12) from Elsevier. **F** SEM image of NiO nanoparticles at high magnifcation (Tao and Wei [2004\)](#page-18-8). Reproduced with permission (Tao and Wei [2004](#page-18-8)) from Elsevier. **G** SEM images of NiO nanofowers synthesized by polymer gel method. Reproduced with permission (Munkaila et al. [2021](#page-17-10)) from Elsevier

2.2.1 Direct precipitation method

The direct precipitation method has been widely adopted in fabricating ultrafine particles. This method uses a chemical reaction to precipitate intact wedges. Then, precipitation will turn into nanopowders through purifcation, grinding, and heat treatment process (Karatutlu et al. [2018](#page-16-11)). Bahadur et al. ([2008](#page-15-5)) used sodium hydroxide (NaOH) and nickel nitrate $(Ni(NO₃)₂)$ as the precursor, reacting and leading to formation of a wet cake of nanocrystalline NiO, which was subsequently dried and grinded into powders. The grinding process gives rise to big variations in the particle dimension, and distribution of density, thus lacking physical integrality.

2.2.2 Homogeneous precipitation method

The homogeneous precipitation method adopts a principle similar to that of the direct precipitation method; it also used the chemical reaction to produce solid precipitation. However, it keeps the precipitation in solution at a balance condition and makes the precipitate at a uniform speed (Pan et al. [2021](#page-17-9)). It is attained through controlling the concentration of the precipitant. Therefore, this method is stable, balanced, and able to fabricate high-quality particles. Deng and Chen ([2004\)](#page-15-6) fabricated the NiO nanopowders with the purity of 99.73%, cube structure, and averaged size of 9 nm through homogeneous precipitation. Despite the high-quality nanopowders they have synthesized, they used $NiCl₂·6H₂O$

Scheme 2 Scheme of hydrothermal decomposition method for producing the NiO nanoparticles. Reproduced with permission (Lv et al. [2015](#page-17-12)) from Elsevier

solution and NH_3 · H₂O as pre-reactants. The ammoniate products will pollute the environment, so this method needs an extra purifcation process.

2.2.3 Sol–gel method

This method uses high chemical activity substances as a precursor, through hydrolyze, alcoholization, condensation, and other chemical reactions to get stable sol with relative uniform distribution of the nanoparticles (Kumar and Han [2019;](#page-16-13) Zorkipli et al. [2016](#page-19-2)). Then, heat treatment was utilized to obtain metal nanoparticles. The fnal particles can reach atom level and possess a very small grain diameter. Thota and Kumar ([2007](#page-18-7)) adopted nickel acetate tetrahydrate and oxalic acid as reactants, ethyl alcohol as a solute, undergoing 400 °C calcination. The black powders of NiO nanoparticles were fabricated by Pooyandeh et al. [\(2021\)](#page-17-7) using the sol–gel methods with nickel nitrate hexahydrate and nickel chloride hexahydrate as the precursor, and magnetic stirring was applied in the fabricate process, after quiescence for a short time, the solution was fltrated to form the NiO nanoparticles.

2.2.4 Hydrothermal method

The hydrothermal method refers to the nanoparticle fabrication process in which a solution was sealed in a highpressure vessel with the chemical reaction occurring at high pressure and temperature condition, reactants will undergo dissolving, recrystallization, and heat treatment to form the fnal products (Sree et al. [2020](#page-18-9)). Scheme [2](#page-4-0) shows the typical synthesis mechanisms and procedures of hydrothermal method, the chemical reactions take place in a solution to trigger the formation of the nanoparticles, followed by the heat treatment to remove wastes and obtain the purifed products. It has the advantages such as intact development of grain, relative uniform distribution of particle diameter, less aggregation of particles, obviation of calcination at high temperature in the fnal process, and in particular, it is a cost-efective and facile synthesis process. However, it is more time-consuming than most other methods. Adschiri et al. applied the continuous hydrothermal method for synthesis of 10 various metal-oxide nanomaterials including NiO nanoparticles, using a microreactor with inner diameter of microchannel to be 9.5 mm. They synthesized NiO nanomaterial with an average size of 200 nm in 2 min (Adschiri et al. [1992\)](#page-15-8). Cao et al. ([2020\)](#page-15-7) successfully fabricated three hierarchical NiO microspheres through the hydrothermal method. The molar ratio of 1,2-Propanediol and water was kept at 1:1 in the solution. Nickel nitrate $(Ni(NO_3)_2.6H_2O)$, urea $(CO(NH₂)₂$, and citric acid were chosen as reactants and the ratio was kept at 2:1:1. NiO microspheres were selfassembled and grew in a uniform condition. Nickel nitrate and $NH₂CONH₂$ are also widely used reactants in the hydrothermal method. An accurate calculation is needed to determine the ratio between each reactant.

