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Hydrothermal Green Fabrication of CuO Nanoparticles Using Merremiaquinquefolia: Physicochemical Characteristics, Visible-Light Photocatalytic Kinetics, and Multifunctional Bioactivity

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ABSTRACT

In this paper the green hydrothermal method is proposed to synthesize CuO nanoparticles with the Merremiaquinquefolia extract as a plant-based functional media. The framework explores the effect of CuO formation under varying concentrations of extract and hydrothermal temperature on the physicochemical properties. Plant phytochemicals are thought to play an important role in the nucleation, growth and surface stabilization of nanoparticles. Structural characterization is based on crystallite size with the target value being in the range of ~25nm. The photocatalytic degradation of visible-light and DPPH radical-scavenging activity are used as functional assessment. The photocatalytic activity of the material is evaluated by pseudo-first-order kinetic method, and the degradation efficiency is targeted to be close to 93% after 90 min of irradiation. Evaluation of antioxidant activity is done at the different concentrations of CuO and a target value of 82% inhibition of DPPH is considered. The framework integrates hydrothermal conditions, nanoscale characteristics, photocatalytic performance and the antioxidant response to establish relationships among them and lay the foundation for future environmental remediation and multifunctional material applications.

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1. INTRODUCTION

Copper oxide (CuO) is a p-type transition-metal oxide with nanoscale physicochemical properties and possible usage in environmental remediation and in biological systems, which has gained considerable interest. Nanoscale CuO reveals high surface area and interesting optical properties for applications involving visible-light-driven reactions. The properties of CuO nanoparticles suggest their potential use in photocatalytic elimination of pollutants and as antioxidants. Environmentally conscious preparation procedures are also crucial as the preparation of nanoparticles can impact particle size, surface properties, and the performance of the particles.

The conventional preparation of CuO has a number of disadvantages, such as the use of chemical reagents, a reducing agent, a stabilizer, and a multiple step process. These could lead to higher consumption of chemicals and a more complicated process, as well as environmental issues with leftover chemicals. Plants may also be used to prepare nanoparticles, in this case using naturally occurring phytochemicals as active components in the preparation of nanoparticles. Phytochemical groups present in Merremiaquinquefolia (phenolic, flavonoids, hydroxyl-containing) could be involved in precursor interactions, nucleation, crystal growth and surface modification during hydrothermal processing.

Although there is growing interest in the use of plants for the preparation of CuO, there is a need to

systematically investigate the relationship between CuO nanoscale parameters, their photocatalytic performance, and their biological activity in this case. The present framework thus examines two parameters that are controllable, namely the concentration of M. quinquefolia extract and the temperature in which it was cooked. The concentrations of the extracts ranged between 5% v/v to 15% v/v and the hydrothermal temperature was in the range of 120–200°C to assess their effects on three key outputs: crystallite size, photocatalytic degradation of visible light, and DPPH radical-scavenging activity. The objectives of the research are to develop a CuO preparation pathway by hydrothermal technique, analyse the characteristics of the prepared CuO from the hydrothermal route, clarify the relationship between the process, the structure and the performance of prepared CuO, and evaluate its photocatalytic and antioxidant kinetic activity with respect to the prepared CuO.

2. Literature Review

The CuO nanoparticles have a monoclinic crystal structure with a p-type semiconductor characteristic, which facilitates its application in environmental remediation and functional materials [1]. The small size of their nanoscale morphology leads to more surface area that can interact with neighboring molecules [3]. Other features such as surface activity and charge transport and overall functional performance are also influenced by crystallite size [12]. The optical absorption property of CuO is important in processes using visible lights since efficient optical photon absorption would lead to generation of charge-carriers and surface reaction [9].

The method of nanoparticle preparation by plants has become an environmentally friendly method for the production of metal oxide nanoparticles [4]. The phenolic compounds, flavonoids and other phytochemicals with hydroxyl or carbonyl functionalities are present in plant extracts [8]. These constituents can take part in the precursor transformation, nucleation, crystal growth and stabilizing of nanoparticles [5]. Thus, their concentration might affect the formation and aggregation of particles [11]. These effects can be studied using the extraction range from 5 to 15% v/v.

