

Asian Journal of Chemistry; Vol. 35, No. 11 (2023), 2618-2630

https://doi.org/10.14233/ajchem.2023.30223

## REVIEW

# Polyol Synthesis of Nanoparticles: A Decade of Advancements and Insights

RASHMI KHULBE<sup>1,\*,0</sup>, ASHA KANDPAL<sup>1,0</sup>, JAGDISH PRASAD<sup>2,0</sup>, BHUWAN CHANDRA<sup>1,0</sup> and N.D. KANDPAL<sup>1,0</sup>

<sup>1</sup>Department of Chemistry, Kumaun University, S.S.J. Campus, Almora-263601, India <sup>2</sup>Department of Chemistry, H.S.B. Government P.G. College, Someshwar-263637, India

\*Corresponding author: E-mail: rashmikhulbe00@gmail.com

Received: 13 August 2023;	Accepted: 24 September 2023;	Published online: 31 October 2023;	AJC-21416
---------------------------	------------------------------	------------------------------------	-----------

This article presents a focused overview of advancements in the field of polyol synthesis of metal, metal oxide nanoparticles and nanocomposites, specifically those reported in the current decade. The aim of this article is to present the pertinent physico-chemical factors associated with nanoparticle synthesis in polyol solutions, highlighting their applications in both biological and industrial domains. The scope of this review article is selective rather than exhaustive, concentrating on noteworthy contributions to polyol nanoparticle synthesis that hold potential for refining synthesis methods. To comprehend the role of polyols in nanoparticle synthesis, an examination of the distinct physical properties of various polyols is crucial. Moreover, understanding the particular functions and mechanisms governing nanoparticle formation is imperative in this context.

Keywords: Polyol, Metallic nanoparticles, Bimetallic nanoparticles, Metal oxide nanoparticles, Nanocomposites.

## **INTRODUCTION**

Polyol has long been recognized as both a reducing and stabilizing agent in the synthesis of metallic and metallic oxide nanoparticles. However, significant advancements in polyol-based nanoparticle synthesis have transpired not merely over the last few decades. These advancements have undergone multiple rounds of review by various researchers specializing in polyol-based nanoparticle synthesis [1-6]. Among the various studies conducted, only a handful have been deliberated within the context of specific physico-chemical conditions governing nanoparticle synthesis. Exploring nanoparticles through the polyol method might introduce entirely novel experimental parameters for investigation, proving valuable for both theoretical inquiry and practical application in domain of nanotechnology.

Polyols refer to polyhydric alcohols, specifically  $\alpha$ -diols like 1,2-ethanediol (ethylene glycol), 1,2-propanediol (propylene glycol), or ether glycols such as di(ethylene) or tri(ethylene glycol). Fievet, Lagier and Figlarz were the first to introduce the use of polyols for the synthesis of tiny particles in 1989, they also coined the term or phrase 'polyol process' or 'polyol

synthesis'. The polyol process represents an innovative method for synthesizing fine metal nanoparticles using metals that are easily reducible, such as copper, noble metals like Au, Pd and Ag, as well as their alloys. Additionally, it is applicable to less reducible metals like cobalt, nickel, iron and their alloys [7,8].

ASIAN JOURNA

The polyol procedure is favoured over the others because it does not produce hazardous gases such as carbon monoxide like thermal decomposition or contamination of the product [9]. It is a simple and eco-friendly liquid phase chemical process that generally does not require inert gas atmosphere [10]. Controlled form of metal nanoparticles can also be performed using long-chain capping agents or polymers such as PVP (polyvinylpyrrolidone). In modified polyol synthesis of nanoparticles, polyvinyl pyrrolidone (PVP) is commonly employed as a capping agent. Capping agents, also referred to as surface stabilizers and shape regulators, not only provide stability to the nanoparticles surfaces but also serve as ligands for the solvated species. It has been reported earlier that particle size can be changed by varying the PVP concentration [11].

**Synthesis of nanoparticles in polyol:** Polyol synthesis is a widely used technique for synthesizing metal nanoparticles, particularly noble metals like gold, silver and platinum, as well

This is an open access journal, and articles are distributed under the terms of the Attribution 4.0 International (CC BY 4.0) License. This license lets others distribute, remix, tweak, and build upon your work, even commercially, as long as they credit the author for the original creation. You must give appropriate credit, provide a link to the license, and indicate if changes were made.

as metal oxides and nanocomposites. It is known for its bottomup assembly technique with atomic precision and minimal defects, proves to be effective in creating nanostructures [12]. The type of nanoparticle formed depends on the choice of metal precursors, reaction conditions and stabilizing agents used during the synthesis process. The reaction of polyol in nanoparticle formation is a crucial step in the synthesis. In general, procedure for polyol synthesis of nanoparticles involves the following steps:

**Preparation of precursors:** In the first step, the metal precursors or metal salts are dissolved in a polyol to make a homogeneous solution. For the synthesis of nanoparticles, solid precursors can be taken in the form of nitrate, chloride, acetate (quite soluble in polyols) or oxide, hydroxide (slightly soluble). It is important to mention here that metallic salts with a hydrated form serve as good precursors for the synthesis of nanoparticles. In almost all the studies, variations in pH can influence the formation of nanostructures, which can be altered by the addition of acids or alkalis during the reaction process. The nature of nanoparticles also depends on the acidity or basicity of medium. For instance, in acidic media, the formation of CuO takes place [13].

**Heating and reduction:** The precursor solution is then heated to a specific temperature with or without inert atmosphere (usually in the presence of a protective gas like nitrogen or argon). Typically, the heating temperature varies reaction to reaction. In recent years, in polyol synthesis, variations in solvent media have been employed, in which a smaller amount of polyol has been used in aqueous media [14].

**Nucleation:** As the solution reaches the reaction temperature, the metal precursors undergo chemical reduction by polyol. Polyols act as both a solvent and a reducing agent. It can donate hydrogen atoms to reduce metal ions to their zerovalent states.

**Growth and stabilization:** The reduced metal atoms then begin to aggregate and form nanoclusters. These nanoclusters continue to grow by further reduction and coalescence. The size and shape of the nanoparticles are influenced by several factors, including reaction temperature, precursor concentration and the presence of stabilizing agents (capping agents) that control the growth and prevent uncontrolled agglomeration. Anions such as acetate, hydroxide, nitrate and chlorides of the metallic cations have the optimum capacity to form micronanostates with dimensions ranging from 1 to 100 nm. In some reactions, additional compounds containing sulfur, nitrogen and oxygen have been utilized as stabilizers or capping agents, such as PVP (polyvinylpyrrolidone), CTAB (cetyltrimethylammonium bromide), *etc.* [15,16].

**Quenching:** To control the size of the nanoparticles, the reaction can be quenched by rapidly cooling the reaction mixture after the desired nanoparticle size is achieved.

**Purification:** After the synthesis, the nanoparticles are typically separated from the reaction mixture, often by centrifugation or other separation techniques and then washed to remove excess reagents and byproducts.

Characterization: Finally, the nanoparticles are characterized using various techniques like transmission electron microscopy (TEM), scanning electron microscopy (SEM), Xray diffraction (XRD) and UV-Vis spectroscopy to determine their size, shape, crystal structure and optical properties.

The choice of metal precursor, reaction conditions and stabilizing agents can significantly influence the size, shape and properties of the nanoparticles obtained. Polyol synthesis is a versatile and widely used method for producing nanoparticles with controlled sizes and shapes, making it valuable for various applications across different industries.

Mechanism of nanoparticle formation in polyols: Polyol method of synthesis is a versatile method for the synthesis of metal nanoparticles of various sizes, shapes, compositions and crystallinity. Polyol acts as both the solvent and the reducing agent in the synthesis process, enabling the fabrication of finely shaped granular and nanostructured materials, including metals, oxides, chalcogenides, halides, alkoxides and hydroxides. These synthesized materials find diverse applications in renewable energy, human health, environmental solutions and microelectronics [17]. The main distinctive characteristic of the polyol mechanism is the reduction process occurs in a solution instead of the solid state. This is facilitated by the high dielectric constant of polyol, which makes it an adaptable solvent for inorganic metal precursors [18]. Additionally, the high boiling point of polyols allows the reaction to be carried out at elevated temperatures, which promotes the reduction and nucleation processes, ultimately leading to the formation of nanoparticles. Polyols oxidized to different aldehyde and ketone species at high temperatures, allowing metal precursors to be reduced [19]. The formation of nanoparticles using ethylene glycol as a polyol reducing agent involves several chemical reactions. The exact reactions can vary depending on the metal precursors used and the reaction conditions. The key steps involved in the polyol synthesis of nanoparticles are as follows:

(i) **Reduction:** The polyol solvent, typically heated to a high temperature, acts as a reducing agent. Polyols with hydroxyl groups are capable of forming hydrogen bonds with water molecules, making them soluble and often exhibiting a slightly basic character or acts as weak bases. The presence of hydroxyl groups increases the electron density around the oxygen atom, making it easier for the polyol to donate electrons and act as a base. The hydroxyl groups in the polyol molecules donate electrons to the metal ions, leading to their reduction to zero-valent metal atoms. This reduction, in turn, facilitates the reduction of metal precursors. Additionally, the hydroxyl groups of polyols can shield the metal cations by forming a stable coordination complex, preventing their re-oxidation back to higher oxidation states. The extent of reduction is influenced by the redox potential of the metal precursor and the reaction conditions. However, it's important to note that the basicity of most polyols is relatively weak compared to strong bases like hydroxide ions. For example, reduction of metal hydroxide to metal using ethylene glycol [20] is shown in Scheme-I.

During the synthesis of platinum nanoparticles *via* the polyol method, the presence of CO adsorbed on platinum species, which is confirmed by FTIR, represents that the reduction of the metal in ethylene glycol occurs through aldehyde formation [21].



The use of basic polyols as reducing agents often allows for milder reaction conditions compared to harsher reducing agents. This can be advantageous for reducing metal ions that are sensitive to high temperatures or extreme pH conditions. However, majority of reactions were carried out in ethylene glycol due to its non-toxic behaviour and high boiling point. In some reactions co-solvent of same class such as butanol, glycerol *etc.* were also used [22].

(ii) Nucleation: The reduced metal atoms agglomerate and form nuclei in the polyol solution. The size and number of nuclei depend on factors such as the concentration of metal precursors and the reaction temperature.