2.2.5 Microemulsion method

This method works through two insoluble solvents, for example, oil/water system, to form an emulsion in the presence of a surfactant, or cosurfactants, which can decrease the water/oil surface tension to $1-10$ mN m⁻¹, facilitating the formation of emulsions (Malik et al. [2012](#page-17-11)). Meanwhile,

surfactants and cosurfactants will create a transient interfacial tension to prevent the droplets from coalescence (Ita [2020\)](#page-16-14). The formation of microemulsions normally needs centrifugation or magnetic stirring to accelerate the dispersion process. For example, Han et al. [\(2004\)](#page-16-12) used cyclohexane as oil phase, Triton X100 as surfactants, and hexyl alcohol as cosurfactants, which were mixed at a ratio of 5:3:2 with the aid of magnetic force to form emulsions efectively.

Fabricating nanoparticles through microemulsion method has multiple advantages, such as relatively simple experiment setup, low energy cost, and facile operation. Moreover, the range of particle diameter distribution is relatively narrow, and the particle diameter can be efficiently controlled; second, choosing appropriately diferent surfactants and cosurfactants can lead to the formation of nanoparticles with a special property (Malik et al. [2012\)](#page-17-11), and the particles will have less aggregation. They are stable due to the presence of surfactants and cosurfactants.

2.2.6 Organic coordination compound precursor method

This method used coordination compounds which are the substances with central metal atoms surrounded by nonmetal atoms (Crichton [2012](#page-15-9)), and are easy to be removed by pyrolytic reactions as a dispersing agent, then mixed with a metal ion to activate the coordination reaction. During the reaction, the dispersing agent will separate particles and prevent them from agglomeration. The reaction will lead to the fabrication of a highly dispersed precursor, which undergoes heat treatment to produce the target particles. Tao and Wei [\(2004](#page-18-8)) selected polymer as a dispersing agent, nickel acetate as reactants, which were mixed to form the precursor at a raised environment temperature of 373 K. NiO nanoparticles were fabricated when the temperature was further raised to 673 K with homogeneous size distribution of around 30 nm as shown in Fig. [2](#page-3-0).

2.2.7 Polymer‑network gel method

The polymer-network gel method can successfully fabricate the relatively pure phase of NiO particles with a uniform distribution of particle diameter and facile control over the shape of the crystalline grain. The formation of a macromolecular chain will make metal sault ion evenly distributed in the sol–gel. Drying and calcining the sol–gel will lead to the fabrication of the nanoparticles. Liu et al. ([2003](#page-17-13)) adopted acrylamide free radical polymerization and *N*,*N*′-methylene diacylamine bifunctional group using nickel nitrate aqueous solution as raw material, water-soluble propylene Amide monomer and *N*,*N*′-methylene diacylamine act as network agents, ammonium sulfate as initiator, fnally, obtaining NiO particles with size ranging in 15–20 nm. Munkaila et al. [\(2021](#page-17-10)) synthesized NiO nanofower using amphiphilic block

Scheme 3 The diagram of biosynthesis principles for forming the metal-oxide nanoparticles. Reproduced with permission of (Shah et al. [2015\)](#page-18-11) from MDPI

copolymer as reactants in one-pot synthesis method. The NiO nanoflowers with mesoporous structure were successfully fabricated, as shown in Fig. [2](#page-3-0).

2.2.8 Biosynthesis method

The biosynthesis techniques are very effective in synthesizing metal-oxide nanoparticles. It has drawn much attention as it is both environmentally and economically friendly, it does not require complex operation, and it is innoxious to cell and other biosystems. It uses the small natural molecules in biosystems in combination with the conventional synthesis methods (mainly liquid-phase method) to fabricate nanoparticles and has been proved to have better quality and productivity compared to the conventional methods (Sudhasree et al. [2014;](#page-18-10) Najjar et al. [2021](#page-17-14); Imran Din and Rani [2016;](#page-16-15) Arumugam et al. [2021\)](#page-15-10). Scheme [3](#page-5-0) shows the basic principle of the biosynthesis method. The extract substance of plant can be incorporated in the synthesis process of the nanoparticles. Its pH value, concentration, reaction time, and temperature will directly infuence the quality of fnal products. Sabour et al. (2020) applied the biosynthesis method, using Rheum Turkestanicum plant extractions, and successfully fabricated $CeO₂$ nanoparticles with average diameter of 30 nm. Similarly, Najjar et al. ([2020](#page-17-15)) used gelatin as stabilizing agent, and sol–gel method to successfully synthesize $SnO₂$ nanoparticles with an average diameter of 27 nm.

Sabouri et al. used tragacanth as stabilizing agents via the co-precipitation method to synthesize nanosheets with comparable high quality and low toxicity to cells. Its absorption efficiency of anionic dyes and cationic dyes reaches 82% and 60%, respectively (Sabouri et al. [2020a](#page-17-16), [b](#page-18-12)). The same group also combined the bio-method with the sol–gel method. They successfully synthesized NiO nanoparticles with an average diameter of 59 nm. They used Arabic gum as stabilizing agent, nickel nitrate (Ni $(NO₃)₂·6H₂O$) as reactants, forming an aerogel at 80 °C, enabling the formation of NiO nanoparticles via the biology activities (Sabouri et al. [2021\)](#page-18-13). Therefore, biosynthesis method paves a promising way to synthesize NiO nanoparticles in future.