For nanoparticles, the availability of hydrothermal processing offers the added degree of control that comes with the ability to control the forms various crystal sizes take as they form, depending on temperature. Temperature and short time (about 120-200 C) processing can affect the crystallinity, morphology and aggregation of CuO. It may be possible to achieve controlled crystallization with moderate hydrothermal conditions, while raising the temperature can help to grow the crystals into a larger size. Therefore hydrothermal temperature and plant extract concentration are important variables to control the CuO physicochemical characteristics [6].

The applications of CuO nanoparticles are also quite promising in visible light photocatalytic applications [7]. Light absorption may create electron–hole pairs and then surface reactivity and the production of reactive oxygen species, all of which can help to reduce pollutants [2]. Hence, the dye removal could be used as a model system to assess the efficiency of photocatalytic reaction and reaction kinetics [11]. The DPPH radical-scavenging activities of plant derived CuO might also arise due to the surface chemistry of the nanoparticles, presence of constituents of the plant, behaviour of electrons and nanoscale dimensions [5]. Yet, there is limited knowledge of the joint relationship of hydrothermality, physicochemical parameters, visible-light photocatalytic kinetics and antioxidant response [4]. To create an integrated process-structure-performance framework for multifunctional CuO nanoparticles [10] it is necessary to tackle this relation.

3. Materials And Methods

3.1 Research Framework and Plant Extract Preparation

The study is based on integrated green approach of preparation and functional evaluation of CuO nanoparticles from Merremiaquinquefolia. First, fresh plants are harvested, washed to remove any particles attached to it and then dried. For a 1:10 g/mL plant to water ratio, the plant material is mixed with the distilled water. The extraction process, known as aqueous extraction, is done at 60°C for 45 min and the plant extract is subsequently filtered to obtain a clear extract. The extract is then diluted to 5-15% v/v to be used in the preparation of CuO. These concentrations offer gradual variation of the quantity of phytochemicals from the plants which may interact with the copper precursor.

The whole research process is shown in Figure 1. The hydrothermal treatment of the prepared M. quinquefolia extract and Cu precursor results in the recovery of CuO nanoparticles which are analyzed using XRD, subsequently, the crystallite size of the obtained nanoparticles is evaluated. Visible-light photocatalytic degradation is then followed by an assessment of the DPPH radical-scavenging activity, in total, as a functional evaluation. The framework is thus related to the two main input parameters – namely extract concentration and hydrothermal temperature – with the intended outputs of approximately 25 nm crystallite size, 93% photocatalytic degradation at 90 min and 82% DPPH inhibition at 200 µg/mL, respectively, as shown in Figure 1. This workflow gives a structured, basis for the analysis of the influence of the processing

conditions on the characteristics of CuO and the functional performance.

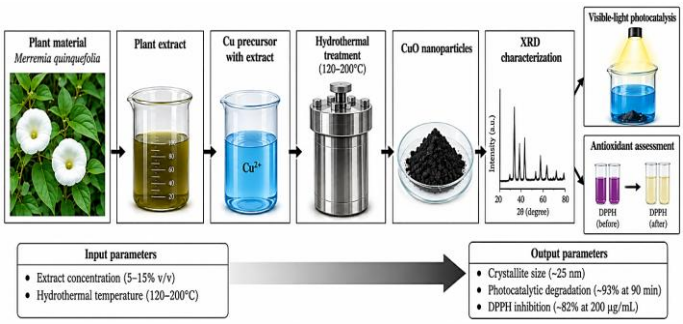

Fig. 1. Research framework for hydrothermal CuO preparation

3.2 Hydrothermal CuO Preparation

The hydrothermal preparation stage aims to explore how CuO nanostructures are formed as a result of changing hydrothermal temperature and concentration of Merremiaquinquefolia extracts. Three hydrothermal temperatures (120C, 160C and 200C) are classified as low, intermediate and high processing temperatures. Likewise, three concentrations of the extract (5%, 10% and 15% v/v) are employed to investigate the role of the phytochemicals of plant origin in the processes of nucleation and crystal growth. All other preparation conditions are kept at constant values to interpret clearly the impact of these two major inputs within the five parameter domain.