(iii) Growth: Once the nuclei formed, further reduction and subsequent nucleation continue, resulting in the growth of nanoparticles. The dimensions and structure of the nanoparticles can be controlled by modifying reaction conditions, including variables like reaction duration, temperature and the utilization of stabilizing agents.

(iv) Surface capping and stabilization: During the reduction process, the reactive species formed from ethylene glycol degradation also act as capping agents. They adsorb onto the nanoparticle surfaces, stabilizing the particles and preventing them from excessive growth or aggregation. The capping agents help maintain the size and shape of the nanoparticles. However, in some cases, additional capping agents can be employed to achieve improved results.

The driving force behind the polyol synthesis lies in the reduction potential of the polyol, which makes it an effective reducing agent for a wide range of metal precursors. The chelating capability of polyol is advantageous for regulating essential aspects, including particle nucleation, growth and agglomeration [23]. The above points illustrate the basic steps involved in the polyol synthesis of metal nanoparticles. It is important to observe that the specific mechanisms and reactions can vary depending on the metal precursor and conditions used during the synthesis. Additionally, for different types of nanoparticles (*e.g.*, metal oxide nanoparticles), the synthesis process may involve additional reactions specific to the material being synthesized.

#### Synthesis of nanoparticles and nanocomposites in polyol

Synthesis of metal nanoparticles in polyol: Several common types of metal nanoparticles that have been synthesized using polyol synthesis over the past decade are discussed below:

#### A. Pure metal nanoparticles

(i) Gold nanoparticles (AuNPs): Gold possesses remarkable qualities that stem from its noble nature. It earns this distinction due to its impressive resistance to both corrosion and oxidation. The unique character of gold extends to its nanoparticle form, where its chemistry takes on a distinct demeanor due to the high surface area-to-volume ratio inherent to nanoparticles. Even at this scale, a gold nanoparticle retains a core similar in arrangement to bulk gold, maintaining a face-centered cubic (FCC) structure [24]. One of the captivating phenomena exhibited by gold nanoparticles is surface plasmon resonance (SPR). This captivating occurrence arises from the synchronized movement of their conduction electrons in reaction to incoming light. As a result of SPR, these nanoparticles become adept at both absorbing and scattering light, which has profound implications for applications such as sensing, imaging and even photothermal therapy [25].

In addition to these fascinating properties, various studies have reported the synthesis of monodisperse gold nanoparticles with tunable sizes and shapes, encompassing spheres, rods, triangles and more. This achievement is often accomplished through methods like the polyol method. In 2015, Yu et al. [26] employed a rapid and environmental friendly microwaveassisted polyol method using PVP as capping agent and HAuCl<sub>4</sub> as salt precursor to synthesize a complex multi-material consists of monodisperse gold nanotriangles with a size of approximately 280 nm, which are uniformly decorated by superparamagnetic iron oxide nanoparticles of 5 nm, whereas earlier used methods for gold nanotudes are based on a multi-step seed-mediated approach involves the highly toxic cationic surfactant cetyltrimethylammonium bromide (CTAB) resulting in a poorly biocompatible or time-consuming process due to an inevitable ligand exchange step. Previous studies [27] highlighted that the initial nucleation step appears to have an impact on the shape of gold nanoparticles using hydrogen tetrachloroaurate(III) trihydrate as salt precursor. When triethylene glycol (TrEG) is present, the resulting nanoparticle shapes tend to be triangular or hexagonal plates. Conversely, when utilizing 1,3propanediol (1,3-POD), cubic shapes tend to be favoured. Further investigations have revealed that the influence of polyvinylpyrrolidone (PVP) in determining the ultimate shape of the nanoparticles is not significant. However, it does serve a role in both stabilizing the nanoparticles and directing the growth of the initial seeds towards achieving the desired final shape [27].

Recently, Long [28] achieved the synthesis of gold nanoparticles with a varied assortment of spherical and polyhedral shapes, all falling within the 50 nm range through a modified polyol method utilizing gold(III) chloride trihydrate (HAuCl<sub>4</sub>·  $3H_2O$ ), ethylene glycol, polyvinylpyrrolidone (PVP), sodium borohydride. In recent endeavor, Ditte *et al.* [29] employed a polyol-based approach under alkaline conditions using same salt precursor, remarkably eliminating the necessity for surfactants. This led to the formation of small-sized nanoparticles with diameters of approximately 10 nm. Subsequently, Quinson *et al.* [30] synthesized colloidal gold nanoparticles at room temperature using an elegantly simple technique. This method called for just an alcohol as a reducing agent, water, a base and a gold precursor, rendering additional reducing agents or stabilizers unnecessary. It is important to mention that nanoparticles synthesized in alkaline mono-alcohols demonstrate nanocatalysts with significantly higher activity, up to three times, in the electrooxidation of ethanol and ethylene glycol, compared to nanoparticles synthesized using polyols.

(ii) Silver nanoparticles (AgNPs): Similar to gold, silver is categorized as a noble metal due to its remarkable resistance against oxidation and corrosion. It predominantly exhibits two common oxidation states *viz.* +1 and +2. The +1 state is more stable and prevalent in its compounds, while the +2 state is rarer and less stable. Notably, silver nanoparticles hold particular significance owing to their distinctive antibacterial properties and their roles in sensing and catalysis.

The polyol synthesis technique enables the synthesis of silver nanoparticles with diverse shapes, including spheres, wires and plates. In 2013, Chiang et al. [31] embarked on the synthesis of silver nanoparticles using silver nitrate salt precursor in polyol at 120 °C. This approach involved the utilization of tripropylene glycol, 3-ethyl-3-oxetanemethanol and polyvinylpyrrolidone (PVP) as protective agents, along with polycaprolactone triol as the reducing agent. Their study illuminated that several factors, such as the type of reducing agent, the molecular weight of PVP, the concentration of PVP, reaction temperature and time, collectively influenced the sizes of the silver particles. In subsequent year, as the continuous-flow microwave assisted polyol method was utilized, small and uniformly spherical silver nanoparticles were rapidly synthesized. This synthesis employed silver acetate and silver nitrate as precursors and took only seconds, resulting in remarkable productivity and high yields. The notably heightened reactivity of silver acetate, as a correlative factor, established it as a vastly superior substrate for generating small (10-20 nm) spherical silver nanoparticles within few seconds [32].

In earlier studies, Fereshteh et al. [33] accomplished the synthesis of silver nanoparticles using silver nitrate as salt precursor and polyethylene glycol (PEG) as a multifunctional agent at 160 °C. The achievement of optimal conditions hinged upon achieving specific concentration ratios of AgNO<sub>3</sub>, PEG and NaBH<sub>4</sub>, leading to impressive levels of efficiency and purity in the synthesis process. When NaBH<sub>4</sub> was present in lower concentrations, the replacement of PEG molecules by ions and/ or free electrons led to the synthesis of the tiniest nanoparticles. Ultimately, the most efficient and pure synthesis was attained when employing the optimal condition with an AgNO<sub>3</sub>:PEG: NaBH<sub>4</sub> concentration ratio of 1:10:0.01, yielding the smallest Ag nanoparticles with an efficiency of 82% and purity reaching to 98.95%. The polymeric coating on these silver nanoparticles marked them as prospective candidates for drug delivery systems. More recently, silver nanoparticles were rapidly synthesized within a mere 2 min duration through the microwaveassisted polyol method [34]. Silver nanoparticles, polyvinyl pyrrolidone (PVP) and ethylene glycol (EG) were employed as precursors in this endeavor, with the process operating at 145 °C.

Silver nanoparticles were synthesized by Zeroual *et al.* [35] through the reduction of silver nitrate using an aqueous emulsion of latex copolymer produced by the polyol process.

These nanofluids exhibited an impressive 3% enhancement in thermal conductivity, particularly when copolymer latex contents were higher. The thermal behaviour of ethylene glycol silver-based nanofluids carries potential for applications as efficient circulating heat transfer fluids. In recent advancement, Pernot *et al.* [36] introduced an automated synthesis method for producing adjustable and uniformly dispersed silver nanoparticles of mean radii 3-5 nm through the polyol method using silver nitrate as salt precursor. This milestone was reached using a versatile and modular automation platform named the Chemputer. This modular system paves the way for synthesizing diverse nanoparticle morphologies, sizes and potentially even more intricate structures.

(iii) Copper nanoparticles (CuNPs): This element is indispensable to many organisms, playing a vital role in enzymes and processes like electron transport during respiration. In line with other metals, copper nanoparticles exhibit distinct attributes due to their minute size, offering applications in catalysis, electronics and antimicrobial functions. Integrating copper nanoparticles into materials such as polymers and ceramics generates nanocomposites with enhanced attributes, finding utility in coatings, packaging and electronics. However, the synthesis of copper nanoparticles encounters challenges due to their high surface-to-volume ratio, making them susceptible to oxidation.

Polyol synthesis emerges as a prominent method to fabricate copper nanoparticles while controlling their dimensions, morphology and characteristics. This technique involves blending a copper precursor like copper chloride or copper acetate with a polyol solvent such as ethylene glycol (EG), diethylene glycol (DEG) or glycerol. The polyol solvent assumes dual roles as a reducing and capping agent. Within its hydroxyl groups, the polyol molecules contribute electrons to copper ions, enabling their reduction to copper atoms. This reduction process's nuances are influenced by variables including reaction temperature, precursor concentration and the type of polyol employed.

Earlier, Morais et al. [37] synthesized copper nanoparticles utilizing copper chloride, polyvinylpyrrolidone (PVP), ethylene glycol, sodium citrate and ascorbic acid. Their findings highlighted that a relatively high concentration of PVP played a pivotal role in stabilizing the nanoparticles' surface chemically and controlling their gradual growth rate. They determined that optimal size and distribution of Cu nanoparticles (CuNPs) were achieved using solvents like glycerol and glycerin, yielding small, spherical copper nanoparticles with exceptional dispersion. In 2020, Lee et al. [38] employed the modified polyol method to synthesize antioxidant copper nanoparticles (CuNPs). They utilized copper sulfate pentahydrate, polyvinylpyrrolidone (PVP) and L-ascorbic acid at 150 °C. These CuNPs exhibited a diameter of approximately  $61 \pm 12$  nm, coated with an 8-nm thick layer of PVP. This imparted them with notable resistance to chemical oxidation and coagulation, even during prolonged exposure to ethylene glycol solution. Notably, these CuNPs demonstrated enduring catalytic activity in reducing 4-nitrophenol and nitrite ions, suggesting potential as efficient electrochemical sensors.

