



A Sight of View on Hydrothermal Synthesis of Copper Oxide

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HIGHLIGHTS

- Considerable focus has been attached to the synthesis of copper oxide nanoparticles
- Copper oxide is used in a wide range of applications such as solar energy and gas sensors
- A high-score scientific manuscript that used copper oxides as electro-optical applications has been studied

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ABSTRACT

In this study, attention was directed to the preparation of nano-copper oxide films and their multiple applications, where a group of recent researchers was presented and compared to reach the optimal phase, then the optimal application, and finally the optimal values in the optimal application. Where metal-oxides are significant technology used in device fabrication in electronic and chemical industries, due to their applications in advanced technologies, which have been attached to the synthesis of copper oxide nanoparticles. These materials present different properties depending on molecular structure. Copper oxide is used in a wide range of applications. The copper oxide phases Cu_xO are interesting materials, because of their electrical, optical, and thermal properties. Nano-structuring technologies can enhance the performance of this material and produce it with unique properties that don't exist in its bulk form. Copper oxide could be found in three different distinct phases, this includes CuO , Cu_2O , and Cu_4O_3 , which can be prepared by various synthesis techniques, depending on chemical and physical roots. The most two common Cupric oxide phase is the CuO formula, an inorganic material with black color, it represents one of the other two oxides of copper, the second being Cu_2O or cuprous oxide with brownish semiconducting properties. Using A technique to prepare the same material with different phases required physical engineering, where the effect of different parameters plays a role. In this article, the chemical and physical properties of particles using hydrothermal technology for the synthesis of copper oxide were briefly reviewed.

1. Introduction

In recent years and during the process of progress of material science, thin films, and metal oxide thin films have received much attention [1]. The current demand for excellent product performance, easy operation, nontoxic, and fewer pollution methods for the synthesis of thin film has increased [2]. However, many limitations to obtaining the required purity, particle size, and morphology of powder using the solid phase method [3]. The liquid phase method mainly includes different methods such as precipitation [4,5], hydrothermal [6,7], hot injection [8,9], and innovative approaches using sol-gel [10,11]. Hydrothermal processing has been extensively applied to prepare these structures. For metal oxide nanostructure synthesis metallic oxide via hydrothermal technique has gained much attention owing to its advantage in preparing highly monodispersed nanoparticles having carefully tailored sizes and shapes [12]. The convenience of operation, ease of the synthesis process, and adjustable particle size are benefits of the liquid phase approach [13]. Hydrothermal methods are widely used in the synthesis of specific materials with different morphological characteristics. According to the vapor pressure of the primary component in the reaction, either low-pressure or high-pressure conditions can be employed to synthesize distinct morphologies of the materials to be synthesized for micro and nanostructure, where temperature control kinetics and thermodynamic stability of the product, while the pressure influenced solubility, supersaturation rang stipulation crystallization process and the thermodynamic stability of the product [14,15]. Both chemical and physical parameters are varied steadily during processing such that there are establishes a direct relation between precursor-product.

Different parameters can be influenced by the synthesis material. In a hydrothermal method, materials can be synthesized in a variety of temperatures, from ambient temperatures to extremely high temperatures [16]. Today, hydrothermal technology

is used in a variety of scientific and technological sectors, including materials science, earth science, metallurgy, physics, chemistry, biology, etc. This method has been used to create a variety of copper oxide materials with different phases for different applications due to its characteristic. Intrinsic imperfections, such as copper and/or oxygen vacancies, primarily govern the optical and electrical properties of copper oxide. Copper oxide could be found in three different distinct phases, this includes CuO, Cu₂O, and Cu₄O₃, which can be prepared by various synthesis techniques. The most two common Cupric oxide phase is the CuO formula, an inorganic material with black color, it represents one of the other two oxides of copper, the second being Cu₂O or cuprous oxide with brownish semiconducting properties. The p-type semiconductor nature of Cu₂O is thought to be caused by copper vacancies [17]. When the density of oxygen vacancies is larger than that of copper vacancies, an n-type character is seen. [18].

This essay introduces the reaction processes and reviews the evolution of hydrothermal methods for producing copper oxide. The morphology's effect from the parameter and its characteristics.