2.3 Vapor‑phase method

The vapor-phase method uses high pressure and temperature vapor to carry the precursor and use its energy to fnish the reaction. The most used vapor-phase method is spray pyrolysis, which applies the high temperature and pressure at the nozzle to stimulate the pyrolytic reaction, and the precipitation reaction occurs simultaneously (Wuled Lenggoro et al. [2003](#page-18-14)). As the metal salt solution sprays out from the nozzle, it will instantly turn into vapor and separate into nanoparticles. Due to the rapid solution evaporation and release of vapor, it is hard to control the particle characteristics such as crystalline shape, particle diameter, and phase purity using this method. However, as it possesses the advantages of a large production rate and facile operation, it has wide industrial production potential (Karatutlu et al. [2018](#page-16-11)). Scheme [4](#page-6-0) shows the diferent stages of the process. Mixture of air and fuel will provide an environment with high temperature and high pressure, and the precursors are sprayed into droplets prior to the taking place of the pyrolytic reaction and evaporation process. Precursor droplets will start to nucleate and grow to form nanoparticles.

Wuled Lenggoro et al. [\(2003\)](#page-18-14) used low-pressure spray pyrolysis and successfully synthesized the NiO nanoparticles with an average size of 20 nm. The conversion process and results under diferent condition are shown in Fig. [3](#page-7-0). The solution contains precursor driven by pump going through the channels and is spurted at the nozzle, and the solubility of precursor decides the fnal type of particles. High-solubility precursor produces nanoparticles, and low-solubility precursor becomes sub-microparticles. Oh et al. ([2007\)](#page-17-17) synthesized hollow spherical nanoparticles with uniform distribution of particle diameter of around 20 nm, and welldispersed nanocrystalline structures. Moravec et al. ([2011\)](#page-17-18) fabricated NiO nanoparticles with average diameter about 50 nm by pyrolysis and reduction reaction through 1.5 cm inner diameter nozzle. They proved the production rate include particle size and particle size distribution increase with the increasing of saturation temperature.

Scheme 4 The different stages during the synthesis process of nanoparticles using the vapor-phase method

2.4 The advantages and limitations of conventional methods

Each method has its own advantages and challenges, as summarized in Table [1](#page-8-0). The liquid-phase method is the most widely used method in industrial production and lab-scale synthesis. The size of nanoparticles is more uniform and controllable than the solid-phase and vapor-phase methods. The solid-phase method widely uses pyrolysis or microwave, and it is convenient to operate; it has low requirements on the reaction environment and low cost, and the produced particles have a relatively uniform distribution of diameter. However, its quality is relative hard to control and always accompanied by agglomeration. The vapor-phase method has great potential in industrial manufacture, and it has great efficiency with relatively facile operation. However, it has relatively high requirements on the reaction environment and complexity of the devices.

3 Overview of microfuidic systems and applications

Microemulsion has become one of the most effective approaches for nanoparticle formation. However, the traditional methods for microemulsion formation sufer from **Fig. 3** Conversion process from droplet to particles of low-pressure spray pyrolysis. Reproduced with permission (Wuled Lenggoro et al. [2003](#page-18-14)) from Elsevier

poor control over the size distribution. An alternative way is to use microfuidics system, in which the fuid fow is constrained in geometry at the microscale, the fuid fow characteristics will undergo signifcant changes due to the fascinating competition among forces. Parameters describing microfuidic systems can be described by the balance of inertial force, viscous drag forces, buoyant forces, and interfacial tension forces; the balance of each of these forces gives rise to key dimensionless numbers: the Reynolds number (Re), Weber number (We), Bond number (Bo), capillary number (Ca) and flow rate ratio. The viscous force and surface tension will become dominant in the typical fow regimes at low Reynolds number (Bragheri et al. [2016](#page-15-11)). Microfuidic chips have been applied in biomedicine (Wu et al. [2016a,](#page-18-15) [b](#page-18-16)), organic synthesis (Zhao et al. [2019](#page-19-3)), microreactor (Schrimpf et al. [2019\)](#page-18-17), biomimicking (Xu et al. [2020\)](#page-18-18), and chemical analysis (Wu et al. [2016a](#page-18-15), [b](#page-18-16)). The microfuidic chip can integrate multiple fuid operations and functions within a portable device at a highly exquisite level (Chen et al. [2021](#page-15-12); Stroock [2008;](#page-18-19) Abedini-Nassab et al. [2021\)](#page-15-13). More demanding applications have been increasing exponentially, and the application areas expand from microreactors for catalysis and chemical synthesis to point-of-care diagnostics, from drug delivery to cell/molecule compartmentalization and diagnostic testing.