Efforts to synthesize stable zero-valent copper nanoparticles face oxidation susceptibility. However, this challenge can be addressed by employing a copper amine complex as a precursor, ascorbic acid as a reducing agent and polyvinyl pyrrolidone as a capping agent [39]. Moreover, studies indicated that using glycerol in CuNPs synthesis offers advantages such as cost-effectiveness, non-toxic nature, mild reducing properties, efficient supramolecular network establishment that immobilizes metal nanoparticles and prevention of re-oxidation [40]. The experimental evidence demonstrated that utilizing alkylamine (1-hexadecylamine) as a protectant significantly reduced copper oxide nanoparticle size (upto 8 nm) compared to using PVP (300 nm). A higher alkylamine/copper molar ratio contributed to forming small, spherical copper nanoparticles [41]. Recently, a novel approach have been used to control the maize pathogens both in vitro and in vivo, utilizing newly synthesized copper nanoparticles using CuSO4 as a precursor, NaBH<sub>4</sub> and ascorbic acid as reducing agents and polyethylene glycol 8000 (PEG-8000) as a stabilizing agent [42].

(iv) Nickel nanoparticles (NiNPs): The chemistry of nickel is characterized by its diverse oxidation states, reactivity and various applications. Nickel exhibits a range of oxidation states, with the most common being +2 and +3. The +2 oxidation state is more stable and prevalent in its compounds. As a transition metal, nickel has the ability to form complex ions and compounds due to its partially filled *d*-orbitals. Similar to other metals, nickel nanoparticles exhibit unique properties at the nanoscale, making them useful in catalysis, electronics and other applications.

Polyol synthesis stands as a prevalent method for crafting nickel nanoparticles while precisely tailoring their attributes like size, shape and properties. This process entails mixing a nickel precursor, such as nickel chloride or nickel acetate with a polyol solvents like ethylene glycol, diethylene glycol or glycerol. Under controlled conditions, typically at elevated temperatures (around 150 to 200 °C), the mixture undergoes reduction, instigated by the heated polyol solvent. The polyol solvent serves a dual role, functioning both as a reducing agent and a capping agent. This is achieved through the donation of electrons from the hydroxyl groups in the polyol molecules to the nickel ions, thus facilitating their transformation into nickel atoms. This reduction process leads to the formation of nickel nanoparticles.

In 2013, Rajakumar *et al.* [43] made significant strides by investigating the larvicidal potential of synthesized nickel nanoparticles, achieved through polyol synthesis. Utilizing Nihydrazine as a precursor and adopting Tween-80 for medium and stabilization, these nickel nanoparticles exhibited substantial anti-parasitic effects against larvae of cattle ticks and mosquitoes.

The earlier work by Zhang *et al.* [44] highlighted the dispersion of nickel nanoparticles within mesoporous silica through an innovative polyol-based delivery mechanism. This yielded catalysts with remarkable resistance to coking (metal surface is obstructed due to the buildup of carbon accumulation and sintering (creation of larger metal particles that reduce both the overall surface area and activity) during methane reforming.

In a rapid process assisted by microwave and polyol, nickel nanoparticles were synthesized and stabilized in an aqueous emulsion of copolymers. This approach involves reducing a metal salt (NiCl<sub>2</sub>· $6H_2O$ ) in the presence of a low concentration of hydrazine hydrate with the assistance of a commercial copolymer (latex) acting as both the reducing and stabilizing agents [45]. Synthesis of nickel nanoparticles using nickel chloride in ethylene glycol and hydrazine as reducing agent [46] can be represented as:



Hydrazine, which acts as a reducing agent, is added to the heated precursor solution. Hydrazine donates electrons to the nickel ions in the precursor, causing the reduction of the nickel ions to form nickel atoms. The ethylene glycol provides a controlled environment for the reduction and stabilization of the nanoparticles. The concentration of the precursor can be adjusted to control the size and concentration of the resulting nanoparticles. This reduction process leads to the nucleation and growth of nickel nanoparticles. Recently, Minh *et al.* [47] employed a combination of the polyol process and hydrothermal treatment to synthesize spherical nickel magnetic nanoparticles. These nanoparticles exhibited ferromagnetic interactions, verging on a superparamagnetic state, which diminished with decreasing nanoparticle size.

#### B. Bimetallic and alloy nanoparticles

(i) Palladium-cobalt (Pd-Co) nanoparticles: Amidst increasing global energy demands and the complexities they bring, there is a significant enthusiasm for discovering novel approaches to energy production. One promising avenue involves fuel cells that use hydrogen gas generated by splitting water through an electrochemical process. Recently, Kaya et al. [48] embarked on the formation of metallic and bimetallic nanoparticles in ethylene gycol using palladium(II) acetylacetonate and cobalt(II) acetylacetonate as salt precursors with a protective coating of polyvinylpyrrolidone. These nanoparticles encompassed Pd, Co and a range of  $Pd_xCo_{1-x}$  compositions (x = 0.5, 0.12, 0.23, 0.49, 0.55, 0.62). Their purpose was to develop catalysts as alternatives to traditional platinum-based ones for driving the hydrogen evolution reaction (HER). A remarkable achievement emerged in the form of Pd<sub>62</sub>Co<sub>38</sub> catalyst, boasting an HER onset potential 16 mV higher than the commercially available Pt/C catalyst.

(ii) Palladium-platinum (Pd-Pt) nanoparticles: Palladiumplatinum bimetallic nanoparticles are hybrid nanomaterials composed of both palladium and platinum metals. These nanoparticles combine the unique properties of both metals, offering enhanced catalytic and functional characteristics compared to individual metal nanoparticles. In 2018, Proenza *et al.* [49] synthesized Pd-Pt bimetallic nanoparticles using chloroplatinic acid hexahydrate (H<sub>2</sub>PtCl<sub>6</sub>·6H<sub>2</sub>O), potassium tetrachloropalladate (K<sub>2</sub>PdCl<sub>4</sub>), ethylene glycol and PVP, displayed an alloy structure and thermodynamic analysis indicated that the Pd-Pt nanoalloys (with compositions of 2:1, 1:1 and 1:2) exhibited higher thermal stabilities compared to palladium monometallic nanoparticles but lower thermal stabilities than platinum monometallic nanoparticles. Findings also indicated that the melting points of the nanoalloys shifted towards higher temperature values with an increase in platinum content within the particles or platinum content led to an enhancement in the thermal stability of the Pd-Pt nanoalloys. Higareda *et al.* [50] also employed the polyol method to synthesize Pt-Pd core-shell bimetallic nanoparticles (CSNPs). The outcomes validated that the catalytic efficacy is contingent upon the elemental composition of PtPd CSNPs. Among these, Pt-Pd (1:0.5)/C showed superior catalytic attributes in comparison to commercial Pt/C.

(iii) Platinum copper (Pt-Cu) nanoparticles: Enhancing the efficiency and endurance of electrocatalysts based on platinum remains a critical endeavor for applications in hydrogen production. Recently, the effectiveness of Cu-doped platinum alloy nanostructures, synthesized through a modified polyol method [51], has come under scrutiny. This synthesis method involves platinum(II) acetylacetonate and copper(II) acetate, along with essential components such as ethylene glycol, NaOH, polyvinyl pyrrolidone (PVP, m.w. 40,000), sodium borohydride (NaBH<sub>4</sub>), DMF, ethanol and dichloromethane. Through systematic investigation, the catalytic attributes of these Cu-doped Pt alloy nanostructures were explored, with a specific focus on their performance in the hydrogen evolution reaction (HER) at ambient conditions. The results were compared with the performance of Pt/C catalysts commercially available on the market. The HER performance of the catalysts revealed an overpotential of approximately -1.0 V (vs. Ag/AgCl), with Pt<sub>0.25</sub>Cu<sub>0.75</sub> and Pt<sub>0.75</sub>Cu<sub>0.25</sub> catalysts emerging as standout performers in 1 M KOH solution. Particularly significant was Pt0.75Cu0.25 catalyst, which exhibited exceptional performance, displaying the highest mass activity at 62.80 mA mg<sup>-1</sup> Pt. Moreover, this catalytic composition demonstrated the most favourable HER attributes, marked by a remarkably low onset potential of 0.989 V and a Tafel slope of 35.5 mV dec<sup>-1</sup>, representing a substantial advancement over commercially available Pt/C catalysts.

(iv) Silver-platinum (Ag-Pt) nanoparticles: Briones *et al.* [52] recently utilized the polyol method to form Ag-Pt nanoparticles, employing a heterogeneous nucleation approach. The process involved incorporating polyvinylpyrrolidone (PVP) as surfactant with AgNO<sub>3</sub> and potassium tetrachloroplatinate(II) (K<sub>2</sub>PtCl<sub>4</sub>) as metal precursors within a temperature range of 160 to 190 °C. Ethylene glycol was used as a reducing agent. The catalytic activity and durability of the Ag-Pt (1:3)/C electrocatalyst were evaluated through chronoamperometry and accelerated electrochemical degradation tests. The introduced silver weakened the chemisorption of carbonaceous species, resulting in enhanced performance. Consequently, this cost-effective AgPt (1:3)/C electrocatalyst exhibits significant potential for ethanol oxidation, outperforming commercial Pt/C counterparts [52].

(v) Platinum-tin (Pt-Sn) nanoparticles: Zhou *et al.* [53] synthesized PtSn alloy nanoparticles through the co-reduction of Pt and Sn precursors along with stabilizing agents. Subsequently, these nanoparticles underwent an *in situ* trans-formation into Pt-SnO<sub>2</sub> hybrid nanostructures on alumina supports through

calcination. Comparing it to  $Pt/Al_2O_3$ , the  $Pt1-(SnO_2)_{0.3}/Al_2O_3$  catalyst displayed improved catalytic efficiency in the hydrogenation of furfural to furfuryl alcohol. This enhanced performance of  $Pt-SnO_2/Al_2O_3$  can be attributed to the formation of closely interacting  $Pt-SnO_2$  interfaces, which exhibit high catalytic activity due to the synergistic effect between Pt and  $SnO_2$  in the furfural hydrogenation.

(vi) Cobalt nickel (Co-Ni) nanoparticles: Nanomaterials have gained significant attention in various biomedical areas, particularly in the battle against cancer. In recent years, CoNi alloy nanoparticles synthesized by Sargazi *et al.* [54] using a simple polyol process and their potential in cancer treatment, *in vitro* cytotoxicity assessments were conducted. Remarkably, the Co<sub>0.5</sub>Ni<sub>0.5</sub>, Co<sub>0.6</sub>Ni<sub>0.4</sub> and Co<sub>0.4</sub>Ni<sub>0.6</sub> NPs at a dosage of 6.25  $\mu$ g/mL showed no adverse effects on normal human cells. However, they exhibited significant cell death in cancerous cells, indicating their potential as promising candidates for cancer therapy.