2. Hydrothermal Method

Simply said, hydrothermal synthesis is a single-crystal synthesis process that relies on a mineral's solubility in hot water at high temperature and pressure conditions [19]. The reaction solution is placed in an apparatus called an autoclave, a pressure vessel made of steel or Teflon, to carry out the synthesis process. The growing chamber's opposite ends are kept at different temperatures. The nutrient solute dissolves at the hotter end while it is deposited on a seed crystal at the cooler end, where it grows the desired crystal [20]

The hydrothermal strategy means the production of materials by chemical processes that occur in a sealed reaction chamber at elevated temperatures and pressures, typically exceeding 100°C and 1 bar [21]. A material that is weakly soluble under ordinary situations is dissolved and recrystallized using this technique [22]. On stainless steel, carbon steels, magnesium alloys, aluminum alloys, pure copper, and copper oxide, biomaterials, and antifouling micro-nanostructures have recently been created using the hydrothermal approach [23].

Temperature can obviously affect the rate of nucleation and crystal growth. The rate of nucleation and the linear rate of crystal growth is shown as follows [24] :

$$DN/dt = A [\exp (Et)^{-1}] \tag{1}$$

Where DN/dt is the growth rate, A, and E is the nucleation coefficients, and t is the time all increase with temperature, indicating linear rates of crystal growth.

Figures 1(a) and (b) demonstrate, respectively, the conventional hydrothermal synthesis equipment and general processes involved in hydrothermal synthesis:

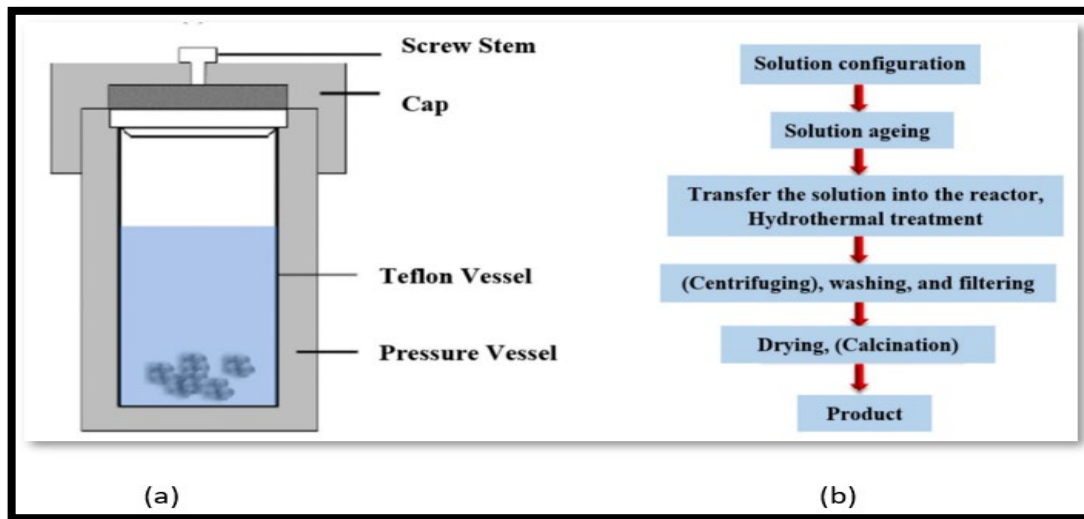


Figure 1: Hydrothermal synthesis: (a) schematic diagram of typical apparatus, and (b) typical procedures involved [7]

Today, a variety of materials, including bulk crystals and particles from small to very small with regulated sizes and morphologies, are processed using the hydrothermal approach. When compared to traditional techniques of material processing, the hydrothermal approach has several benefits [25]. In order to evaluate the applicability of the solution-based hydrothermal synthesis in regard to different reducing agents and additives, the yield of each reaction was calculated using the following Equation [26]:

$$\text{Yield (\%)} = (\text{mass of precursor})/(\text{mass of "as obtained" material}) \tag{2}$$

The following Figure 2 shows the development of a hydrothermal method for different applications.