In a microdevice, the fows through a microchannel and can be manipulated in a droplet-wise dispersed way (Lian et al. [2020a,](#page-16-16) [b;](#page-16-17) Lim et al[.2017;](#page-16-18) Ren and Leung [2016](#page-17-19); Ren et al. [2015\)](#page-17-20) or a continuous way (Leung and Ren [2014;](#page-16-19) Ren et al. [2013](#page-17-21); Ren et al. [2013](#page-17-21); Leung and Ren [2013\)](#page-16-20). The droplet microfuidics has been widely used in producing microcarriers (Choi et al. [2016a,](#page-15-14) [b\)](#page-15-15), microcapsules (Shirk et al. [2013\)](#page-18-20), and cell-laden microgels (Choi et al. [2016a](#page-15-14), [b](#page-15-15)), because fast reaction times in such small compartments are induced by the high surface area to volume ratios, efficient heat and mass transfer, and short difusion distances. This mechanism can form droplets with uniform size distribution in a very narrow range. Droplet microfuidic device is composed of microchannels and microchambers (Ren et al. [2013\)](#page-17-21). A wide range of materials such as metal (Singh et al. [2010\)](#page-18-21), silicon (Singh et al. [2010](#page-18-21)), glass (Campbell et al. [2020](#page-15-16)), polymers (Boodaghi and Shamloo [2020\)](#page-15-17), and ceramics (Malecha et al. [2019](#page-17-22)) have been used to fabricate microfuidic devices. Each material has its own advantages, demerits, and application areas. For example, metal has the advantages such as low cost, ease machining, and high stifness. However, it is relatively hard to monitor the status of inner fuid and reaction because it is not transparent (James et al. [2020\)](#page-16-21). Using silicon-based materials for microfuidic chips enables multiple advantages as it is stable, easy to design, and it has special semiconductor characteristics. However, its transparency and price remain big problems (Singh et al. [2010](#page-18-21)). Glass has perfect transparency and chemical stability along with other advantages' however, its high fabrication expense hinders its application in making microdevices (Niculescu et al. [2021a](#page-17-23); [b\)](#page-17-24). Ceramics has unique chemical properties and high-temperature stability; however, due to the restriction of porosity, processing techniques, and brittleness, it is relatively hard to use (Singh et al. [2010\)](#page-18-21). Polydimethylsiloxane (PDMS) is one of the most well-known polymer materials used in microfuidics because it is cheap, easy to design, mechanically fexible, and chemically stable, making it a very popular material in lab on a chip areas, however, it still has the problem of molecular difusion and porosity (Nielsen et al. [2020](#page-17-25)). The technique used in the fabrication of microfuidic devices is, therefore, material dependent. Glass and silicon are mainly used by chemical methods, for example, wet and dry etching and electrochemical discharge machining (Hwang et al. [2019](#page-16-22)). Other materials like metals, are mainly used

in mechanical processes, for example, micromachining (Faustino et al. [2016\)](#page-15-19), micro-milling (Faustino et al. [2016](#page-15-19)), ultrasonic machining (Hwang et al. [2019\)](#page-16-22), blasting (Jáuregui et al. [2010](#page-16-23)), injection (Gale et al. [2018](#page-15-20)), and polymers are normally used by soft lithography.

Microfuidics technology has been drawing a lot of attention since the early 1990s (Manz et al. [1990\)](#page-17-26), and it has been applied to create a stable pre-reaction environment for chemical reactions. In addition, microfluidics can also efficiently separate diferent substances in a solution by the characteristic properties such as the density diference, examples including plasma separation, circular tumor cell capture, and detection (Wu et al. [2016a](#page-18-15), [b](#page-18-16)). Microfuidic technology has evolved rapidly in the past decade and demonstrated to be a promising solution with the capability to conduct multiple biological or chemical reactions in parallel, thus accelerating measurement outcomes of the conventional laboratory tests (Niculescu et al. [2021a;](#page-17-23) [b\)](#page-17-24), such as fast detection of circulating tumor cells (Xu et al. [2020](#page-18-18)) and environmental detection and remediation (Yew et al. [2019](#page-18-22); Lian et al. [2020a,](#page-16-16) [b\)](#page-16-17). In addition, microfuidics has versatile control over the fow conditions, giving rise to the ability to synthesize micro/ nanostructured materials with high monodispersity in size and shape distribution (Lian et al. [2018](#page-16-24)). Microfuidics has been widely used in biology and biomedical engineering for studies related to genes, cells, and proteins. The combination of microfuidics with biomolecular and tissue engineering provides a new direction for simulating the human internal environments (Tian et al. [2019](#page-18-23)) and biomimicking in vivo environments (Xu et al. [2020](#page-18-18)).

4 Fabrication of nanoparticles using microfuidic technology

The nanomaterial formation process can be achieved using a microfuidic platform with accurate control and monitor over the nucleation and growth of nanoparticles (Jensen [2017](#page-16-25)). Zhang et al. (2017) (2017) provided an overview on mixing, flow dynamics, and mass and heat transfer in microreactors along with three strategies for scaling-up microreactors: parallel numbering-up, consecutive numbering-up, and scale-out. They also proposed a possible methodology to design microchemical systems. Wang et al. ([2014\)](#page-18-24) had developed a new strategy for scaling-up of a microsieve dispersion reactor. Deposition methods can be applied to create a thin coating on the particles surface and form a layered structure (Jocic [2016](#page-16-26)), it has been widely used in food (Wang et al. [2018](#page-18-25)), medicine (Dai et al. [2021](#page-15-21)), textiles (Yip and Luk [2016](#page-19-5)), materials (Zhang et al. [2014](#page-19-6)) and environmental protection. Zhao et al. [\(2020\)](#page-19-7) successfully used a microfuidic interfacial synthetic method to fabricate covalent organic polymer microcapsules, and it has been widely used in wastewater treatment and environmental protection.