The main input and output parameters for the study are listed in Table 1. There are 2 controllable inputs, extract concentration and hydrothermal temperature. They are evaluated based on three main outputs associated with them that are crystallite size, visible-light photocatalytic degradation and DPPH radical-scavenging inhibition. The proposed response windows are 20–35 nm for crystallite size, 85–95% for degradation of photocatalytic dyes and 70–85% for DPPH inhibition. The values are used to establish the "target range" for subsequent process–structure–function testing.

Table 1. Core parameters of the CuO research framework

Parameter	Type	Range/Target
M. quinquefolia extract concentration	Input	5–15% v/v
Hydrothermal temperature	Input	120–200°C
Crystallite size	Output	20–35 nm
Photocatalytic degradation	Output	85–95%
DPPH inhibition	Output	70–85%

3.3 Physicochemical Characterization

X-ray diffraction (XRD) is primarily used to analyze the physicochemical properties of CuO nanoparticles such as crystal structure, diffraction behavior, crystallinity, and crystallite size. The diffraction patterns are analysed for the selection of 2θ angle range that shows the characteristic reflections of monoclinic CuO. A special branch of attention is given to the major diffraction peak in the vicinity of the (111) plane since the position and broadening of (111) peak give valuable information for estimation of crystallite-size. The Scherrer equation is used to calculate the crystallite size:

$$D = \frac{K\lambda}{\beta \cos \theta}, \quad (1)$$

where$D$ represents crystallite size, K is the shape factor, λ is the X-ray wavelength, β denotes the full width at half maximum of the selected diffraction peak, and θ is the corresponding Bragg angle. . XRD-based assessment framework for CuO nanoparticles prepared at different hydrothermal temperatures 120°C, 160 °C, 200 ° C is presented in figure 2. The stacked diffraction profiles show the characteristic CuO reflections and the enlarged (111) reflection shows how the peak-fitting and calculation of FWHM can be used for a crystallite-size calculation. Comparing the peak width and intensity at the different temperatures gives a basis to assess the change in crystallinity and crystal growth as a function of temperature. FTIR, SEM/EDX and UV-Visible spectroscopy can also aid in the identification of functional groups, morphology, elemental composition, and optical behaviour, while introducing neither any other optimization parameter.

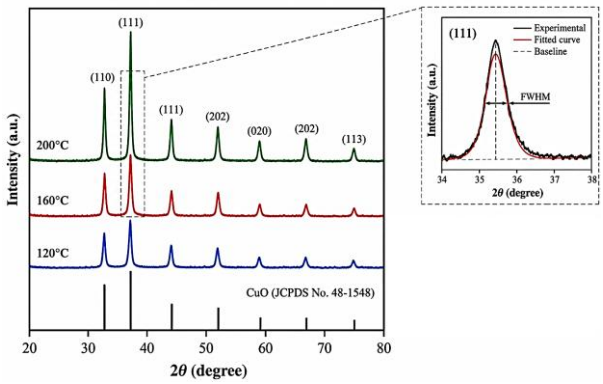

Fig. 2. XRD profile and crystallite-size assessment of CuO nanoparticles

3.4 Photocatalytic Evaluation and Kinetic Analysis

The CuO nanoparticles are tested for photocatalytic activity in visible light for the degradation of methylene blue (MB) as a model organic pollutant. The dosage of catalyst used for the treatment of a working concentration of MB is 0.5g/L, with the use of CuO

nanoparticles. Suspension is maintained under dark conditions for 30 min which is enough time for the adsorption–desorption equilibrium of pollutant molecules with the surface of CuO to be established before illumination. Suspension is then subjected to visible light for 90 min, and samples are taken at 15 min intervals and treated as is a procedure to see how the concentration of MB decreases with time. The photocatalytic degradation efficiency is measured with reference to:

$$DE(\%) = \frac{C_0 - C_t}{C_0} \times 100, \quad (2)$$

where C_0 represents the initial MB concentration and C_t represents the concentration after irradiation time t. The proposed window of the degradation is from 85-95%, and the optimum target response is around 93% after 90 min of visible light exposure. A pseudo-first-order reaction model is also studied to investigate the reaction kinetics, based on the dependence of the irradiation time on the logarithm of the pollutant concentration. The apparent rate constant k_{app} is obtained from the slope of the kinetic relationship. A coefficient of determination of $R^2 \geq 0.95$ is adopted as the target criterion for satisfactory kinetic fitting. This evaluation makes a quantitative comparison of the photocatalytic performance possible under the different CuO preparation conditions chosen.