(vii) Silver-palladium (Ag-Pd) nanoparticles: Silverpalladium nanoparticles have special importance particularly in catalysis. In recent years, a simple polyol method reported by Uribe *et al.* [55] synthesized Ag-Pd bimetallic nanoparticles, enabling better control over their morphology and particle size. The catalytic behaviour of these bimetallic nanoparticles was anal-yzed through electrochemical characterization and the results revealed that  $Ag_2Pd_1/C$  bimetallic nanoparticles exhibited the superior catalytic activity for the oxygen reduction reaction at room temperature compared to Ag/C.

(viii) Platium-rohodium (Pt-Rh) nanoparticles: In 2019, Bundli *et al.* [56] exhibited the effective achievement of bimetallic Pt-Rh nanoparticles in a single phase, employing precise control over composition by simultaneous decomposition precursor metals Pt and Rh in a polyol environment. The comprehensive approach used is not only applicable to the Pt-Rh system but also demonstrates the significance of controlling precursor reactivity to achieve precise metal compositions in bimetallic nanoparticles, which can be valuable for fundamental studies and practical applications.

(ix) Platinum-gold (Pt-Au) nanoparticles: In 2013, the  $Pt_xAu_y$ -starch/HTs catalysts synthesized in polyol media by Ebitani *et al.* [57] exhibited superior selectivity in the oxidation of the primary hydroxyl group in GLY and PG to yield glyceric acid (GA) and lactic acid (LA), respectively, using molecular oxygen in an aqueous solution [57]. Among the synthesized bimetallic catalysts,  $Pt_{60}Au_{40}$ -starch/HT exhibited the highest activity, making it the most effective catalyst for the selective aerobic oxidation of polyols.

### C. Semiconductor nanoparticles

(i) Cadmium selenide nanoparticles (CdSe NPs): Polyol synthesis can be used to obtain cadmium selenide nanoparticles with tunable bandgaps for optoelectronics and photovoltaic applications. In 2013, Karthika *et al.* [58] used magnetic stirrer-assisted polyol method to synthesize lead-cadmium selenide nanoparticles, using cadmium acetate, lead acetate and selenium powder as precursor. Synthesized nanoparticles of size smaller than 60 and 150 nm exhibited a favourable band gap of 3.1

eV, signifying its excellent semiconducting characteristics. Comparing these outcomes with those of cadmium selenide nanoparticles, it was observed that lead-cadmium selenide exhibited a slightly enhanced semiconducting property, rendering it more suitable for semiconductor-based electronic applications.

(ii) Hybrid/core-shell nanoparticles: Polyol synthesis can be used to synthesize core-shell nanoparticles, where a core of one material is encapsulated by a shell of another material. The concept of core-shell nanostructures offers a promising route for developing advanced dielectric nanocomposites. Recently, core-shell nanoparticles of FeCo-MnO2 with varying MnO<sub>2</sub> shell thicknesses were successfully synthesized by Park et al. [59] using polyol-coating technique. Interestingly, the imaginary permeability remained nearly constant across different MnO<sub>2</sub> shell thicknesses, while the complex permittivity showed an increase with thicker MnO<sub>2</sub> shells. Specifically, the FeCo-MnO<sub>2</sub> nanoparticles with a shell thickness of 71 nm exhibited a distinctive Lorentz-type dielectric resonance attributed to a displacement current lag at the core-shell interface. This approach provides a convenient and feasible method for designing magnetic core-dielectric shell-based microwave absorbers.

The polyol route synthesized core-shell nanoparticles  $(Fe_2O_3/Gd_2O_3 CSNPs)$  with gadolinium oxide  $(Gd_2O_3)$  encapsulating iron oxide  $(Fe_2O_3)$  were synthesized by Rajesh *et al.* [60] examined their frequency-dependent dielectric dispersion and cobalt ferrite-cobalt oxide core-shell nano-particles were successfully synthesized by Martinez *et al.* [61] through seed-mediated growth in a polyol medium.

Platinum displays an outstanding electrocatalytic performance for methanol oxidation, however, its drawback lies in its high cost and susceptibility to carbon monooxide (CO) poisoning. Carbon monooxide (CO) is generated as a reaction intermediate during methanol oxidation. This issue arises from CO getting adsorbed on the active sites of platinum, leading to potential deactivation. However, CO adsorbed on these sites can be eliminated through oxidation by nearby  $OH_{ads}$  as described by the subsequent equation:

$$CO_{ads} + OH_{ads} \longrightarrow CO_2 + H^+ + e^-$$

Above reaction proceeds through bifunctional mechanism. Among the range of bimetallic catalysts, the Pt-Ru system is often highlighted as exceptionally favourable due to the heightened attraction of Ru towards –OH groups. Above equation of the Ru-Pt system becomes relevant at lower potentials and can be formulated as:

 $Pt - CO + Ru - OH \longrightarrow Pt + Ru + CO_2 + H^+ + e^-$ 

Muthuswamy *et al.* [62] in 2013 used polyol method to encapsulate pre-synthesized Ru nanoparticles with platinum at different pH values (6, 7, 8 and 10). The results suggested that the core-shell catalysts achieve their highest catalytic enhancement through the dominant bifunctional character of the alloy shells, rather than ligand-effect-promoted platinum enriched shells.

In recent years, silver copper core shell nanoparticles [63] and nickel-silver core-shell nanoparticles [46] synthesized by

polyol method in silver copper CSNPs polyoxyethylene-(80)sorbitan monooleate (Tween-80) as non-toxic stabilizer. Spherical copper (core)–silver (shell) nanoparticles with a diameter of 40-50 nm synthesized by Kheawhom *et al.* [64] using polyol successive reduction method in glycerol, with the addition of NaOH. The impact of various synthesis parameters was investigated, including the molar ratio of NaOH: Cu (0:1, 1:1, 3:1 and 5:1) as well as the molar ratio of Ag:Cu (0.01:1, 0.05:1, 0.10:1, 0.15:1 and 0.20:1) on the size, structure and composition of the resulting nanoparticles and it was observed that the average size of the nanoparticles decreased as the ratio of sodium hydroxide increased.

#### D. Magnetic nanoparticles

(i) Iron-platinum nanoparticles (Fe-Pt NPs): Recently, Fe-Pt/VC catalyst with an average size of  $2 \pm 0.3$  nm and FCT phase was successfully synthesized by Kondo-Francois Aguey-Zinsou *et al.* [65] making it suitable for both the electroreduction of molecular oxygen and the electro-oxidation of molecular hydrogen. Remarkably, this Fe-Pt/VC catalyst exhibits a mass activity 2.8 times higher than that of Pt, surpassing the performance of traditional platinum catalysts.

**E.** Synthesis of metal oxide nanoparticles: Below are some metal oxide nanoparticles that have emerged through the application of polyol synthesis.

(i) Iron oxide nanoparticles: Polyol synthesis can produce iron oxide nanoparticles, which are used in magnetic resonance imaging (MRI) and drug delivery applications. Recent research suggests that the introduction of 70-80 ppm of iron oxide nanoparticles, synthesized by polyol method, resulted in enhanced biogas production in both quantitatively and qualitatively [66].

Water-dispersible iron oxide nanoparticles (IONPs) of superior relaxivity values when compared to commercially available contrast agent synthesized by polyol method, modified by incorporating high pressure and high temperature conditions shows promising potential of these novel particles as MRI contrast agents [67].

Later in 2021, the microwave-assisted polyol method used for the synthesis of both single-core and multicore magnetic iron oxide nanoparticles, which can be utilized as self-heating catalysts for the degradation of anionic dye (acid orange 8) and cationic dye (methylene blue) [68]. Recently, Kim *et al.* [69] demonstrated that angle-shaped superparamagnetic iron oxide nanoparticles (SPIONs), synthesized through the bromideassisted polyol method, have the potential to deliver highly effective magnetic hyperthermia therapy for cancer treatment when exposed to biologically safe alternating magnetic fields with 140 Oe and 100 kHz frequency.

(ii) Zinc oxide nanoparticles (ZnO NPs): Polyol synthesis can yield zinc oxide nanoparticles that exhibit unique optical and photocatalytic properties. Zinc oxide has garnered significant attention in research owing to its exceptional properties. With features like a wide band gap, high refractive index, excellent electrical conductivity and high optical transmission in the visible spectrum, zinc oxide finds applications in various fields, including gas sensors, varistors, optoelectronic devices and more [70]. Previous studies have confirmed a significant relationship between nanoparticle morphology and size with the structural isomerism of the surfactant in terms of polyol synthesis. For instance, the use of *ortho*-aminobenzoic acid resulted in spherical nanoparticles, while *meta*-aminobenzoic acid led to the formation of twin rods and triangular nanoparticles formed an oriented mesosphere. In the case of *para*aminobenzoic acid, the mutual alignment of crystal faces induced by interparticular forces led to the agglomeration of triangular nanoparticles, ultimately forming oriented mesospheres [71].

In 2018, Alves *et al.* [72] reported the effect of particle shape and size on the morphology and optical properties of zinc oxide nanoparticles synthesized *via* polyol method utilizing zinc acetate and/or sodium acetate in propylene glycol medium with different hydrolysis reaction times (10, 60 and 300 min). The investigation revealed a significant impact of hydrolysis reaction time and acetate ion concentration on the morphology and size of the ZnO nanoparticles. Later, ZnO nanoparticles synthesized using the polyol method exhibited superior minimum inhibitory concentration (MIC) values against both Grampositive and Gram-negative bacteria when compared to particles synthesized through the aqueous precipitation method [73].

In 2019, Mahamuni *et al.* [74] synthesized zinc oxide nanoparticles through refluxing zinc acetate dihydrate in diethylene glycol for 3 h to achieve a small size of approximately 15 nm, displayed significant antibacterial and antibiofilm properties. In 2022, uniform ZnO nanoparticles synthesized using a solvent-free solid-state approach, wherein zinc acetate dihydrate and ethylene glycol are thermally degraded at 500 °C for a duration of 3 h [75]. These findings indicate that the presence of ethylene glycol effectively prevents nanoparticle sintering, resulting in the formation of uniform particles and these particles possess an optical bandgap of 3.39 eV.