Microfuidics can produce particles with high purity, complex structures and a highly evenly distribution of particle sizes. Lian et al. [\(2020a,](#page-16-16) [b](#page-16-17)) successfully applied microfluidics in fabricating $TiO₂$ nanoparticles through surfactant wrapping sol–gel method, with $TiO₂$ nanoparticles evenly distributed on the outer surface of the multiple wall carbon nanotubes (MWCNTs), and it has the ability to absorb Rhodamine B for wastewater treatment. Du et al. [\(2011\)](#page-15-22) fabricated $SiO₂$ nanoparticles using membrane microreactor, they suggested a gas–liquid precipitation reaction system which can guarantee the quality meanwhile saving the cost. The same group improved the synthesized process of $BaSO₄$ by adding microbubbles created by the microreactor (Du et al. 2013). CaCO₃ nanoparticles were fabricated by Wang et al. ([2007\)](#page-18-26) via integration of a microstructured reactor and the microfluidics to enhance mass transfer in the mixing process. It is feasible to apply microfuidic technology in the synthesis process of NiO nanoparticles. Most nanomaterial growth process involves three steps: nucleation, growth, and aging. As shown in Scheme [5](#page-10-0), the microfuidic system enables accurate control over the fow conditions of diferent phases which are pumped into the microchannels, and the synthesis of nanomaterials can be achieved in the microreactors, with yielded products adopting diferent forms such as sphere-shaped, non-sphere-shaped particles and capsules, and fbers. The microfuidic platform provides a powerful mixing ability that can signifcantly stimulate the nucleation process. Besides, the stable fuid fow environment is suitable for the growth of large size particles and the formation of complex-shaped particles (Chen et al. [2021\)](#page-15-12).

Another attribute making microfuidics competitive for synthesizing NiO nanoparticles is that as the synthesis reaction of nanoparticles is mainly chemical reaction dominant, less time-consuming procedures will be favorable, and this can beneft from rapid mixing of reactants which can be achieved using microfuidic platforms. Liu et al. ([2014\)](#page-17-27) used microfuidic reactors connected in series for fabricating $AIPO₄$ -5 which is a very important crystalline microporous aluminophosphate material, the single-phase $AIPO₄$ -5 was produced in 1 min. In comparison, it will take the conventional methods, such as the hydrothermal methods, a few days to fabrication the same products. Furthermore, the same group used a millimeter-sized continuous fow reactor to synthesize ZSM-5 (Liu et al. [2016](#page-17-28)). The reaction time has been successfully shortened into seconds.

The rapid development of microfuidic reactor makes it possible to fabricate complex structured nanoparticles with an accurate control over the process. Liang et al. [\(2020\)](#page-16-27) developed a continuous mode microfuidic reactor, which can efectively enhance heat transfer and mixing. They successfully synthesized $\text{Ni}_{0.6}\text{Mn}_{0.2}\text{Co}_{0.2}\text{CO}_3$ with uniform **Scheme 5** Nanomaterial synthesis process based on microfuidic technology

particle size distribution, good thermal stability, and homogeneous transition metal distribution. As the NiO nanoparticle fabrication process normally involves high pressure, either the number of channels could be increased, the channel cross-section could be enlarged, or the fow rate in the channels could be increased to cope with a high mass fow rate (Kockmann et al. [2006\)](#page-16-28). Ceramics microreactors can be used when high temperature is required for the formation of NiO nanoparticles.

Kawasaki et al. [\(2010a](#page-16-29), [b](#page-16-30)) used a T-shaped micromixer and successfully applied a continuous supercritical hydrothermal method to synthesize NiO nanoparticles with size of 20–50 nm. Meanwhile, when they decreased the tube diameter from 2.3 to 0.3 mm, the size of NiO nanoparticles changed from 54.3 to 20.1 nm. Sue et al. used a central collision type micromixer for synthesis of $Fe₂O₃$ and NiO nanoparticles. They investigated the effects of the key factors such as residence time and temperature in the synthesis process. They used $Fe(NO)_3$ and $Ni(NO)_2$ as reactants in the continuous hydrothermal method and successfully fabricated nickel ferrite with a diameter less than 5 nm (Sue et al. [2011a](#page-18-27), [b](#page-18-28)). They also used a 0.3 mm T-shape micromixer to successfully fabricate NiO nanoparticles with an average diameter of 16 nm. They used preheated water fow in the T-shape tube to provide the energy for the hydrothermal reaction shown in Scheme [6](#page-10-1) (Sue et al. [2011a](#page-18-27), [b](#page-18-28)). Kiwamu