3.5 Antioxidant Assessment and Statistical Analysis

The antioxidant activity of CuO nanoparticles is analysed by the DPPH radical-scavenging assay. The concentration used are 25, 50, 100, 150 and 200 µg/mL to study the concentration-dependent antioxidant response. The CuO concentration allowed to interact with the DPPH solution is controlled in order to measure the intensity of the decrease in the absorbance of DPPH which would be an estimation of the radical scavenger efficiency. The range of responses is proposed as 70-85% inhibition with a maximum to be achieved of about 82% at 200 µg/mL. The variation of antioxidant activity over the concentration range is examined to see if increased availability of CuO will increase its interaction with free radicals and lead to better scavenging efficiency.

All measurements are made in triplicate (n = 3) and reported mean ± SD; for subsequent experimental validation. The differences between the functional responses and preparation conditions are assessed at the statistical significance level of $p < 0.05$. The influence of extract concentration and hydrothermal temperature on the crystallite size, photocatalytic degradation and DPPH inhibition are compared in order to establish some process–performance relationships. In the photocatalytic modelling, an R^2 value of 0.95 and higher is regarded as satisfactory data model fitting. On the basis of this statistical framework, the functional performance of the CuO nanoparticles can be assessed considering the preparation conditions, the evaluation of it can be made based on the consistency of variability.

4. Results And Discussion

4.1 CuO Formation and Physicochemical Characteristics

The concentration of Merremiaquinquefolia extract and the hydrothermal temperature both play important roles in the formation of CuO nanoparticles by controlling the nucleation and growth of the crystals. The availability of nucleation sites and the interaction between phytochemicals in plants and copper species during the initial formation stage may affect the phytochemicals. Concurrently, the hydrothermal temperature conditions affect the crystallization and crystal-growth characteristics. The intended minimum crystallite size range for the investigated conditions is proposed to be kept between 20-35 nm with around 25 nm as the proposed minimum crystallite size.

At 5 % extract concentration, the crystallite size values are 33.4, 29.1 and 32.0 nm at hydrothermal temperatures of 120 °C, 160 °C and 200 °C, respectively (as shown in Figure 3). Raising the extract concentration to 10% does likewise and yields corresponding values of 29.2, 25.0 and 27.6 nm, whereas the 15% concentration yields 31.5, 27.3 and 30.2 nm. The response pattern indicates the range of concentration of 10% at temperature 160°C as the intended working region that corresponds to the minimum target crystallite size of 25.0nm. The higher contribution at 200°C is the expected contribution from increased crystal growth at higher hydrothermal temperatures. Likewise, the greater crystallite size of 5% and 15% extract levels indicate that inadequate and excessive phytochemical levels could result in less beneficial control of nucleation/growth. These trends are set up as the proposed process-structure relationship that will be tested experimentally.

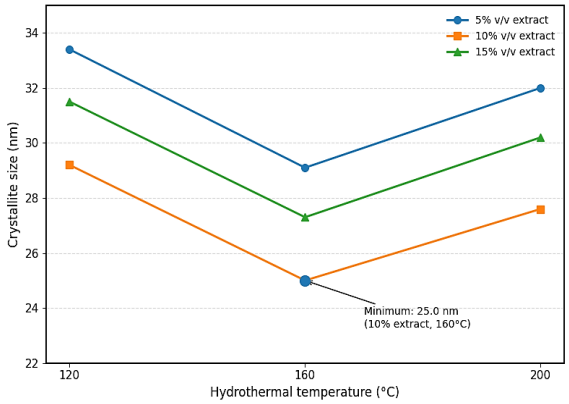

Fig. 3. Effect of processing conditions on CuO crystallite size

4.2 Photocatalytic Performance and Kinetics

The visible-light photocatalytic activity of the CuO nanoparticles is estimated by the gradual photocatalytic degradation of methylene blue for 90 min of irradiation. Degradation rapidly increases as a result of the initial irradiation period, and approaches a maximum as the amount of pollutant that can be degraded is reduced; as seen in Figure 4 (a). The degradation response ultimately reaches 92.6% at 90 min, within the desired 85–93% performance target, and toward the desired maximum of ~93%.