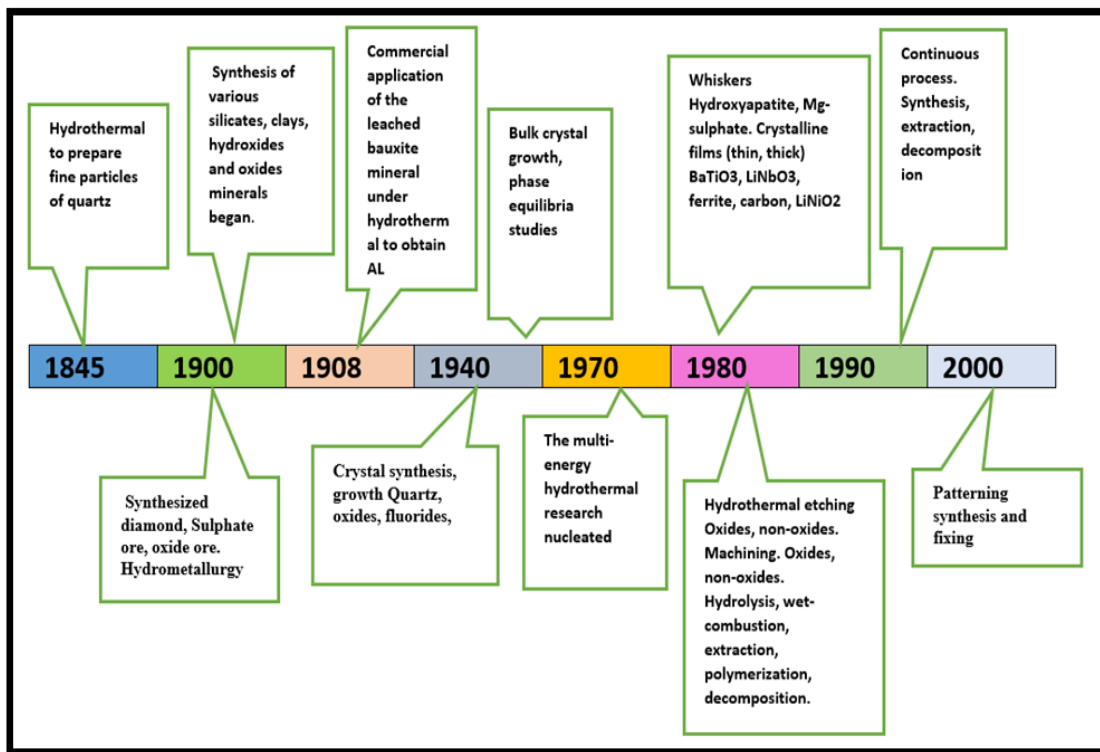


Figure 2: Shows hydrothermal development from 1845 to 2000

There has been much research published on the hydrothermal method on different topics and different applications as shown in Figure 3.

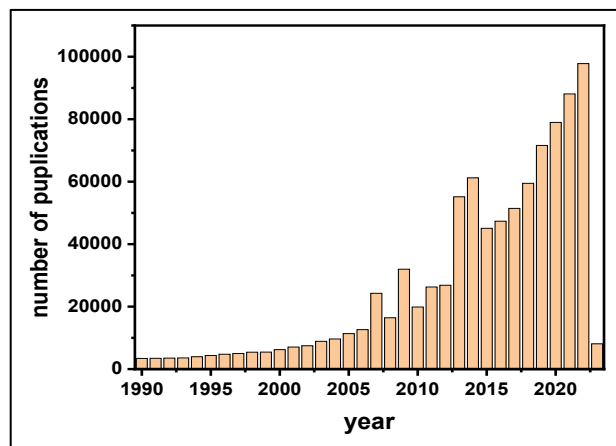


Figure 3: Number of publications of hydrothermal per year

The number of publications increased with the year indicating the importance of hydrothermal this importance is due to the different morphology that can be obtained by tailoring the precursor used and another parameter like pH, time of the experiment, temperature, etc. In recent years, many research studies was achieved on p-Type oxide semiconductors [27-29]. In the last five years, p-type oxides have experienced improved stability and a significant increase in the switching ratio of devices. A small number of oxides have unusual energy band structures that reduce the oxygen's binding impact on hole carriers, improve the mobility of transmitters, and enable the achievement of p-Type conductivity in the material. The p-type oxide semiconductor has so far been extensively researched, with NiO, SnO, and copper oxide among the materials [30].

3. Copper Oxide Phase

Copper oxide has favorable chemical and physical stability, is relatively inexpensive, and is simple to combine with polymers and polarized liquids (such as water). The two elements copper and oxygen combine to form copper oxide. Figure 4 shows different phases of copper oxide based on the correlation of copper to the oxygen. Copper oxide could imply one of the following phases [31]:

- Cuprous oxide(Cu₂O)

- Cupric oxide(CuO)
- Copper (III)oxide(Cu₂O₃)
- Copper (IV)oxide(CuO₂)
- Copper peroxide(CuO₂)

