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Microreactor technology in the synthesis of copper oxide (CuO) metal nanoparticles

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ABSTRACT

In this study, the synthesis of copper oxide (CuO) nanoparticles using continuous flow tubular microreactors has been investigated. Microreactors, as one of the advanced technologies in chemical engineering, allow for precise control of reaction conditions and, due to their microscopic dimensions, high mixing speed, and exceptional ability to manage reaction parameters, provide an ideal environment for the synthesis of uniform nanoparticles. The results of this study show that the use of microreactors can significantly reduce the inhomogeneous particle size distribution and improve the physical and chemical properties of CuO nanoparticles. The nanoparticles produced in this process have a uniform size distribution, high stability, and optimal properties for industrial applications. These properties make CuO nanoparticles find wide applications in areas such as catalysts, electronic technologies, and environmental processes. This research shows that the use of microreactors can help improve production efficiency, and develop sustainable and uniform processes in nanoparticle synthesis

Keywords: Microreactor, Nanoparticle Synthesis, Copper Oxide (CuO) Nanoparticles

Introduction

In the present era, technological advances in chemical engineering have provided new solutions for optimizing material production processes. In the meantime, microreactors, as advanced and precise tools, have created a major revolution in the synthesis of materials, especially nanoparticles. By providing precise control over reaction conditions, such as temperature, residence time, and mixing rate, these systems enable the production of nanoparticles with uniform size and controlled distribution[1]. One of the fundamental challenges in the synthesis of nanoparticles is achieving particles with specific dimensions and uniform distribution, which directly affects their physical, chemical, and performance properties. Due to their small dimensions and high mixing speed, microreactors provide ideal conditions for controlling the nucleation and growth processes of nanoparticles and prevent the formation of particles with inhomogeneous sizes[2]. This article reviews the role of microreactors in improving the size control and uniform distribution of nanoparticles and analyzes the importance of these features in various industrial and scientific applications such as catalysts, pharmaceuticals and electronics.

Special emphasis on the impact of this technology on increasing efficiency and reducing costs doubles its importance in sustainable development. Microreactors have become important tools in the synthesis of nanoparticles due to their features such as high contact surface area, precise control over temperature and residence time, and the possibility of mixing reactants uniformly. These devices usually consist of narrow channels in which the flow of liquids or gases is continuously and precisely controlled, which is especially important in the production of nanoscale materials[3].

In the synthesis of nanoparticles, especially metal and metal oxide nanoparticles such as cuo (copper oxide), one of the fundamental challenges is the production of particles with uniform sizes and desirable surface properties.

These properties not only affect the physical and chemical properties of nanoparticles but also their applications in fields such as catalysis, pharmaceuticals, electronics, and sensors.

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Due to the nanoscale, synthesis processes must be designed in such a way that the shape, size, and uniformity of distribution of individual particles can be precisely controlled. Microreactors have become a very efficient tool in this field due to the ability to precisely control parameters such as temperature, pressure, and flow rate. Microreactors have several advantages, especially in the synthesis of nanoparticles, due to the possibility of creating very precise and controllable reaction conditions. For example, in the synthesis of cuo nanoparticles, simultaneous processes such as synthesis and oxidation can be carried out optimally and with the desired particle size. Using these microfluidic systems, precise control over the reactant concentration, molar ratio, temperature, and residence time is possible, all of which contribute to the production of nanoparticles with desired properties. Finally, due to the significant advantages of microreactor technology in precise process control, this technology has been able to find a special place in the field of chemical engineering and nanoparticle production and is recognized as an advanced and efficient tool for the synthesis of new and innovative materials in various industries. In this article, we will examine the processes of nanoparticle synthesis in microreactors and their effect on the properties of the produced nanoparticles[4].

Fluid Flow Equations

For tubular microreactors, the assumption of laminar flow is often used. The Navier-Stokes equation for single-phase Newtonian flow is as follows:

$$f + \mu \nabla^2 v - pv = \rho(\frac{\partial v}{\partial t} + v.\nabla v) \quad (1)$$

Where ϱ is the fluid density, v is the fluid velocity, P is the pressure, μ is the dynamic viscosity, and f is the volumetric forces such as gravity. For steady, tubular flow, the equation can be simplified, in which case the hydrodynamic velocity profile becomes parabolic[5].

Mass transfer

Mass transfer plays an important role in the uniform distribution of reactants and the nucleation of nanoparticles. The mass conservation equation for substance A is as follows:

$$-r_A + D_A \nabla^2 C = (\frac{\partial C_A}{\partial t} + v \nabla C) \quad (2)$$

where the concentration of substance C_A, D_A is the molecular diffusion coefficient, r_A is the reaction rate for substance A[6].