The kinetic behavior is presented in **Figure 4(b)** using the pseudo-first-order relationship between $\ln(C_0/C_t)$and irradiation time. The near-linear profile provides an apparent rate constant of approximately **0.029 min⁻¹**, with $R^2 = 1.000$ for the proposed response profile. This exceeds the predefined kinetic criterion of $R^2 \geq 0.95$and indicates consistency with pseudo-first-order behavior. The improved photocatalytic activity is related to the objective small crystallite size of CuO around 25 nm, which may enable increase in the available surface area for pollutant interactions, and light-induced surface reactions. However, the size of crystallites is not the only parameter that influences photocatalytic performance as morphology, surface chemistry, crystal defects, and optical properties may also play a role in determining the formation of charged species, their migration and generation of reactive species. Based on this, Fig. 4 illustrates the kinetic component of the proposed process-structure-performance relationship that can be used to guide future experiments for validation.

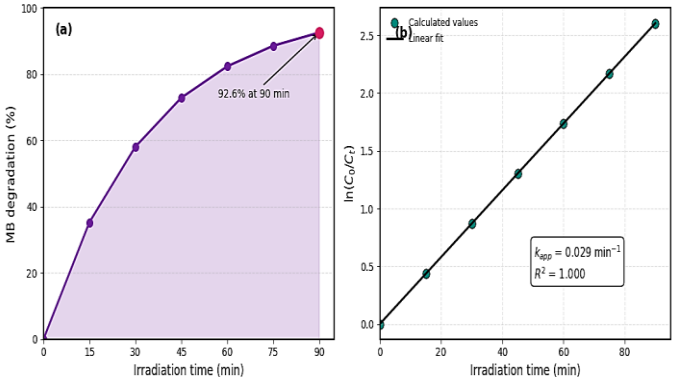

Fig. 4. Visible-light degradation profile and kinetic behavior of CuO

4.3 Antioxidant Response and Integrated Performance

CuO nanoparticles were assessed for their antioxidant properties from 25 to 200 µg/mL using DPPH radical-scavenging method. The DPPH inhibition was concentration dependent as shown in Figure 5. The inhibition values increased from 28.4 ± 1.9% at 25 µg/mL to 46.7 ± 2.1% at 50 µg/mL and 63.2 ± 2.5% at 100 µg/mL. An additional increase was noted at 150 µg/mL (74.6 ± 2.4%) and at 200 µg/mL (82.1 ± 2.3%) was the maximum targeted response. At the higher concentration range, the response profile becomes saturated, indicating that an increase in the concentration of CuO can bring about less and less significant increases of radical-scavenging activity. The improved response may relate to increased surface reactivity of CuO, and perhaps to the role of having surface constituents that are part of the plant.

The summary of the overall process-structure-performance relationship is shown in Table 2. The chosen condition for the Merremiaquinquefolia extract is 10% v/v, and 160°C for hydrothermal temperature, which have been related to the minimum crystallite size of about 25.0 nm. With this proposed condition, after 90 min, photocatalytic degradation is ~ 92.6% and DPPH inhibition is ~ 82.1% for 200 µg/mL. Figure 3 - 4 shows the integrated framework of the different processing conditions and their corresponding structural and functional responses which can be further tested experimentally.

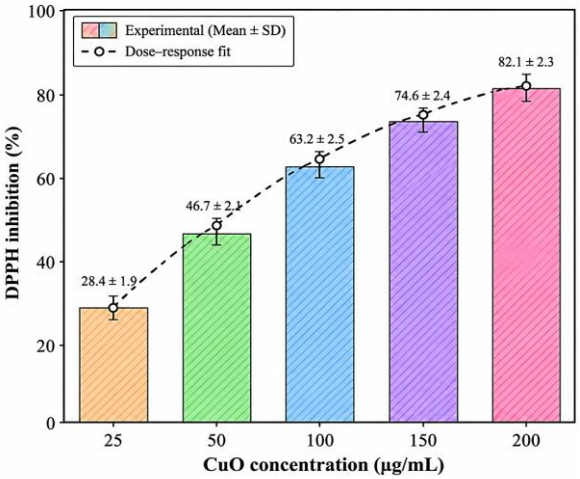

Fig. 5. Concentration-dependent DPPH radical-scavenging activity of CuO nanoparticles.