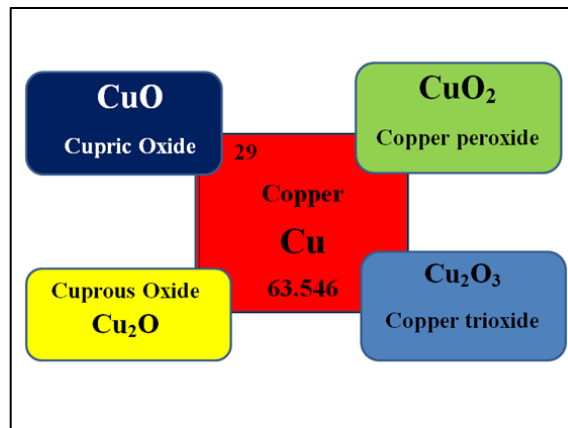


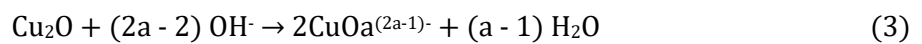
Figure 4: Copper oxide phases

Cuprous oxide, and cuprite oxide with their properties such as bad gap energies, absorption coefficients, lattice structure, and resistivity show simply by the following Table 1:

Table 1: Copper oxide phase

Copper oxide phases	Eg eV	Absorption cm ⁻¹	color	Structure	Resistivity Ω·cm
Copper(I) oxide (cuprous oxide, Cu ₂ O)	1.8–2.7	10 ⁵	red	cubic	(3.7 ± 0.3) × 10 ³
Copper(II) oxide (cupric oxide, CuO)	1.3 - 2.1	>10 ⁴	black	monoclinic	(1.6 ± 6) × 10 ¹

Based on how copper and oxygen combine to generate the copper oxide phase [32]. Due to its inexpensive fabrication method, the availability of copper in the crust of the earth, and its non-toxic properties, Cu₂O is one of the potential candidates for a variety of uses. Devices based on p-n junctions, such as diodes and transistors, solar cells, and optoelectronics have been made possible by semiconductor thin film technology. many low-temperature thin film deposition methods, such as the electrodeposition process, anodic oxidation, oxidation/reduction, and thermal oxidation, and high-temperature methods, such as the hydrothermal method, have been used to synthesize Cu₂O nanocrystals with a variety of structures and surface morphologies [33]. Kuzmina and Kaidukov (1977) have worked out a detailed technology of hydrothermal synthesis of cuprite, and the solubility of cuprite can be written as following Equation [34].



A semiconducting substance with a monoclinic structure is a cupric oxide (CuO). The family of copper compounds' simplest component (CuO) has drawn particular interest because it possesses several potentially practical physical characteristics, including high temperature, superconductivity, electron correlation effects, and spin dynamics [35].

A wide range of methods was used for the formation of copper oxide thin film like electrodeposition, in which some strategies include controlled deposition of material on conducting surfaces using the electric current from a solution containing ionic species, that have been employed to regulate the surface shape and orientation [36]. Other techniques for synthesizing orientated copper oxide films include polyol approaches [37]. However, to activate and regulate the formation of crystals and enhance the surface morphology of the films, surface capping agents, oxidants, reductants, and other additives must be added throughout these synthesis processes. Therefore, it would be advantageous to create a straightforward and safe manufacturing method for copper oxide films with adjustable shapes [38]. The study's Cu₂O synthesis and CuO by hydrothermal method at different parameters were discussed.

4. Hydrothermal Processing Parameter for Synthesis of Copper Oxide

4.1 Hydrothermal Based on The Time and Temperature

The manufacturing process is positively impacted by temperature when carried out within a specified temperature range. Based on application data, a maximum temperature has been proposed. But the temperature can also affect the kind of product that must be produced. As the amount of water in the liquid phase cause to rise in temperature, more dense products begin to crystallize. The rate of nucleation and film growth may, of course, be influenced by temperature.

In 2015 Xiao-Lin Luo et al used sodium tartrate as a chelating agent, and Cu_2O micro-crystals of various morphologies (including three branching structures) were created by the hydrothermal process. Temperature and the quantity of sodium tartrate have a significant impact on the branching growth patterns of Cu_2O microcrystals. The sample was prepared at different temperature and different amount of sodium tartrate and poly(vinylpyrrolidone) PVP was added with stirring until PVP was absolutely dissolved for preparing cuprous oxide [39].

The temperature has a large effect on the morphology of synthesis copper oxide where this can be observed in the research in 2016 by Xishun Jiang, et al, they prepared Cu_2O by the hydrothermal method using $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, NaOH, $\text{CH}_3\text{CH}_2\text{OH}$, CH_3COCH_3 , CuSO_4 , glucose, and NaOH. The temperature is controlled at [70-130 °C] for 2.5 h. The X-ray analysis shows the presence of Cu_2O , and different crystal size for different temperature has been determined. This shows the effect of the temperature on the morphology and the application. The samples formed at a reaction temperature of 130°C have an average absorption coefficient that is higher than those deposited at 70°C. Due to its smaller particle number and greater particle size, the sample produced at 130°C can absorb light of longer wavelengths. [40].