Heat transfer equations

Temperature control is crucial for the uniform synthesis of nanoparticles. The energy equation is written as:

$$q + k\nabla^2 T = \rho C_p(\frac{\partial T}{\partial t} + v.\nabla T) \quad (3)$$

where Cp is the specific heat capacity, T is the temperature, k is the thermal conductivity, q is the rate of heat generation from chemical reactions, and v is the fluid velocity[7].

Chemical Reaction Kinetics and Nanoparticle Formation Rate

For the synthesis of nanoparticles, the nucleation rate (J) and particle growth (G) are of great importance:

$$Aexp\left(-\frac{\Delta G}{k_B T}\right) = J \quad (4)$$

$$G = Ck_g - C^* \quad (5)$$

where A is the projection coefficient, ΔG is the nucleation activation energy, kB is the Boltzmann constant, C is the precursor concentration, C∗ is the equilibrium concentration, kg is the particle growth constant[8]

Particle Conservation Equation (Size Distribution)

To predict the size distribution of nanoparticles, the Popkin-Fokker-Planck equation is used:

$$R(L,t) = [G(L,t)f(L,t)]\frac{\partial}{\partial L} + \frac{\partial f(L,t)}{\partial t} \quad (6)$$

where f(L,t) is the distribution of the number of particles with length L at time t, G(L,t) is the particle growth rate, R(L,t) is the nucleation rate[9]

Important dimensionless numbers

For analyzing system behavior and optimal design of microreactors, key dimensionless numbers are used including:

Peclectic number (Pe): to examine the ratio of convective to diffusive mass transfer

$$Pe = \frac{uL}{D} \quad (7)$$

where u is the average velocity of the fluid flow (m/s), L is the characteristic length (or hydraulic diameter) of the microreactor (m), D is the molecular diffusion coefficient of the reactants in the fluid (m2/s)

Reynolds number (Re): to determine the type of flow

$$Re = \frac{\rho_h u D}{\mu} \quad (8)$$

Where, ϱ is the fluid density (kg/m3), u is the average fluid flow velocity (m/s), Dh is the hydraulic diameter of the microreactor,

μ is the fluid dynamic viscosity (Pa·s)

Damkohler number (Da): to compare chemical reaction time with mixing time

$$Da = \frac{C^{n-1}L}{u} \quad (9)$$

where k is the reaction rate constant (m3(n-1). s(n-1)/mol), C0 is the initial reactant concentration (mol/m3), n is the reaction order,

L is the characteristic length of the microreactor (m), u is the average fluid flow velocity (m/s)[10].

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Materials And Methods Of Experiments

Materials and Equipment Required

1. Copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$)
2. Sodium hydroxide (NaOH)
3. Deionized water
4. Tubular microreactor (Teflon or silicone material with two inlets for solutions and one outlet for the product)
5. 60 ml syringes (two)
6. Flask or flask for preparing solutions
7. Laboratory glassware such as beaker and funnel
8. Heat source (hot water bath)
9. Micromixer for better mixing
10. Syringe pump

Testing Method

1. Step 1: Preparation of Solutions
2. 0.1Copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) solution:
3. 1.4982 g of copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) accurately
4. Dissolve this in 60 mL of deionized water to obtain a 0.1 M solution of copper sulfate pentahydrate.
5. Transfer the resulting solution to one of the 60 mL syringes.
6. Sodium hydroxide (NaOH) solution:

- 0.48 g of sodium hydroxide (NaOH) accurately
- Dissolve this amount in 60 ml of deionized water to obtain a 0.2 M sodium hydroxide solution.
- Transfer the resulting solution to a second 60 ml syringe.

The copper sulfate pentahydrate ($CuSO_4 \cdot 5H_2O$) solution in the first syringe is slowly and carefully injected into the tubular microreactor through a micromixer inlet. The sodium hydroxide (NaOH) solution is injected into the microreactor in the same manner.

Note: The flow of each solution must be slow and simultaneous into the microreactor to ensure a uniform and controlled precipitation reaction. The adjustment of reaction conditions, including temperature, flow rate, and reactant concentration, has a fundamental effect on the size and distribution of the synthesized nanoparticles.

- Step 3: Reaction and precipitation
- Due to the simultaneous injection of copper sulfate dihydrate ($CuSO_4 \cdot 5H_2O$) and sodium hydroxide (NaOH) solution, the following reaction occurs inside the microreactor:
- $Na_2SO_{4(aq)} + Cu(OH)_{2(s)} + 5H_2O_{aq} \rightarrow 2NaOH_{aq} + Cu(SO_4), 5H_2O_{aq}$

In this reaction, copper hydroxide ($Cu(OH)_2$) is formed as a precipitate.