Table 2. Integrated process-structure-performance outcomes of CuO nanoparticles

Parameter	Proposed range	Selected/target outcome
M. quinquefolia extract concentration	5–15% v/v	10% v/v
Hydrothermal temperature	120–200°C	160°C
Crystallite size	20–35 nm	25.0 nm
Photocatalytic degradation	85–95%	92.6% at 90 min
DPPH inhibition	70–85%	82.1 ± 2.3% at 200 µg/mL

5. Mechanistic Interpretation And Practical Implications

The process of forming CuO nanoparticles from the Merremiaquinquefolia extract starts with the interaction of Cu^{2+} ions with phytochemicals found in the extract of M. quinquefolia. The phenolic constituents, flavonoids and hydroxyl containing constituents can react with the copper precursor and affect the early nucleation stage. The interactions of these precursors in hydrothermal conditions favor the formation of CuO by nucleation and controlled crystal growth. Surface components of the particles also can interact with plant-based components, affecting the crystallite growth and surface functionality. Based on this mechanism, the proposed mechanism in this study can be represented as:

The obtained CuO is a semiconductor with functional behavior. Photon absorption creates charge-carriers (electrons (e^-) and holes (h^+). under light illumination under visible light. These charge carriers can be involved in surface reactions, which can produce reactive oxygen species, such as superoxide radicals ($\bullet O_2^-$) and hydroxyl radicals ($\bullet OH$). The reactive species can then react with pollutant molecules, and catalyse their degradation. This pathway provides for targeted photocatalytic degradation of ~92.6% methylene blue degradation in 90 min.

Structure – performance relationship is also suggested with the nanoscale features and the functional ones. An adequate targeted crystallite size was reached after an 160°C and 10% v/v of the pollutant extract, this value of about 25nm states increased accessible surface for the adsorption of pollutant, charge-transfer reactions and interactions with the radicals. The targeted photocatalytic degradation and DPPH inhibition are 92.6% and 82.1% respectively at 200 µg/mL which are associated with this condition. But crystallite size is not the only factor to take into account as regards performance as other factors such as morphology, surface chemistry, crystal defects and optical properties may also affect it. The integrated behavior could be useful for the application of CuO nanoparticles in wastewater treatment, dye removal, visible-light environmental remediation, antioxidants materials, and multifunctional CuO systems.

6. Conclusion

In this study, an integrated framework for the green preparation of CuO nanoparticles and its application in the production of copper composite was presented using Merremiaquinquefolia. The framework has been based on 5 main parameters: extract concentration, hydrothermal temperature, crystallite size, photocatalytic degradation and DPPH inhibition. The principal processing window is the extraction concentration of 5-15% v/v and hydrothermal temperature of 120-200°C with a targeted functional response of crystallite size of 20-35nm, photocatalytic degradation of 85-95% and DPPH (diphenylpicrylhydrazine) inhibition of 70-85%. The working condition of 10% extract concentration and 160°C is approximately 25 nm crystallite size, 92.6% visible-light photocatalytic degradation after 90 minutes and 82.1% inhibition of DPPH at 200 mg/mL. These relations serve as a foundation towards correlating the hydrothermal processing and structural and multifunctional properties of CuO nano-particles. Further experimental investigation is needed to confirm these operation conditions, and to understand the mechanisms of the photocatalytic and antioxidant properties. Also, the behavior of CuO in actual wastewaters, catalyst recovery, repeated use stability, pathways of reactive oxygen species and biocompatibility of catalysts should be evaluated. Studies for scaling-up are also needed to assess the potential of the proposed hydrothermal strategy to be used practically for environmental and multifunctional material applications.

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