In 2017 Due to their high surface-to-volume ratio, low density, and adequate interior space for possible applications in some fields, such as gas sensing and photocatalysis, open-hollow-nanostructured Cu_2O has drawn a lot of interest. The Cetyltrimethyl Ammonium Bromide (CTAB) and detergent temperature were key factors in this preparation method for the creation of the Cu_2O open hollow nano-spheres at 120°C for 24 h. The resulting sample is separated into three sections (samples A, B, and C), after which the obtained samples are repeatedly washed with distilled water and ethanol at various temperatures (40°C, 20°C, and 60°C, respectively). A solid sphere with a diameter of 0.8–1 μm is observed in the SEM and TEM images of sample B as a result of the influence of detergent temperature, whereas sample A has irregular pieces with a diameter of around 500 nm. Based on these findings, it can be assumed that the detergent temperature plays a significant role in the production of the Cu_2O open hollow spheres and that the detergent temperature of 40°C is the most favorable for the formation of the open hollow spheres [41].

Chirag Mevada in 2017 demonstrates the impact of temperature and precursor concentration on the shape of copper oxide produced hydrothermally on glass surfaces. The appearance and size of the produced CuO microstructures were shown to be significantly influenced by the heating duration and precursor concentration. Copper sulfate, acetate, chloride and. Aqueous ammonia is used for synthesized copper oxide. Microstructures like 3D flowers were created using nanorods and nanoplates. At a constant pH and duration, the deposition temperature ranged from 70°C to 105°C. Therefore, when the number of nuclei rose and further aggregated to lower the surface energy of the system, increasing the temperature from 70°C to 105°C caused an increase in the size of the structure. Jia et al(2009) found that the size of the resulting structure changed from a nanosphere to a microsphere as the hydrothermal treatment temperature was raised from (90°C to 105°C) [21]. When the precursor's initial concentration was 12 mM, a CuO sphere with a diameter of around 1.5 μm was produced. Additionally, only part of the assemblages fully developed into spheres when the precursor concentration was increased from 12mM to 40mM while maintaining the same pH and temperature [42].

Binxia Yuan, et al in 2019 the flower-like Cu_2O had been produced in distilled water at a very low reaction temperature when the reaction temperature was adjusted between (50 and 200°C). This offered a very straightforward and inexpensive approach to produce flower-like Cu_2O , which would have enormous promise for the synthesis of other metal chalcogenides. Elementary copper also showed up in the sample as it was produced. Optical properties revealed the flower-like Cu_2O by UV-VIS absorption spectrum. It comprised various absorption peaks. They calculated the band gap of the flower-like Cu_2O to be 1.88 eV based on the tangent graph of $(\alpha h\nu)^2$ vs. $h\nu$. On the other hand, the formula ($E_g = 1240/\lambda_{\text{max}}$, λ_{max} is the first exciton peak) could be used to determine the band gap with absorption peak, which was 2.26 eV. The sample was more suited for the creation of a photovoltaic device based on the absorption curve, which revealed that the absorption performance of the ultraviolet and visible light was significantly higher than that of the near-infrared area [43].

In 2020 Metal-oxide via a hydrothermal process heated to different temperatures. CuO nanoparticles were shown to have a single-phase monoclinic structure using X-ray diffraction analysis (XRD), with an average grain size that increases with heating temperature. The maximum transmittance of the three was shown by CuO nanoparticles produced at the highest temperature (150°C). To demonstrate that the band gap increases as the temperature rises, the bandgap was estimated using UV-VIS measurements [44].

In 2022Martha Claros et al. developed a simple and repeatable hydrothermal synthesis of copper oxide in this work. The findings indicate that the hydrothermal process is primarily driven by reaction time, reducing agent concentration, and temperature. After the hydrothermal processing for the gas sensor application, two different annealing temperatures (205 and 450°C) were evaluated to see their impact on the shape, chemical content, and crystal size of the nanowires [45].