Hexagonal crystal structure ZnO nanoparticles synthesized by Zakiyah *et al.* [76] in 2022 *via* polyol and coprecipitation technique was subjected to photoesterification reaction of candlenut oil in the presence of UV-LED irradiation in order to investigate photocatalytic activity of nanoparticles. The results showed that both ZnO polyol and coprecipitation methods effectively converted free fatty acids (FFA) into fatty acid methyl esters (FAME), yields approximately 57% and 59% for ZnO coprecipitation and polyol methods, respectively.

ZnO nanoparticles, synthesized through the experimental polyol refluxing approach using ethylene glycol as a chemical reagent and TBAB as a capping agent, gives a viable alternative to the conventional chemical method [77]. These polyol mediated ZnO nanoparticles hold great potential for biomedical applications owing to their remarkable antibacterial and antifungal activities. The acid-catalyzed sol-gel process can also employed to synthesize ZnO nanoparticles using  $ZnNO_3 \cdot 6H_2O$  as precursor, aqueous isopropanol as solvent and glycerin to create the polyol system [78].

(iii) Copper oxide nanoparticles: The polyol method offers a single-step solution phase synthesis for cuprous oxide nanofluids. The process entailed the reduction of copper acetate using glucose and sodium lauryl sulfate (SLS) as surfactant within a mixture of water and ethylene glycol, which acted as base fluid. This technique has proven to be straightforward, dependable and swift for synthesizing Newtonian nanofluids containing cuprous oxide nanoparticles [79].

In earlier research, the antibacterial nanocellulose/Cu<sub>2</sub>O hybrid films were produced using glycerol as a green reducing agent through the microwave-assisted polyol synthesis [80]. Interestingly, the Cu<sub>2</sub>O nanoparticles did not exhibit toxicity towards mammalian cell cultures at the same concentrations but highly toxic to bacteria. These hybrid nanocellulose/Cu<sub>2</sub>O films, comprising non-toxic and biodegradable materials, hold promising potential for applications in the treatment of wounds and skin infections. The formation of Cu<sub>2</sub>O in glycerol is shown in **Scheme-III**.

In glycolic medium, Dahonog et al. [81] synthesized copper oxide nanostructures (Cu<sub>2</sub>O and CuO) by adjusting the pH. The XRD analysis validated the presence of cubic Cu<sub>2</sub>O under acidic conditions using HCl and monoclinic CuO under basic conditions using NaOH. Recently, Maturost et al. [82] synthesized catalysts Pd/xCuO-10CNT (where x = 1, 2, 3, 4) using an improved polyol method. These catalysts displayed remarkable stability and an outstanding ability to withstand carbon monoxide (CO) poisoning. This resilience and tolerance can be attributed to the modified Pd structures present on CuOsupported carbon nanotubes [82]. The introduction of CuO onto the surface of the CNT before loading Pd led to the creation of additional electrochemically active sites, resulting in an improved geometric and bifunctional system. CuO played a role in facilitating the adsorption of oxygen-containing species (OH<sub>ads</sub>) on the catalyst's surface, while the interaction between Pd and Cu metals facilitated favourable charge transfer effects.

Amid this narrative, the trend of doping-based creation weaves its spell. For instance, the recently employed polyolmediated refluxing method used by Baste *et al.* [83] synthesized nanomaterials of silver-incorporated copper oxide (Ag@CuO NMs) with utilization tetra butyl ammonium bromide (TBAB) as capping agent and ethylene glycol as a reducing agent for Ag<sup>+</sup> to Ag. The Ag@CuO nanomaterials that are obtained have promising possibilities in the field of photocatalytic applications, specifically in the purification of wastewater. Among the samples, Ag@CuO nanomaterial with a weight percentage



Scheme-III

of 5% exhibited the highest effectiveness in degrading methylene blue dye.

**F. Synthesis of nanocomposites:** A nanocomposite is a material that consists of a matrix or base material combined with nanoparticles. Nanoparticles with dimensions typically in the nanometer range (1 to 100 nm) and can be made from various materials, such as metals, metal oxides, polymers, carbon based materials and more. The incorporation of nanoparticles into a matrix material leads to the creation of a nanocomposite, which often exhibits unique and enhanced properties compared to the individual components. Nanocomposites can be created using various methods, including solution mixing, melt blending, *in situ* polymerization and more. The choice of method depends on factors such as the type of matrix material, nanoparticles and desired properties.

Nanocomposites formed using polyols typically involve the incorporation of nanoparticles into a polymer matrix where the polyol acts as a precursor or a component in the synthesis process. Polyols are typically polymeric compounds containing multiple hydroxyl groups (-OH) and are commonly used in the synthesis of polymers and polymer-related materials. When combined with nanoparticles, they can lead to the formation of nanocomposites with enhanced properties.

Adekoya *et al.* [84] achieved the successful synthesis of Ag/Pd bimetallic nanoparticles employing a seed-mediated approach mediated by polyvinylpyrrolidone (PVP). The process involved the use of PVP; PdCl<sub>2</sub> and AgNO<sub>3</sub> as sources of Pd and Ag, respectively. The reduction of these metal ions occurred simultaneously in solutions containing ethylene glycol, glycerol, diethylene glycol, pentaerythritol and sodium borohydride, under temperatures ranging from 160 to 200 °C. The nanostructured Ag/Pd bimetallic particles were produced through the seed growth or successive addition method within an aqueous medium, utilizing pentaerythritol (PET) as a capping agent.

Expanding on these findings, Shamsi et al. [85] extended their investigations into polyurethane (PU) synthesis to incorporate hydroxylated multi-walled carbon nanotubes (CNT), resulting in PU/CNT nanocomposites within a temperature range of 140-210 °C. The successful production of these nanocomposites further underscores the versatility of their synthetic approach. The study's findings revealed a distinct advantage in mechanical properties and thermal stability for PU samples primarily derived from 1,4-butane diol (BDO). More significantly, PU samples synthesized with 1,6-hexamethylene diisocyanate (HDI) displayed enhanced properties compared to those prepared using aromatic diisocyanates. This enhanced performance was attributed to the increased crystallinity and more densely packed microstructure achieved in the presence of HDI. These findings highlight the potential for tailoring the properties of PU nanocomposites through the selection of specific polyol and diisocyanate components, contributing to the advancement of materials design in the field of polymer nanocomposites.

Continuing on this trajectory of nanocomposite research, Okram *et al.* [86] contributed significantly to the field in recent years. Their use of a facile polyol method to synthesize nanocomposites of  $Cu_{1,x}Ag_xS$  with varying compositions (x = 0-0.75) has expanded the possibilities in materials engineering. The synthesis of CuS nanodisks via modified polyol approach involved the careful heating of a mixture containing Cu(NO<sub>3</sub>)<sub>2</sub>. 3H<sub>2</sub>O and thiourea diethylene glycol at temperatures between 175 and 180 °C, in the presence of a continuous nitrogen gas flow. Additionally, appropriate amounts of AgNO<sub>3</sub> were introduced to achieve nominal compositions of  $Cu_{1-x}Ag_xS: x = 0.05$ , 0.1, 0.2, 0.5, 0.75 and 1. The characterization of these nanocomposites revealed a distinct anomaly at 55 K in both the thermopower and Hall resistivity data, attributed to structural phase transitions. Notably, with an increase in the Ag content (x), a systematic reduction in thermal conductivity was observed around 300 K. As a result, a significant 50% enhancement in the figure of merit was achieved for Cu<sub>0.9</sub>Ag<sub>0.1</sub>S compared to pure CuS at 300 K. These findings underscore the potential for tailoring the properties of Cu<sub>1-x</sub>Ag<sub>x</sub>S nanocomposites through compositional adjustments to enhance their thermoelectric performance.

Building upon this innovative methodology, the introduction of hydrolysis forced by PVA via microwave-assisted sol-gel method by Souza et al. [87] in 2021 represents a notable stride in nanocomposite synthesis. By harnessing the potential of acetic acid and Zn(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O and coupling it with microwave assisted reflux at 100 °C, this approach exemplifies a controlled and efficient pathway for creating ZnO-PVA nanocomposites [87]. The synthesis of both ZnO-PVA nanocomposites and PVA films was accomplished through phase inversion. Authors believe that this strategy holds significant promise, as it can be extended to other polymers characterized by -OH groups in their side chains. This method not only demonstrates an inventive approach to synthesize ZnO-PVA nanocomposites but also opens avenues for broader applications by capitalizing on the presence of similar functional groups in diverse polymer systems.

Recently, Donadelli et al. [88] employed two distinct synthetic pathways to fabricate nanocomposites consisting of silver and iron oxides. These methodologies encompassed polyol synthesis and laser ablation techniques. The resulting nanocomposites exhibited a configuration where silver nanoparticles, sized between 10 and 20 nm, were embedded within iron oxide structures spanning 100 to 300 nm. It's worth noting that the Ag-to-Fe ratio was significantly higher in the nanomaterial produced through the chemical route. Both variants of nanomaterials demonstrated proficiency in catalyzing the NaBH<sub>4</sub>-mediated reduction of 3-nitrophenol. Interestingly, the effect of photo-irradiation yielded dissimilar outcomes for the two nanocomposites. While its influence remained negligible on the nanocomposite originating from laser ablation, it yielded a substantial enhancement in the catalytic efficacy of the nanomaterial synthesized through the polyol approach. This enhancement was intrinsically linked to the silver content within the nanomaterials, thus highlighting the intricate connection between composition and catalytic behaviour.

**Benefits of polyol synthesis of nanoparticles:** Polyol synthesis offers several benefits that make it widely used method for the synthesis of nanoparticles. Some of the key benefits include:

(i) Versatility: Polyol synthesis of the nanoparticles is renowned for its versatility, primarily attributed to its ability to accommodate a wide range of materials, sizes and shapes [89-91]. This method is applicable to diverse nanoparticles, encompassing noble metals, metal oxides and even bimetallic alloys. The adaptability of the polyol method empowers researchers to fine-tune synthesis conditions, resulting in nanoparticles tailored to specific needs across various fields of application.

(ii) Scalability: Polyol synthesis stands out for its simplicity and scalability, making it an ideal choice for large-scale nanoparticle production. This method's straightforward nature allows researchers to efficiently replicate the process in larger quantities, ensuring consistent results [92,93]. As a result, the potential for industrial applications of nanoparticles becomes more feasible due to the ease of upscaling through the polyol synthesis approach.