Step 4: Heating and conversion to copper oxide

The copper hydroxide ($Cu(OH)_2$) precipitate resulting from the reaction is heated at 600°C for 2 hours. Heating causes the water molecules to disappear and the copper hydroxide to convert to copper oxide (CuO).

Step 5: Washing and drying

- After heating, the resulting copper oxide (CuO) precipitate is carefully washed to remove impurities and excess ions.
- Then, the copper oxide (CuO) is dried at 100°C to form dry copper oxide (CuO) nanoparticles ready for use in subsequent experiments.

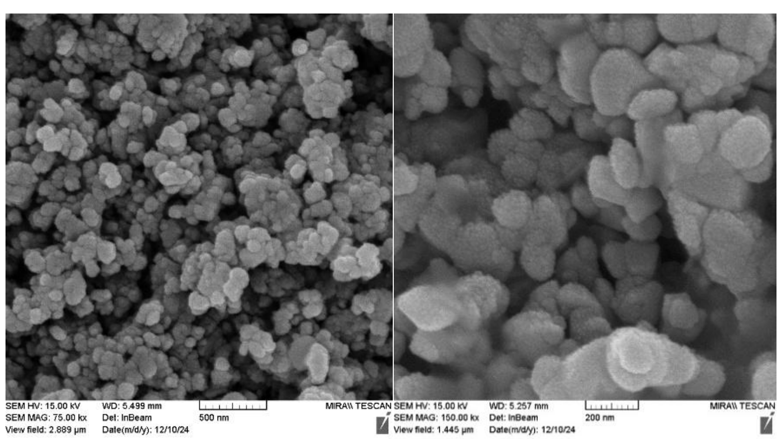

Figure 1. SEM images of CuO nanoparticles calcined at 600 °C, copper sulfate concentration (0.2 M), sodium hydroxide concentration (0.2 M), inlet flow (1.5 mL/min), microreactor length (10 m)

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Table 1. Different laboratory conditions for the synthesis of CuO nanoparticles

Experiment No.	Copper sulfate concentration (molar)	Sodium hydroxide concentration (molar)	Inlet flow (Q, ml/min)	Microreactor length (L, m)	Residence time (τ\, s)	Nanoparticle size (nm)	Efficiency (η, %)	Copper sulfate mass (g)	Sodium hydroxide mass (g)
1	0.1	0.1	.05	5	18868	150-120	92	1.498	0.240
2	0.1	0.1	1	5	9434	150-180	90	1.498	0.240
3	0.2	0.1	1	10	18868	100-120	94	2.996	0.240
4	0.2	0.2	1.5	10	12579	130-160	88	2.996	0.480
5	0.3	0.2	2	15	14150	80-100	95	4.494	0.480
6	0.3	0.2	2.5	20	15094	60-90	96	4.494	0.480
7	0.3	0.3	3	20	12579	70-110	92	4.494	0.720
8	0.3	0.3	3	30	18868	50-80	98	4.494	0.720
9	0.1	0.3	2	25	23596	40-70	99	1.498	0.720

1. This table shows the results of experiments conducted for the synthesis of copper oxide (CuO) nanoparticles in microreactors. In the columns of the table, the main parameters of the synthesis process such as temperature (°C), feed flow rate (mL/min), initial concentration of reactant solutions (mol/L), residence time (s), and mixing rate are investigated. Also, the characteristics of the produced nanoparticles such as particle size (nm), particle size distribution (σ), and product uniformity are reported.
2. Temperature (°C): The effect of temperature on nucleation and particle growth has been investigated. Higher temperatures increase the reaction rate and reduce the particle size, but may affect the particle size distribution.
3. Flow rate (mL/min): By changing the inlet flow rate, the mixing rate and residence time change. Increasing the flow rate results in better mixing and higher uniformity.
4. Initial concentration of solutions (mol/L): The concentration of reactants plays an important role in the nucleation and growth of nanoparticles. Higher concentrations contribute to a faster rate of particle formation and increased reaction efficiency.
5. Residence time (s): This parameter indicates the length of time that the materials remain in the microreactor. Longer residence times usually lead to the formation of larger particles.
6. Particle size (nm): Indicates the average size of the produced nanoparticles, measured using an electron microscope.

Conclusion

The results of this study show that the use of microreactors in the synthesis of copper oxide (CuO) nanoparticles can significantly improve the particle size distribution and produce nanoparticles with more uniform size and optimized physical and chemical properties with an average size below 200 nm. Investigations based on experimental conditions showed that careful control of parameters such as temperature, inlet flow rate, raw material concentration, and mixing rate play a key role in the quality and uniformity of the produced nanoparticles. Also, the use of microreactors reduced energy and raw material consumption and prevented uncontrolled aggregation or precipitation. The results obtained prove that this method, in addition to reducing costs, also improves production efficiency and provides a sustainable route for the production of nanoparticles with advanced properties.

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