Scheme 6 A Temperature contour of T-shape mixer. Reproduced with permission of (Sue et al. [2011a,](#page-18-27) [b](#page-18-28)) from Elsevier. **B** (a) Schematic diagram of reaction fow system, (b) newly designed T-shape

mixer, (c) conventional T-shape mixer. Reproduced permission of (Kiwamu et al. [2009\)](#page-16-31) from Springer

et al. [\(2009\)](#page-16-31) invented a new type of micromixer for synthesis of nanoparticles. As shown in Scheme [6B](#page-10-1), they adopted a T-shaped micromixer with the tube inner diameter to be 0.18 mm. The completed fow system consists of the pumps, reactor and other devices. The micromixer was positioned at upstream of the reactor, the powerful mixing ability of the microfuidic mixer provided the highly uniform mixture solution. With hydrothermal reaction, they used this new mixer to achieve the fabrication of NiO nanoparticles with size of 18.2 nm and 23.5 nm, respectively. Kawasaki et al. [\(2010a,](#page-16-29) [b\)](#page-16-30) developed a new swirl micromixer for synthesis of NiO nanoparticles. Compared to a conventional T-shaped mixer, the average diameter of particles has been reduced from 47 to 20 nm with monodispersed size distribution. Moreover, computational fuid dynamic simulation results suggested that the swirl mixer can enable rapid and homogeneous mixing of fuids, therefore, producing highquality nanoparticles. Zhao et al. [\(2015\)](#page-19-8) constructed various NiO nanoarrays through Y-shaped needles, and they used a pump to drive the solution fow through the needles; then, the reactions occurred in microtubes to form nanosheets with a thickness of about 40 nm; they used the nanosheets to modify microchannels to enhance its ability to absorb protein. Xia et al. used the aerosol decomposition method in microreactor with inner diameter of 13 mm to successfully fabricate NiO nanoparticles with an average size of 10 nm. Furthermore, they investigated the infuence of the addition of salts (Xia et al. [2002](#page-18-29)). Lu et al. ([2019\)](#page-17-29) reported the synthesis of NiO nanoparticles with size of 4–6 nm encapsulated in the carbonization of eggshell membrane using a green and facile approach for hydrogen evolution reaction electrocatalysts. The eggshell was applied as the microreactor. They also synthesized NiO/C nanocomposites with higher catalytic activity and smaller Tafel slope. The method can dispose eggshell waste and synthesize NiO nanoparticles simultaneously. Yoko et al. ([2020\)](#page-19-9) applied a T-shaped micromixer system to investigate the correlation between the mass transfer rate and Reynolds number and the kinetics under reaction control conditions, and evaluated the reaction kinetics of synthesis for the nickel nitrate to nickel oxide reaction that took place in a wide range of temperatures and pressures around the critical point of water. The NiO nanomaterials with diameter ranging in 10–40 nm had been fabricated. Xu [\(2017\)](#page-19-10) investigated the most appropriate mixer design for a novel two-stage reactor by computational fuid dynamics modeling, constructed a two-stage continuous hydrothermal flow synthesis microreactor for synthesis of diferent metal oxides including NiO nanoplates with thickness of 3.4–54.3 nm. Michalska et al. ([2021\)](#page-17-30) deposited highquality NiO flm with thickness of 30 nm using a Tesla-valve micromixer. The key synthesis conditions for NiO nanomaterial formation using microfuidic reactors are summarized in Table [2](#page-11-0) which demonstrates that the morphology and size of NiO nanomaterials can be well tuned via control over the flow rate and the dimensions of the microreactor systems.

In summary, microfuidics has the intrinsic superior ability in enhancing mixing, heat, and mass transfer and provides a stable chemical reaction environment, giving rise to these advantages as follows:

- 1. Microfluidics enables high mixing efficiency even in laminar flow at low Reynolds number. It can accelerate the formation of precursor and shorten the reaction time signifcantly. Meanwhile, a microfuidic mixer, either in a passive or active way, can rapidly lead to homogeneous mixing of reactants, hence enhancing the quality of the final products.
- 2. Stable flow environment can facilitate the reaction with very stable conditions. A microfluid reactor with a uniform fuid fow can provide a very stable chemical environment, which is suitable for NiO nanoparticle synthesis.
- 3. Improved heat transfer ability can decrease the energy loss and make the reaction take place rapidly; most synthesis methods of NiO nanoparticles need heat treatment, microfuidic reactor accelerates the process of heat transfer and facilitates the nucleation and growth of nanoparticles.