Additionally, Shahroz Saleem in his 2022 work used the hydrothermal technique to create (CuO) specimens at three different calcination temperatures: 350, 450, and 550°C. For use in optoelectronic device applications, the structural, morphological, vibrational, functional group, and optical characteristics of CuO were examined using UV-VIS spectroscopy. With the increase in calcination temperature, the pure single-phase monoclinic structure, with no impurity phases of produced samples and excellent crystallinity are confirmed by XRD. The increased nucleation and growth processes brought by the increased calcination temperature lead to an increase in the crystallite size. Also, by raising the calcination temperature, the parameters d-spacing, lattice constant, unit cell volume, porosity, dislocation density, and microstrain were reduced. Due to an increase in crystalline size at the maximum calcination temperature, a little decrease in dislocation density and microstrain was observed in CuO samples. According to optical research, CuO optical characteristics are enhanced by high calcination temperature by adjusting its optical bandgap [46]. All these details clarified above was mention in the Table 2 to show the

experimental parameter at different condition which include experiential time, precursor, structure, application, temperature, parameter, and product.

Table 2: Shows hydrothermal based on the temperature

Experiential time (h)	precursor	Structure	Application	Temperature °C	Parameter	Product	Ref.
4	Copper acetate hydrate [Cu(OAc)2H2O, AR], sodium tartrate (AR), AP (AR). Poly(vinylpyrrolidone) (PVP, K30) Milli-Q water	cubes	Study	160 175 190 200	amount of sodium tartrate and temperature	Cu ₂ O	[39]
2.5	CuSO ₄ .5H ₂ O, NaOH, CH ₃ CH ₂ OH, CH ₃ COCH ₃ , CuSO ₄ , glucose and NaOH	round-likely cubes to octahedral	Study photocatalysis performance	70, 90 110 and 130	Change temperature	Cu ₂ O	[40]
24	CuSO ₄ and CTAB (Cetyltrimethyl Ammonium Bromide), deionized water: ethanolamine NaOH	open hollow nanospheres	gas sensing	120	Cleaned many times with distilled water and ethanol at different temperatures (40 °C, 20 °C and 60 °C),	Cu ₂ O	[41]
3	Copper sulfate, acetate, chloride and. Aqueous ammonia	flower-like microstructures	study	70 105	Change time and temperature	CuO	[42]
2 6 12 24 48	Copper acetate, copper nitrate, copper sulfate glucose, 'Hexadecyl trimethyl ammonium bromide (CTAB), Polyvinyl Pyrrolidone (PVP), and ethanol amine'	flower-like Cu ₂ O	Study	50 90 110 150 200	Temperature and time	Cu ₂ O	[43]
12	(CuCl ₂ . 2H ₂ O), ACS grade, NaOH	Different morphology	Study	105 120 150	Change temperature	CuO	[44]
10	copper acetate, salt, and deionized water. And pyrrole	fine and long nanowires	Gas sensing	150	changes in temperature and concentrations	Cu ₂ O	[45]
6	sodium hydroxide (NaOH) and copper sulfate (CuSO ₄ .2H ₂ O) and double-distilled	monoclinic structure	optoelectronic device	200	Different calcination temperature; 350, 450, and 550°C.	CuO	[46]

4.2 Hydrothermal Based on Precursor and Concentration

The precursor variation has a significant impact on the crystallization processes right down to the level of nucleation and crystallization kinetics, the type of crystallization material, the content and distribution of the lattice, and the size and shape of the crystals. Due to the highly complex nature of the chemical precursors, this is one of the most challenging components of the synthesis process and plays a significant role. Lithium hydroxide, sodium hydroxide, and potassium hydroxide can be used to create cuprite monocrystals under hydrothermal conditions in aqueous solutions of alkali ammonium halogenides. [47].

In 2016, they constructed the 3D rose-like Cu₂O nanoflowers with an average diameter of 200-250nm, which display rose-like nanoflowers built by many nanosheets, and investigate their gas sensing characteristics utilizing cetyltrimethyl ammonium bromide (CTAB). The CTAB is essential for creating these distinctive nanoflowers that resemble roses. A unique growth process is also shown clearly. This demonstrates how (CTAB) has an impact on the device's morphology and functionality [48].

The hydrothermal reaction-based one-step synthesis of cuprous oxide nanostructures by Aziz GEN was reported in 2018. Using copper(Cu) acetate monohydrate as the copper source, glucose as the reducing agent, and polyvinylpyrrolidone(PVP) as the detergent in basic aqueous systems at 140°C for 6 h, nanoflower-like Cu₂O with average diameters of around 230 nm is produced. Investigations on the impact of the NaOH content on the shape of synthesized structures produced hexapods when no NaOH was employed and submicron-sized octahedra when the NaOH level was raised [49].