(iii) Homogeneity: The polyol synthesis has gained recognition for its ability to consistently yield nanoparticles characterized by a narrow size distribution and remarkable homogeneity [94,95]. This method's controlled conditions promote the formation of particles with minimal variation in size, ensuring a high degree of uniformity. The precise control over synthesis parameters empowers researchers to finely tune the process, resulting in nanoparticles that possess highly desirable properties for various applications.

(iv) Cost-effectiveness: Polyols have established themselves as readily accessible and economically efficient reducing agents. Their widespread availability, coupled with their costeffectiveness, renders them a practical choice for nanoparticle synthesis [96,97]. The utilization of polyols not only ensures efficient reduction of precursor materials but also contributes to the overall viability of nanoparticle production processes.

(v) **Temperature control:** The distinctive advantage of polyol synthesis lies in its capacity to be carried out at comparatively low temperatures, effectively mitigating concerns of particle agglomeration or undesired side reactions [98-100]. This controlled and milder temperature environment safeguards the formation of nanoparticles from undesirable interactions, ensuring the production of homogeneous and well-dispersed nanoparticles. By minimizing the risk of agglomeration and side reactions, polyol synthesis stands as a reliable method to obtain nanoparticles with consistent properties and performance.

(vi) High yield: Polyol synthesis is esteemed for its ability to generate a significant yield of nanoparticles while minimizing waste. This approach's efficiency in converting precursor materials into nanoparticles is key to its waste reduction capabilities. By optimizing the reaction conditions and precursor utilization, researchers can maximize the conversion efficiency, resulting in a higher percentage of nanoparticles and less wastage, aligning with sustainable and resource-efficient practices [101,102].

(vii) Controlled morphology: Polyol synthesis offers a remarkable degree of control over nanoparticle shapes and structures by manipulating reaction conditions and incorporating stabilizing agents. This method's flexibility allows researchers to precisely guide the growth and arrangement of nanoparticles, resulting in tailored shapes and structures [103-

105]. By strategically adjusting factors such as temperature, precursor concentration and stabilizing agents, polyol synthesis becomes a versatile tool to engineer nanoparticles with specific morphologies, opening avenues for applications reliant on distinct geometries and functionalities.

(viii) Facile surface functionalization: Polyol synthesis holds a unique advantage in enabling facile functionalization of metal surfaces with diverse ligands or stabilizing agents [106-109]. This adaptability empowers researchers to introduce specific functionalities or tailor-made modifications to nanoparticles, catering to specific applications. By customizing the surface properties through ligands or stabilizers, the polyol synthetic method enhances the nanoparticles' suitability for targeted applications, amplifying their potential impact in fields ranging from catalysis to biomedicine.

The unique properties of polyols, resembling the water's solidities, allow for easy and cost-effective utilization of metal salts as starting materials, making polyols akin to water equivalents with high-boiling solvents. A polyol serves as both a solvent and a reducing agent, offering numerous advantages. Its high boiling point enables elevated temperature synthesis without pressure requirements and its reducing nature safe-guards nanoparticles from oxidation. The coordination of polyols with metal precursors minimizes coalescence and their high viscosity promotes controlled growth, leading to precise structures. Moreover, adapting the polyol, like using triethylene glycol (TEG), allows for diverse nanoparticle shapes [110]. The microwave-polyol process further enhances reaction kinetics, energy efficiency and phase selectivity.

**Limitations of polyol synthesis of nanoparticles:** Polyols can be considered as green solvent because of its biocompatibility, biodegradability and sustainability, which is highly relevant for industrial synthesis and have contributed to its widespread adoption across various scientific disciplines and industries. Some of the key limitations of synthesis are:

(i) Stabilization and agglomeration issues: Polyolsynthesized nanoparticles may require surface modification or stabilizing agents, such as surfactants or capping ligands, to prevent nanoparticles from agglomerating or coalescing. Achieving proper functionalization without altering the desired properties can be complex. The presence of stabilizing agents may affect the surface chemistry of the nanoparticles and can pose challenges in some applications such as catalysis or biomedical uses.

(ii) Reactivity and reproducibility: Polyol synthesis process can be sensitive to various parameters, such as reaction temperature, time and choice of precursors and stabilizing agents. As a result, it may be challenging to reproduce the same nanoparticle characteristics across different experiments or between different research groups.

(iii) Limited material compatibility: Polyol synthesis is particularly suitable for metallic nanoparticles but may not be as effective for producing nanoparticles of certain other materials. Some materials may not readily dissolve in polyols or may decompose under the reaction conditions.

(iv) Challenges in doping and alloying: Incorporating dopants or forming alloyed nanoparticles through polyol synthesis

can be difficult, as it requires precise control over the incorporation process and may lead to compositional inhomogeneity.

### Conclusion

Polyol synthesis is widely used method for the preparation of nanoparticles, due to its simplicity, scalability and verstalility. The polyol synthesis method allows for the production of nanoparticles with well-controlled size, shape and composition. By varying the reaction parameters and the type of metal precursors used, researchers can tailor the synthesis process to obtain nanoparticles with specific properties suitable for various applications, as mentioned in the previous research. Although polyol synthesis has certain limitations but it remains a valuable method for the synthesis of a wide range of nanoparticles. Scientists and researchers continue to explore ways to overcome the challenges of polyol synthesis through the development of modified synthesis methods, use of different precursors and stabilizing agents and better understanding of the underlying chemical processes. Moreover, combining polyol synthesis with other techniques, such as sol-gel or hydrothermal methods, can enable the production of nanoparticles with enhanced properties and better control over their characteristics.

## **CONFLICT OF INTEREST**

The authors declare that there is no conflict of interests regarding the publication of this article.

### REFERENCES

- F. Fiévet, S. Ammar-Merah, R. Brayner, F. Chau, M. Giraud, F. Mammeri, J. Peron, J.Y. Piquemal, L. Sicard and G. Viau, *Chem. Soc. Rev.*, 47, 5187 (2018); <u>https://doi.org/10.1039/C7CS00777A</u>
- S. Ammar and F. Fiévet, *Nanomaterials*, 10, 1217 (2020); https://doi.org/10.3390/nano10061217
- H. Dong, Y.C. Chen and C. Feldmann, *Green Chem.*, **17**, 4107 (2015); https://doi.org/10.1039/C5GC00943J
- T. Rice, E. Zannini, E. K. Arendt and A. Coffey, *Crit. Rev. Food Sci. Nutr.*, **60**, 2034 (2020); <u>https://doi.org/10.1080/10408398.2019.1625859</u>
- K. Lang, R.J. Sánchez-Leija, R.A. Gross and R.J. Linhardt, *Polymers*, 12, 2969 (2020);
- https://doi.org/10.3390/polym12122969 6. F. Fiévet and R. Brayner, *Nanomaterials: A Danger or a Promise*, 1 (2012);
- https://doi.org/10.1007/978-1-4471-4213-3\_1 7. F. Fievet, F. Fievet-Vincent, J.-P. Lagier, B. Dumont and M. Figlarz, J.
- Mater. Chem., **3**, 627 (1993); https://doi.org/10.1039/jm9930300627
- F. Fievet, J.P. Lagier and M. Figlarz, *MRS Bull.*, 14, 29 (1989); <u>https://doi.org/10.1557/S0883769400060930</u>
- M. Abbas, M. Nazrul Islam, B. Parvatheeswara Rao, T. Ogawa, M. Takahashi and C.G. Kim, *Mater. Lett.*, **91**, 326 (2013); <u>https://doi.org/10.1016/j.matlet.2012.10.019</u>
- T. Zhao, R. Sun, S. Yu, Z. Zhang, L. Zhou, H. Huang and R. Du, *Colloids Surf. A Physicochem. Eng. Asp.*, **366**, 197 (2010); https://doi.org/10.1016/j.colsurfa.2010.06.005
- T.M.D. Dang, T.T.T. Le, E.F. Blanc and M.C. Dang, *Adv. Nat. Sci: Nanosci. Nanotechnol.*, **3**, 035004 (2012); <u>https://doi.org/10.1088/2043-6262/3/3/035004</u>
- M. Hosni, I. Hinkov, C. Ricolleau, T. Pauporté, S. Farhat and N. Jouini, J. Surf. Eng. Mater. Adv. Technol., 06, 1 (2016); https://doi.org/10.4236/jsemat.2016.61001
- N. Zayyoun, L. Bahmad, L. Laânab and B. Jaber, *Appl. Phys., A Mater. Sci. Process.*, **122**, 488 (2016); <u>https://doi.org/10.1007/s00339-016-0024-9</u>