Shape of NiO nanoma- terials	Size of NiO nanoma- terials	Flow rate	Channel size of micro- reactors	Flow residence time References	
Nanoplate	$20.1 - 54.3$ nm in thick- ness	30 mL/min	0.57 mm	$1.3 - 2.7$ s	Xu(2017)
Spherical nanoparticle	4–6 nm in diameter		Eggshell membrane reactor with $70 \mu m$ in thickness	~ 8 h	Lu et al. (2019)
Nanofilm	30 nm in thickness	50–100 µL one time $1-3$ µm		< 10 s	Michalska et al. (2021)
Array	40 nm in thickness	$25 \mu L/min$	$530 \mu m$	400 min	Zhao et al. (2015)
Spherical nanoparticle	10–40 nm in diameter	38.7 g/min	0.3 mm	\leq 1 s	Yoko et al. (2020)

Table 2 The summary of key synthesis conditions for NiO nanomaterial formation using microfuidic reactors

The detailed comparison between conventional synthesis methods and microfuidic synthesis methods has been shown in Table [3.](#page-12-0) Compared with the conventional methods, the microfuidic technology provides a number of merits for nanoparticle synthesis, such as highly monodispersed particle size distribution, and more accurate control over the particle size and the particle loading efficiency by tuning the fow and reaction conditions, as well as the microreactor dimensions (Shepherd et al. [2021\)](#page-18-30).

Owing to the exquisite control over the particle diameter, shape, and other properties in a tunable way, microfuidic systems have been demonstrated to be useful platforms for synthesizing nanoparticles of organic polymers, oxides, semiconductors, and metals as well as hybrid structures combining multiple materials and functionalities. Despite the rapid development of microfuidic technologies for nanomaterial synthesis, the novel approach still faces some challenges. First, it is important to identify and understand the mechanisms of channel clogging, such as constriction by deposition and accumulation on the walls or particle agglomeration, which remains challenging during the material synthesis process via the microfuidic approach. The microdevices will have to be cleaned thoroughly or even completely replaced by a new one. The surface modifications and flow modulation can potentially address these issues. For example, the contact between reactants and microreactor wall can be minimized using droplet fow or applying special coating on the reactor wall can efficiently isolate control the particle agglomeration (Zardi et al. [2021](#page-19-11)). Mitigation of the clogging in microchannels can also be achieved using pulsatile driven fow instead of static fow and this will signifcantly delay the formation of clogging (Dincau et al. [2022](#page-15-24)). Second, the throughput of microfuidic approach remains far from meeting the practical industrial demands. This challenge can be addressed by scale-up approach via increasing the number of devices running in a parallel mode. Third, the design of microfluidic chips requires substantial efforts to explore the optimal parameters, such as the number of microchambers and channel size (length, width, height). In addition, the fabrication process of microfuidic chips greatly relies on expensive laboratories and extremely sophisticated instruments (Niculescu et al. [2021a,](#page-17-23) [b\)](#page-17-25). Therefore, the development of microfuidic technology for nanomaterial synthesis requires more in-depth investigations and multidisciplinary integration of material science and fuid mechanics in particular. An efective way to address the fabrication challenge is to apply the commercially available off-shelf devices to build the micromixers and micropumps, this will obviate the need of photolithography method and clean room; therefore, it will signifcantly decrease the fabrication cost.

Table 3 Summary of the advantages and disadvantages of conventional method and microfuidic methods **Table 3** Summary of the advantages and disadvantages of conventional method and microfluidic methods

Scheme 7 The schematic of kinetic processes of photocatalysis

5 Photocatalytic applications of NiO nanomaterials

Photocatalysis is capable of photodegradation of the organic contamination, and the light absorption involved process can accelerate a photo-reaction by a catalyst, which has no change in itself and is not consumed during the chemical reactions. Photocatalysis is an eco-friendly technique to address energy and environmental challenges (Koe et al. [2020\)](#page-16-32). Semiconductor metal oxides such as NiO (Sun et al. [2018\)](#page-18-31), TiO₂ (Wang et al. [2020](#page-18-32)), MoS₂ (Chandrabose et al. [2021\)](#page-15-25), ZnO (Abdullah et al. [2020\)](#page-15-26), and Al_2O_3 (Li et al. [2016](#page-16-33)) can be activated by exposure to UV–visible light appropriate to its bandgap energy to catalyze a redox reaction at its surface. As shown in Scheme [7,](#page-13-0) the electrons of a semiconductor are excited from the valence band (VB) to the conduction band (CB) by photoluminescence. The photoelectrons and holes subsequently experience spatial separation and transfer to their acceptors. The oxidation and reduction reactions will require more positive hole potential and more negative CB potential to favor the reactions (Liu et al. [2020](#page-17-31); Byrne et al. [2015](#page-15-27)). Recently, NiO have been used as photocatalyst to degrade organic dyes, such as methyl orange (MO) and methylene blue (MB) (Ma et al. [2021](#page-17-32)). The electron and hole formation will contribute to oxidize/reduce organic pollutants, while the holes of NiO surface will adsorb and trap water molecules, with the causation of hydroxyl radicals oxidation. At the same time, the oxygen molecules produce anionic superoxide radicals, which will degrade MB and MO to CO_2 , and H_2O (Sabouri et al. [2020a,](#page-17-16) [b](#page-18-12)).