Zimbovskii reported in 2019 that by hydrothermally treating copper substrates in aqueous NaOH solutions of different concentrations, Cu₂O films were created on the surface of metallic copper. It has been demonstrated that hydrothermal processing of copper in an oxygen-saturated 0.3 M NaOH solution at 180°C for 1 h results in the production of Cu₂O thin films

with the maximum thickness on the surface of metallic copper. Analytical-grade NaOH solutions were used for the hydrothermal synthesis, which was conducted for one hour at a temperature of 180 °C with concentrations ranging from 0.05 to 3 mol/L. The cell had a 70% fill factor. They prepared samples in different concentrations of NaOH solutions and discovered that the quantity of copper(I) oxide that formed was a nonlinear function of alkali concentration. This was caused by the competition between two processes: film growth and dissolution. Cu₂O content rises to 0.3 M with increasing alkali concentration [50].

Cu₂O micro-sized crystals were created in 2021 by Xudong Chen et al. using a hydrothermal reaction between CuCl₂ and NaOH solution, ascorbic acid (AH₂) as a reductant, and CTAB. Along with the rising molar ratio of NaOH/CuCl₂, the shape varied from a truncated octahedral to six corners truncated octahedral. The concentration of [Cu(OH)₄]²⁻ influenced the growth rates, which led to morphological change. A quasi-spherical Cu₂O structure is created by an etching action caused by the presence of enough AH₂. Higher adsorption is seen in the extremely truncated octahedral Cu₂O with the predominate {1 1 0} faces. The SEM pictures make it easy to discern between various morphologies. The concentration of sodium hydroxide can be used to explain the morphological changes [51].

Johannes Seidler demonstrated in 2021 that the size and shape of the Cu₂O crystals influence the reactivity and catalytic efficiency of cuprous oxide (Cu₂O) particles. This work uses non-toxic, inexpensive carboxylic acids (O, O-type) and amino acids (N,O-type) as structure-directing agents and new precursors to produce innovative, homogeneous cuprous oxide microcrystals by a simple, mild hydrothermal process. In comparison to O,O-type ligands, N,O-type complexing agents showed a greater capacity to decrease the copper species. Purified Cu₂O was produced using the oxalate and citrate complexes after 4 hours at 200°C; however, the citrate complex, used as a precursor, produced octopods that resembled flowers rather than evenly stepped microcubes [52].

CuO thin films with a broccoli-like structure were created in 2022 by Nguyen Hoang Lam et al using a hydrothermal technique for a water-splitting application. By adjusting the precursor concentration and reaction temperature, CuO's structure and morphology may be managed. The substance contains sodium hydroxide, acetic acid, and copper (II) acetate hydrochloride. The glass slide with fluorine-doped tin oxide coating served as the substrate (FTO glass). Cu(C₂H₃O₂)₂·H₂O was employed in two distinct concentrations (0.1M and 0.5M) and at two different temperatures C the synthesis of the material was essential in determining how the bandgap changed. CuO thin films' photoluminescence spectra are presented as two emission peaks at 399 nm (purple) and 496 nm (green) in these spectra [53]. The effect of precursor and concentration on the hydrothermal method show in the Table 3.

Table 3: Hydrothermal based precursor and concentration

Experiential time (h)	precursor	Structure	Application	Temperature °C	Parameter	product	Ref.
4	CuCl ₂ ·2H ₂ O and CTAB, deionized water, NaOH	rose-like Cu ₂ O nanoflowers	Gas sensing	340	Change pH value	Cu ₂ O	[48]
6	Cu source, Copper Acetate Monohydrate (C ₄ H ₆ CuO ₄ ·H ₂ O), distilled water, D(+)-Glucose and Polyvinylpyrrolidone (PVP, (C ₆ H ₉ NO))	hexapods and submicron-sized octahedra	Study characteristic	140	The effects of the NaOH content on the morphology	Cu ₂ O	[49]
1	isopropanol and acetone, hydrochloric acid solution, and distilled water. NaOH	polyhedral	Study	180	Change concentration	Cu ₂ O	[50]
9	CuCl ₂ , NaOH solution, and acid (AH ₂)	truncated octahedral to six corners-truncated flower-like octopods	visible-light photocatalytic	lower reaction temperature	Changing concentration of NaOH/CuCl ₂ and pH ₂	Cu ₂ O	[51]
4	“(O,O-type) and amino acids (N,O-type) as structure-directing agents. N,O-type complexing”			200	Change structure directing agents.	Cu ₂ O	[52]
12	opper (II) acetate hydrate (Cu(C ₂ H ₃ O ₂) ₂ ·H ₂ O, 98%), acid acetic (CH ₃ CO ₂ H, ≥99%), and sodium hydroxide (NaOH, ≥98%). fluorine-doped tin oxide-coated glass slide (FTO glass).	Broccoli-like Structured	Photoelectrochemical Water Splitting	100 150	varying the precursor concentration and reaction temperature	CuO	[53]