- 14. Y. Zhao, J.J. Zhu, J.-M. Hong, N. Bian and H.Y. Chen, Eur. J. Inorg.
- Chem., 4072 (2004); https://doi.org/10.1002/ejic.200400258
   J. Zhu, Y. Wang, X. Wang, X. Yang and L. Lu, Powder Technol., 181,
- J. Zhu, T. wang, X. wang, X. rang and L. Lu, *Powder Technol.*, 101, 249 (2008); https://doi.org/10.1016/j.powtec.2007.05.008
- T. Sheela, Y.A. Nayaka, R. Viswanatha, S. Basavanna and T.G. Venkatesha, *Powder Technol.*, 217, 163 (2012); <u>https://doi.org/10.1016/j.powtec.2011.10.023</u>
- C. Iacovita, G.F. Stiufiuc, R. Dudric, N. Vedeanu, R. Tetean, R.I. Stiufiuc and C.M. Lucaciu, *Magnetochemistry*, 6, 23 (2020); <u>https://doi.org/10.3390/magnetochemistry6020023</u>
- F. Fiévet, J.P. Lagier, B. Blin, B. Beaudoin and M. Figlarz, *Solid State Ion.*, **32-33**, 198 (1989); https://doi.org/10.1016/0167-2738(89)90222-1
- M. Smuda, J. Ströh, N. Pienack, A. Khadiev, H. Terraschke, M. Ruck and T. Doert, *Dalton Trans.*, **51**, 17405 (2022); https://doi.org/10.1039/D2DT02273G
- S. Komarneni, D. Li, B. Newalkar, H. Katsuki and A.S. Bhalla, Langmuir, 18, 5959 (2002); https://doi.org/10.1021/la025741n
- 21. I.L. Simakova and D.Y. Murzin, *Adv. Nanomat. Catal. Energy*, **99**, 99 (2019);
- https://doi.org/10.1016/B978-0-12-814807-5.00004-8 22. G.Y. Qiao, Q. Xu, J. Yin, A. Wang and G. Xu, *J. Supercrit. Fluids*, **128**, 18 (2017);
- https://doi.org/10.1016/j.supflu.2017.05.004 23. B.G. Rao, D. Mukherjee and B.M. Reddy, *Nanostruc. for Novel Therapy*, **1**, 1 (2017);
- https://doi.org/10.1016/B978-0-323-46142-9.00001-3
- T. Mori and T. Hegmann, J. Nanopart. Res., 18, 295 (2016); https://doi.org/10.1007/s11051-016-3587-7
- H.B. Jeon, P.V. Tsalu and J.W. Ha, *Sci. Rep.*, 9, 13635 (2019); https://doi.org/10.1038/s41598-019-50032-3
- S. Yu, J.A. Hachtel, M.F. Chisholm, S.T. Pantelides, A. Laromaine and A. Roig, *Nanoscale*, 7, 14039 (2015); https://doi.org/10.1039/C5NR03113C
- K. Gharbi, A. Mezni, V. Collière, K. Philippot, C. Amiens, D.C. Pradines and L.S. Smiri J. Tunisian Chem. Soc., 19 335 (2017).
- N.V. Long, Commun. Phys., 31, 329 (2021); https://doi.org/10.15625/0868-3166/2021/15933
- D.R. Rasmussen, M.F. Nielsen and J. Quinson, *Chemistry*, 5, 900 (2023); https://doi.org/10.3390/chemistry5020061
- J. Quinson, T.M. Nielsen, M. Escudero-Escribano and K.M.Ø. Jensen, *Colloids Surf. A Physicochem. Eng. Asp.*, 675, 131853 (2023); <u>https://doi.org/10.1016/j.colsurfa.2023.131853</u>
- T.H. Chiang, K. Wu and T.-E. Hsieh, *IEEE Trans. Nanotechnol.*, 13, 116 (2014); https://doi.org/10.1109/TNANO.2013.2294174
- G. Dzido, P. Markowski, A. Malachowska-Jutsz, K. Prusik and A.B. Jarzêbski, J. Nanopart. Res., 17, 27 (2015); https://doi.org/10.1007/s11051-014-2843-y
- Z. Fereshteh, R. Rojaee and A. Sharifnabi, Superlattices Microstruct., 98, 267 (2016);
- <u>https://doi.org/10.1016/j.spmi.2016.08.034</u>
   Z. Lalegani, S.A.S. Ebrahimi, B. Hamawandi, L. La Spada and M.S. Toprak, *Opt. Mater.*, **108**, 110381 (2020); <u>https://doi.org/10.1016/j.optmat.2020.110381</u>
- S. Zeroual, P. Estellé, D. Cabaleiro, B. Vigolo, M. Emo, W. Halim and S. Ouaskit, *J. Mol. Liq.*, **310**, 113229 (2020); https://doi.org/10.1016/j.molliq.2020.113229
- M. Pernot, B. Jaspard-vinassa, A. Abelanet, S. Rubin, S. Jeanningros, I. Forfar, L. Cetran, M.H.-Y. Yu, E. Balse, S. Hatem, P. Dufourcq, T. Couffinhal and C. Duplàa, *Sci. Rep.*, **12**, 8 (2022); <u>https://doi.org/10.1038/s41598-021-03795-7</u>
- J. Boita, L. Nicolao, M.C.M. Alves and J. Morais, *New J. Chem.*, 41, 14478 (2017); https://doi.org/10.1039/C7NJ03056H
- Y.J. Lee, K. Kim, I.S. Shin and K.S. Shin, J. Nanopart. Res., 22, 8 (2020); https://doi.org/10.1007/s11051-019-4727-7
- V.V. Gande and S. Pushpavanam, J. Flow Chem., 11, 661 (2021); https://doi.org/10.1007/s41981-021-00169-y

- T.D. Bao, N.H. Le and H.H. Lam, *Chem. Eng. Trans.*, 97, 331 (2022); https://doi.org/10.3303/CET2297056
- 41. P. Sankarasubramanian and E.N. Ganesh, *Int. J. Composite Constitent Mater.*, **8**, 27 (2022).
- L. Dorjee, R. Gogoi, D. Kamil, R. Kumar and A. Verma, *Phytoparasitica*, 51, 593 (2023);
  - https://doi.org/10.1007/s12600-023-01060-3
- G. Rajakumar, A.A. Rahuman, K. Velayutham, K. Jeyasubramanian, J. Ramyadevi, A. Marikani, G. Elango, C. Kamaraj, T. Santhoshkumar, S. Marimuthu, A.A. Zahir, A. Bagavan, C. Jayaseelan, A.V. Kirthi, M. Iyappan and C. Siva, *Vet. Parasitol.*, **191**, 332 (2013); <u>https://doi.org/10.1016/j.vetpar.2012.08.028</u>
- 44. T. Xie, L. Shi, J. Zhang and D. Zhang, *Chem. Commun.*, **50**, 7250 (2014); https://doi.org/10.1039/C4CC01441C
- A. Moumen, M. Fattouhi, K. Abderrafi, M. El Hafidi and S. Ouaskit, J. *Cluster Sci.*, **30**, 581 (2019); <u>https://doi.org/10.1007/s10876-019-01517-8</u>
- 46. N.A. Nguyen, J.G. Park and S.H. Kim, *Bull. Korean Chem. Soc.*, 34, 2865 (2013);
- https://doi.org/10.5012/bkcs.2013.34.10.2865 47. N.N. Minh, H.T.N. Quyen and T.T. Xuan, *Dig. J. Nanomater. Biostruct.*,
- 17, 597 (2022);
- https://doi.org/10.15251/DJNB.2022.172.597
- D. Kaya, H.H. Isik, I.B. Isik, G. Sigircik, T. Tuken, F. Karadag and A. Ekicibil, *Int. J. Hydrogen Energy*, 48, 14633 (2023); https://doi.org/10.1016/j.ijhydene.2023.01.049
- C. Rodríguez-Proenza, J. Palomares-Báez, M. Chávez-Rojo, A. García-Ruiz, C. Azanza-Ricardo, A. Santoveña-Uribe, G. Luna-Bárcenas, J. Rodríguez-López and R. Esparza, *Materials*, 11, 1882 (2018); <u>https://doi.org/10.3390/ma11101882</u>
- 50. A. Higareda, F. Mares, D. Bahena and R. Esparza, *ChemCatChem*, **15**, e202300030 (2023);
- https://doi.org/10.1002/cctc.202300030
- D. Kaya, I. Demiroglu, I.B. Isik, H.H. Isik, S.K. Çetin, C. Sevik, A. Ekicibil and F. Karadag, *Int. J. Hydrogen Energy* (2023); https://doi.org/10.1016/j.ijhydene.2023.06.100
- F. Mares-Briones, A. Higareda, J.L. Lopez-Miranda, R. Mendoza-Cruz and R. Esparza, *Nanomaterials*, 13, 1396 (2023); https://doi.org/10.3390/nano13081396
- 53. Y. Wang, Z. Wang, C. Xu and S. Zhou, *J. Phys. Chem. C*, **127**, 4033 (2023); https://doi.org/10.1021/acs.jpcc.2c07661
- S. Sargazi, M.R. Hajinezhad, A. Rahdar, M. Mukhtar, M.K. Jahromi, M.A. Kashi, S.A. Arani, M. Barani and F. Baino, *Appl. Phys., A Mater. Sci. Process.*, **127**, 772 (2021); <u>https://doi.org/10.1007/s00339-021-04917-8</u>
- A.S. Uribe, J.M. Cornejo, D. Bahena, J. Ledesma, R. Pérez and R. Esparza, *Electrocatalysis*, 11, 536 (2020); https://doi.org/10.1007/s12678-020-00613-y
- S. Bundli, P. Dhak, M. Jensen, A.E. Gunnæs, P.D. Nguyen, H. Fjellvåg and A.O. Sjåstad, *J. Alloys Compd.*, **779**, 879 (2019); <u>https://doi.org/10.1016/j.jallcom.2018.11.301</u>
- D. Tongsakul, S. Nishimura and K. Ebitani, ACS Catal., 3, 2199 (2013); https://doi.org/10.1021/cs400458k
- P.C. Karthika, A. Ratnakar, N. Balasubramaniam, P. Sahatiya, K. Teja and A.A. Mohan, *Asian J. Chem.*, 25(Suppl. 1), S381 (2013).
- 59. Y.J. Park, J.-H. Park, J. Chul Ro and S.-J. Suh, *Appl. Surf. Sci.*, **613**, 155976 (2023);
- https://doi.org/10.1016/j.apsusc.2022.155976
  60. A. Rajesh, S. Vignesh, M. Srinivasan, G. Venkatesh and P. Ramasamy, *Ferroelectrics*, **599**, 16 (2022);
- https://doi.org/10.1080/00150193.2022.2113636
- N.F. Martinez, G. Franceschin, T. Gaudisson, S.H. Khlifa, S.G. Derouich, N. Yaacoub, J.M. Greneche, N. Menguy, R. Valenzuela and S. Ammar, *Sci. Rep.*, 9, 19468 (2019); <u>https://doi.org/10.1038/s41598-019-55649-y</u>
- N. Muthuswamy, J.L.G. de la Fuente, D.T. Tran, J. Walmsley, M. Tsypkin, S. Raaen, S. Sunde, M. Rønning and D. Chen, *Int. J. Hydrogen Energy*, 38, 16631 (2013); <u>https://doi.org/10.1016/j.ijhydene.2013.02.056</u>
- N. Hikmah, N.F. Idrus, J. Jai and A. Hadi, *IOP Conf. Ser. Earth Environ.* Sci., 36, 012050 (2016); https://doi.org/10.1088/1755-1315/36/1/012050