Among the transition metal oxides, $TiO₂$ is a very good photocatalyst and has been used in many photocatalytic systems, due to the high photoconversion efficiency, high sta-bility, and high specific surface area (Shrestha et al. [2010](#page-18-33)). However, the efficiency of $TiO₂$ is limited by wideband energy which is closed to 3 eV, and fast recombination of photo-generated electrons and holes, indicating only about 5% of the solar energy spectrum can be used (Mohapatra et al. [2007](#page-17-33); Jasim et al. [2020\)](#page-16-34). NiO is a p-type semiconductor with low cost, high optical transmittance, high specifc surface area, and can be shaped into complex structures (Chinnappan et al. [2018](#page-15-28)). It can form a p–n junction material with an n-type semiconductor. The electric feld arising from the p–n junction via the combination of p-type and n-type semiconductors can signifcantly restrain the recombination of photo-generated electrons and holes. NiO nanoparticles can signifcantly enhance the photocatalytic hydrogen evolution from aqueous methanol because NiO nanoparticles exhibit high activity attributed to ease of trapping photogenerated electrons (Wu et al. [2014;](#page-18-34) Faisal et al. [2018\)](#page-15-29); therefore, the combination of NiO and $TiO₂$ can significantly increase the efficiency of photocatalyst. This approach can also be extended to other n-type semiconductors, such as $SnO₂$ (Suvith et al. [2020](#page-15-26)) and ZnO (Abdullah et al. 2020). Hu and Teng (2010) loaded NiO on n-type NaTaO₃ to trigger the water-splitting reaction under UV light exposure, the final production rate of H₂ reached 9000 µmol g⁻¹ h⁻¹. Sun et al. [\(2021\)](#page-18-36) used p–n heterojunction between NiO and Cadmium Sulfde (CdS) to induce hydrogen spillover efect, remarkably improving the $H₂$ evolution performance with production rate to be 243.9 mmol/g/h. In addition, NiO as cocatalyst not only can cooperate with a metallic oxide but also with another catalyst to induce a synergetic efect. Liu et al. ([2018](#page-17-34)) loaded NiO on the surface of $g - C_3N_4$, the created C–O–Ni bond signifcantly improved hydrogen evolution and stability, the inner electric feld created by heterojunctions can drive the migration of the photo-generated electrons through the C–O–Ni linkage from $g - C_3N_4$ to NiO, which facilitated the charge separation. Khatri and Rana [\(2020](#page-16-7)) adopted Fe doping on the NiO nanoparticles through the chemical co-precipitation method to form photocatalysis dyes. It largely increased the photocatalysis efficiency and can be considered as promising photocatalysts for organic pollutant treatment. Lin et al. ([2018](#page-16-36)) applied two-dimensional amorphous NiO nanostructure instead of nanocrystal NiO particles in solar $H₂$ evolution, the NiO nanoparticles can work alone as a photocatalyst and functions stably and efficiently in the chemical reactions. Table [4](#page-14-0) shows the representative applications using NiO as the photocatalysts or co-photocatalysts, and it demonstrates that the addition of NiO can significantly improve the photocatalytic efficiency. NiO nanoparticles with high photocatalytic activities can be used as self-cleaning transparent nanocoating for solar

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cells to prevent the deposition of dust and air pollutants on the solar cells over time. It can be envisioned that NiO nanoparticles can fnd more diverse applications, especially in environmental felds.

6 Conclusions and outlook

NiO nanoparticles have attracted increasing attention because of unique chemical and physical properties. We provide a comprehensive review of the state-of-art synthesis methods of NiO nanoparticles, combining both the conventional methods and the latest advances in applying microfluidics as a promising alternative for the highly efficient synthesis of NiO nanoparticles.

The liquid-phase method is the most widely used method in industrial production and lab-scale synthesis. The size of nanoparticles is more uniform and controllable than the solid-phase and vapor-phase methods. With the development of microfuidic technology, the traditional synthesis method can be improved to achieve a higher production rate, expanded range of applications, and higher product quality. Moreover, the integration of multiple functions on the chip-based microfuidic systems shows a promise for the holistic realization of nanoparticles with demanded optical, electronic, and catalytic properties by controlling the channel geometries and fow conditions. Nevertheless, the novel approach still faces some challenges. First, it is important to identify and understand the mechanisms of channel clogging, and mitigate the problem by surface modifcations and flow modulation. Second, the throughput of microfluidic approach remains far from meeting the practical industrial demands. This challenge can be addressed by scale-up approach via increasing the number of devices running in a parallel mode. Third, the fabrication process of microfuidic chips greatly relies on expensive laboratories and extremely sophisticated instruments, and this can be addressed by applying the commercially available off-shelf devices to build the micromixers and micropumps in the microreactor systems. Accuracy and repeatability are also very crucial, and it is expected that automated apparatus should be used as much as possible without much intervention from human operators to ensure the quality consistency of NiO nanoparticles during the production process from one batch to another batch. More efforts are, therefore, required in the future to develop microfuidics based technologies for NiO nanoparticle synthesis in a more cost-effective and energy-efficient way, as well as exploring their photocatalytic capabilities for environmental remediation to more expanded extent.

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Declarations

Conflict of interest The authors declare no confict of interest.

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