5. Alkalinity (pH)

The media's alkalinity is crucial for film development, material synthesis and preparation, and processing in general. As the primary mineral agent, anions (OH⁻) have an impact on the supersaturation, kinetics, morphology, shape, size, and crystallinity of particles and materials. The reactants and their concentrations/ratios have the biggest impact on pH, also, affected by temperature and time. Additionally, the pH changes quickly in the system when organics are introduced, making pH the most important factor in determining the crystallization rate. Generally speaking, an increase in OH concentration will speed up crystal formation and reduce the time needed for induction before viable nuclei form [54].

In 2020, M. Ozga In this study used a modified hydrothermal technique to demonstrate the very quick development of polycrystalline CuO thin films. The synthesis process was achieved at the open system, low temperature (below 100°C), and a short time of about (6min-38sec). The essential components in the reaction solution are deionized water and copper (II) acetate. For pH, the regulator has usually utilized sodium hydroxide (NaOH). The technique enhancement allowed for a controllable film thickness. It was showing how several factors, including pH, heating force, processing time, and Cu(II) concentration, affect layer thickness. Monoclinic CuO is present in the resulting films. The layers' structure is polycrystalline, and they contain nanocrystallites of 7–12 nm. According to transmittance calculations, the optical energy band gap lies between 1.8 and 1.87 eV. The thickness increase as the PH decreases and as the temperature increase as the power decrease. The disclosed inventive technology has great promise for numerous applications, particularly photovoltaic ones. This method is very important and competitive with existing technologies due to its ease of use, low cost, and great efficiency [55].

Kexing Liu synthesizes Cu₂O in 2022 by electrochemically splitting water in an acetate solution with hydrothermal as the photocathode. A systematic investigation was done into how pH affected the production of Cu₂O throughout the formation. It has been discovered that the performance of the Cu₂O photocathode and the properties of the films were related to the pH of the solution. The outcomes demonstrated that solution pH played a significant role in producing consistent Cu₂O films. As the Cu²⁺ concentration in the acetate solution decreased, the Cu₂O stability region shrank. As the pH value increases this caused it to remain Cu₂O stable. By adjusting the pH levels, Cu₂O films' homogeneity and microstructure can be changed. The autoclave was preheated for 4 hours to 200°C. The stability and activity of Cu₂O fluctuated with the pH of the solution, in which the Cu⁺ ion is a crucial and essential intermediary, therefore the impact of pH on the creation of Cu₂O was taken into consideration from the perspective of thermodynamics [56]. Due to its low solubility, Cu⁺ ion saturation is thought to be the cause of Cu₂O production. Due to the continual consumption of OH⁻ ions, the solution pH then dropped while the hydrothermal synthesis process continued [57]. The following Table 4 show how hydrothermal method affected by Alkalinity and their effect on the preparing sample and its characteristics.

Table 4: hydrothermal based pH concentration

Experiential time	precursor	Structure	Application	Temperature °C	Parameter	product	Ref.
38sec-6 min	“Copper (II) acetate monohydrate (Chempur), deionized water and NaOH”	monoclinic	study	90	pH	CuO	[55]
4 h	“Cu(CH ₃ COO) ₂ ·H ₂ O, NaOH and Polyvinylpyrrolidone Na ₂ SO ₄ and NaOH and Cu sheets”.	cubic grains	study	200	pH	Cu ₂ O	[56]

6. Conclusion

Hydrothermal method-based copper oxide has a lot of interesting in the recent year as a result of tuning different parameters which can strongly influence the morphology, and chemical, and physical properties. The parameter plays an important role to obtain the desired structure like the change in temperature of the experiment, also the temperature of the annealing process after the hydrothermal process, the calcination process, and the temperature of the solvent used to clean the sample. The precursor and material used several materials used like CTAB, NaOH, KOH, etc. The pH also affected the hydrothermal process of synthesis of copper oxide which can be changed by several additions like NaOH also by stirring the solution before the hydrothermal process. The time of the experiment and the stirring also play an important role in hydrothermal synthesis.

Author contribution

All authors contributed equally to this work.

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Data availability statement

The data that support the findings of this study are available on request from the corresponding author.

Conflicts of interest

The authors declare that there is no conflict of interest.

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