- S. Kheawhom and P. Panyarueng, MRS Proc., 1630, 625 (2014); https://doi.org/10.1557/opl.2014.83
- 65. P. Sapkota, S. Lim and K.F.A. Zinsou, *Catalysts*, **12**, 1369 (2022); https://doi.org/10.3390/catal12111369
- 66. K. Sreekanth and D. Sahu, J. Chem. Pharm. Res., 7, 353 (2015).
- R. Hachani, M. Lowdell, M. Birchall, A. Hervault, D. Mertz, S. Begin-Colin and N.T.K. Thanh, *Nanoscale*, 8, 3278 (2016); <u>https://doi.org/10.1039/C5NR03867G</u>
- A. Gallo-Cordova, S. Veintemillas-Verdaguer, P. Tartaj, E. Mazarío, M.P. Morales and J.G. Ovejero, *Nanomaterials*, 11, 1052 (2021); <u>https://doi.org/10.3390/nano11041052</u>
- H. Kim, P.W. Im, C. Lee, H. Hong, W.S. Lee, C.H. Koo, S.Y. Park, H.-J. Im, S.H. Paek and Y. Piao, *RSC Adv.*, **13**, 2803 (2023); <u>https://doi.org/10.1039/D2RA07190H</u>
- O.R. Vasile, I. Serdaru, E. Andronescu, R. Truscã, V.A. Surdu, O. Oprea, A. Ilie and B.S. Vasile, C. R. Chim., 18, 1335 (2015); https://doi.org/10.1016/j.crci.2015.08.005
- C. Byl, A. Gloter, J.P. Baltaze, D. Bérardan and N. Dragoe, *J. Sol-Gel Sci. Technol.*, 83, 296 (2017); https://doi.org/10.1007/s10971-017-4403-2
- 72. T.E.P. Alves, C. Kolodziej, C. Burda and A. Franco Jr., *Mater. Des.*, **146**, 125 (2018);
- https://doi.org/10.1016/j.matdes.2018.03.013
- S. Soren, S. Kumar, S. Mishra, P.K. Jena, S.K. Verma and P. Parhi, *Microb. Pathog.*, **119**, 145 (2018); <u>https://doi.org/10.1016/j.micpath.2018.03.048</u>
- P.P. Mahamuni, P.M. Patil, M.J. Dhanavade, M.V. Badiger, P.G. Shadija, A.C. Lokhande and R.A. Bohara, *Biochem. Biophys. Rep.*, **17**, 71 (2019); <u>https://doi.org/10.1016/j.bbrep.2018.11.007</u>
- 75. R. Rathore and N. Kaurav, *Mater. Today Proc.*, **54**, 624 (2022); https://doi.org/10.1016/j.matpr.2021.10.207
- 76. A. Zakiyah, R. Subagyo, Y. Kusumawati and H. Juwono, AIP Conf. Proc., 2553, 020030 (2022); <u>https://doi.org/10.1063/5.0105816</u>
- P. Walunj, A. Roy, V.R. Jadhav, P. Athare, A. Dhaygude, J. Aher, J.S. Algethami, D. Lokhande, M.S. Alqahtani, A.M. Bhagare, S. Alghamdi, L.B. Eltayeb, I.S. Al-Moraya, K.K. Yadav, Y. Ahn and B.-H. Jeon, *Front. Bioeng. Biotechnol.*, **11**, 1177981 (2023); <u>https://doi.org/10.3389/fbioe.2023.1177981</u>
- M. Fekadu, D. Zeleke, B. Abdi, A. Guttula, R. Eswaramoorthy and Y. Melaku, *BMC Chem.*, 16, 1 (2022); <u>https://doi.org/10.1186/s13065-022-00795-0</u>
- S.U. Sandhya and S.A. Nityananda, Nanomat. Nanotechnol., 3, 5 (2013); https://doi.org/10.5772/56626
- O.L. Evdokimova, M. Belousova, A.V. Evdokimova, T.V. Kusova, A.E. Baranchikov, K.S. Antonets, A.A. Nizhnikov and A.V. Agafonov, *Cellulose*, 28, 2931 (2021); <u>https://doi.org/10.1007/s10570-021-03689-x</u>
- L.A. Dahonog, M.S.D.D. Vega and M.D.L. Balela, J. Phys. Conf. Ser., 1191, 012043 (2019);
  - https://doi.org/10.1088/1742-6596/1191/1/012043
- S. Maturost, S. Themsirimongkon, S. Saipanya, N. Pongpichayakul, J. Jakmunee, L. Fang and P. Waenkaew, *Int. J. Hydrogen Energy*, 47, 5585 (2022);
  - https://doi.org/10.1016/j.ijhydene.2021.11.199
- Y.R. Baste, V.R. Jadhav, A. Roy, S. Alghamdi, M. Abbas, J.S. Algethami, M. Almehmadi, M. Allahyani, D. Verma, K.K. Yadav, B.H. Jeon and H.K. Park, *Catalysts*, 13, 1143 (2023); <u>https://doi.org/10.3390/catal13071143</u>
- J.A. Adekoya, E.O. Dare, M.A. Mesubi, A.A. Nejo, H.C. Swart and N. Revaprasadu, *Results Phys.*, 4, 12 (2014); <u>https://doi.org/10.1016/j.rinp.2014.02.002</u>
- R. Shamsi, M. Mahyari and M. Koosha, J. Appl. Polym. Sci., 134, 44567 (2016);

https://doi.org/10.1002/app.44567

- T. Tarachand, S. Hussain, N.P. Lalla, Y.-K. Kuo, A. Lakhani, V.G. Sathe, U. Deshpande and G.S. Okram, *Phys. Chem. Chem. Phys.*, 20, 5926 (2018); <u>https://doi.org/10.1039/C7CP07986A</u>
- I.M. Factori, J.M. Amaral, P.H. Camani, D.S. Rosa, B.A. Lima, M. Brocchi, E.R. da Silva and J.S. Souza, ACS Appl. Nano Mater., 4, 7371 (2021); <u>https://doi.org/10.1021/acsanm.1c01334</u>

- J.A. Donadelli, M. Belen, F. Aparicio and M.M. Virginia Moreno, J. Nanopart. Res., 24, 58 (2022); https://doi.org/10.1007/s11051-022-05446-y
- G. Birant, I.M. Ozturk, D. Doganay, H.E. Unalan and A. Bek, ACS Appl. Nanomater., 3, 12231 (2020); <u>https://doi.org/10.1021/acsanm.0c02694</u>
- 90. K. Wang, Y. Liu, J.C. Wang and X. Liang, *ChemNanoMat*, 9, e202300294 (2023);
- https://doi.org/10.1002/cnma.202300294
- T. Dang-Bao, I. Favier and M. Gomez, eds.: K. Philippot and A. Roucoux, Metal Nanoparticles in Polyols: Bottom-up and Top-down Syntheses and Catalytic Applications; In: Nanoparticles in Catalysis: Advances in Synthesis and Applications, Wiley, Chap. 5, pp. 99-122 (2021).
- 92. Y. Shi and J. Fang, J. Phys. Chem. C, 126, 19866 (2022); <u>https://doi.org/10.1021/acs.jpcc.2c05632</u>
- S. Farhat, N. Ouar, M. Hosni, I. Hinkov, S. Mercone, F. Schoenstein and N.N. Jouini, J. Mater. Sci. Chem. Eng., 2, 1 (2014); <u>https://doi.org/10.4236/msce.2014.29001</u>
- 94. F. Li, G. Zhang and H. Abe, *Open Ceram.*, **9**, 100223 (2022); https://doi.org/10.1016/j.oceram.2022.100223
- 95. F. Shiba, U. Mameuda, S. Tatejima and Y. Okawa, *RSC Adv.*, 9, 34589 (2019); https://doi.org/10.1039/C9RA07080J
- 96. T.K. Lahane, J. Agrawal and V. Singh, *Mater. Today Proc.*, **59**, 257 (2022);
- https://doi.org/10.1016/j.matpr.2021.11.108
- J. Tripathi, G.S. Chandrawat, J. Singh, S.N. Tripathi and A. Sharma, J. Alloys Compd., 861, 157977 (2021); https://doi.org/10.1016/j.jallcom.2020.157977
- 98. Z. Huang, J. Xu, Q. Zhang, G. Liu, T. Wu, T. Lin and P. He, *Mater. Today Chem.*, **30**, 101569 (2023); https://doi.org/10.1016/j.mtchem.2023.101569
- M.S. Lee, Y.J. Choi, S.-J. Bak, M. Son, J. Shin and J.Y. Lee, *Nanomaterials*, 12, 3644 (2022); <u>https://doi.org/10.3390/nano12203644</u>

- 100. S. Kaabipour and S. Hemmati, Colloids Surf. A Physicochem. Eng. Asp., 659, 130806 (2023); https://doi.org/10.1016/j.colsurfa.2022.130806
- 101. S. Yala and N. Petchsang, *IOP Conf. Ser.: Mater. Sci. Eng.*, **1234**, 012034 (2022);

https://doi.org/10.1088/1757-899X/1234/1/012034

- 102. M. Parente, M. van Helvert, R.F. Hamans, R. Verbroekken, R. Sinha, A. Bieberle-Hütter and A. Baldi, *Nano Lett.*, **20**, 5759 (2020); <u>https://doi.org/10.1021/acs.nanolett.0c01565</u>
- 103. A.R. Akbarzadeh, I. Mesgarzadeh and R.E. Malekshah, *Chem. Zvesti*, 76, 5761 (2022); https://doi.org/10.1007/s11696-022-02272-3
- 104. Z. Zhu, X. Wang, H. Yu, W. Zhou, Y. Wang, J. Han and F. Guo, *Cryst. Growth Des.*, **23**, 1455 (2023);
- https://doi.org/10.1021/acs.cgd.2c01031 105. Z. Wu, D. Xiao, J. Lee, P. Ren, M. Song and D. Li, *J. Cryst. Growth*, **521**, 34 (2019);

https://doi.org/10.1016/j.jcrysgro.2019.05.028

- 106. S. Liu, B. Yu, S. Wang, Y. Shen and H. Cong, Adv. Colloid Interface Sci., 281, 102165 (2020); <u>https://doi.org/10.1016/j.cis.2020.102165</u>
- 107. G. Zhang, Q. Zhang, T. Cheng, X. Zhan and F. Chen, *Langmuir*, 34, 4052 (2018); https://doi.org/10.1021/acs.langmuir.8b00286
- 108. N. Akkurt, C.L. Altan and M.F. Sarac, J. Supercond. Nov. Magn., 35, 615 (2022); https://doi.org/10.1007/s10948-021-06132-1

109. M.F. Variava, T.L. Church, A.T. Harris and A.I. Minett, J. Mater. Chem. A Mater. Energy Sustain., 1, 8509 (2013); https://doi.org/10.1039/c3ta11319a

110. K.J. Carroll, J.U. Reveles, M.D. Shultz, S.N. Khanna and E.E. Carpenter, J. Phys. Chem. C, 115, 2656 (2011); <u>https://doi.org/10.1021/jp1104196</u>