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Synthesis of Co_3O_4 NPs by solution combustion synthesis (SCS) and their structure morphology: a mini review

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ABSTRACT

This mini-review aims to present the current status of knowledge about cobalt oxide Co_3O_4 nanoparticles more precisely obtained by solution combustion synthesis, which is a process characterized by its high reaction rate and low cost. Unique features of cobalt (II)(III) oxide Co_3O_4 nanoparticles are size between 12 and 60 nm, and high surface area and porosity that enhances their performances in fields such as energy storage, environmental and sensing applications. By analysis of various synthesis parameters such as fuel-to-oxidizer ratios, precursor materials, and thermal conditions, this review elucidates how these factors influence particle characteristics and functionality. Furthermore, while extensive research highlights the efficacy of Co_3O_4 nanoparticles in energy applications, there is a notable need to further studies focusing on their environmental applications, appealing to young scientists to come up with improved solution seeking innovative research areas of nanotechnology and material science. The review offers more avenues for using Co_3O_4 nanoparticles in solving some of the pressing global environmental issues.

Keywords: Co_3O_4 nanoparticles, SCS method, porosity, sensitive sensors, energy storage

1. Introduction

The particles with sizes between (1-100) nanometers are also known as nanoparticles (NPs) that are widely studied in the 21st century due to their unique properties. Among their high surface area, size in nanometer range, and surface porosity distinguish nanoparticles from their bulk material [1]. They are Compatible with the potential to solve global immediate concerns and achieve the United Nation 17 Sustainable Development Goals through novel developed smart technologies [2]. Moreover, advances in green synthesis methods, such as using natural extracts from lemon [3] or psyllium husk [4], have improved the environmental sustainability of Co_3O_4 nanoparticle production.

Co_3O_4 nanoparticles possess unique properties like high density, high melting point, magnetic properties and an optical band gap, making them suitable for emerging technologies [5]. These attributes make them highly desirable for uses in applications such as energy storage, environmental remediation, sensitive sensing [6], and catalysts [7]. Recent studies indicate that Co_3O_4 nanoparticles exhibit a remarkable ability to generate reactive oxygen species (ROS), which opens pathways for applications in cancer therapy and antibacterial treatments [8]. Additionally, they have shown promising performance as anode materials in lithium-ion batteries, with significant cycling stability and energy density [9]. Their unique spinel structure also facilitates oxygen evolution reaction (OER), making them effective electrocatalysts for clean energy applications [4]. Furthermore, their high porosity and crystal surfaces enhance their performance in superior advanced technological devices, including pollution control and water purification systems [2].

Solution combustion synthesis (SCS) is now considered to be one of the favorable methods for the synthesis of Co_3O_4 NPs. The SCS method was first used by K.C. Patil in the 1980s for obtaining ceramic composites [10]. This method relies on exothermic processes that can be used to synthesize nanoparticles at high reaction rates while having

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small size and uniform shapes [11]. SCS-based synthesis of Co_3O_4 NPs results in high-quality materials that maintain superior optical properties along with increased surface area for optimizing the degradation of organic pollutants including dyes [12]. SCS synthesized Co_3O_4 NPs exhibit high specific capacitance along with excellent charge-discharge cycling stability, making them ideal for supercapacitors [13, 14]. The cost-effectiveness and scalability of SCS further establish it as an important approach in nanomaterial production [15]. Smagulova et al. [16], along with Mansurov and Smagulova [17], highlight its significance in fabricating smart and functional materials, including electroconductive textiles and catalytic coatings.

This review provides a detailed discussion on synthesis and characterizations of Co_3O_4 NPs, particularly through the SCS method. Clarifying the significance of these nanoparticles and their versatility, the review emphasizes the need for the continuous investigation of the nanomaterials' properties and capabilities [18]. Limited publications exist that describe Co_3O_4 NPs synthesized via the SCS method as promising materials for environmental remediation [5] and sensitive sensors [7]. Further research is essential to fully understand their properties and unlock new applications. Expanding the scope of research in this area is critical to developing innovative solutions for global challenges and enhancing the understanding of Co_3O_4 NPs properties. By examining the importance of these nanoparticles and their broad range of applications, this review underscores the need for continued research to fully understand their behavior and unlock their potential. SCS synthesis of Co_3O_4 NPs offers a great deal of prospect for material science development in terms of translating all the theories into practice. The mini review aimed to evaluate previous findings and examines current developments regarding synthesis of Co_3O_4 NPs through the SCS method which is crucial for identifying opportunities for young scientist's further studies, contributing to the growing body of literature on Co_3O_4 NPs and their transformative potential.

2. Materials and methods

Co_3O_4 NPs are synthesized by solution combustion synthesis (SCS) technique using the combination of fuel and oxidizers which results the highly crystalline in nature. This review demonstrates a systematic view in the critical evaluation of these developments in SCS specifically in relation to concerns like the fuel to oxidizer ratio, choice of precursors, thermal conditions and the resulting structures obtained. A literature review was conducted through Scopus, Web of Science, and Science Direct to obtain only the well-reviewed articles. Studies, which were published up to the end of 2024, considering with regard to both the initiation of the topic and present trends. As the main subject of the article was the synthesis of Co_3O_4 NPs through SCS method. For the search, keywords and Boolean operators like 'Co_3O_4 nanoparticles', 'Solution combustion synthesis' Porous nanostructures', Morphological properties were employed. Studies that employed techniques of scanning electron microscopy, transmission electron microscopy, X-ray diffraction, and nitrogen adsorption-desorption isotherms were selected based on the following inclusion criteria: Articles that primarily investigated the synthesis of Co_3O_4 NPs by the SCS method and provided data on the influence of synthesis parameters on porosity, crystallinity, and morphology.

In Fig. 1a, priority was given to articles published between 2010 and 2024 on the synthesis mechanisms, morphological characterization, and performance applications. Studies were thematically categorized into sections: some of them include "Impact of SCS Parameters" [9, 19], "Structural and Morphological Analysis" [20, 21], "Functional Applications" [22, 23] and "Environmental Implications of SCS" [24].

3. Cobalt oxide (Co_3O_4) NPs

Cobalt oxide (Co_3O_4) is a transition metal oxide with a density of approximately 6.11 g/cm³ and a melting point of around 895 °C [25]. This inorganic material exists as a tiny black powder compound that shows significant magnetic properties. As an electronic semiconductor Co_3O_4 exhibits p-type characteristics because positive charge carriers (known as holes) prevail within its material structure [26]. As shown by its chemical formula Co_3O_4 the system features cobalt ions that exist in two different oxidative states of Co^{2+} and Co^{3+}. The mixed valence character of Co_3O_4 allows scientists to represent it structurally as cobalt(II) oxide (CoO) and cobalt(III) oxide (Co_2O_3) combined in a $CoO{\cdot}Co_2O_3$ expression.

The presence of both Co^{2+} and Co^{3+} oxidation states in Co_3O_4 compound enhances its redox reactions and electrical conductivity performance. Photocatalytic potential and semiconducting behavior in Co_3O_4 materials depend on the existence of two intrinsic band gaps which enable electron transitions within valence band to conduction band [25, 26]. As shown

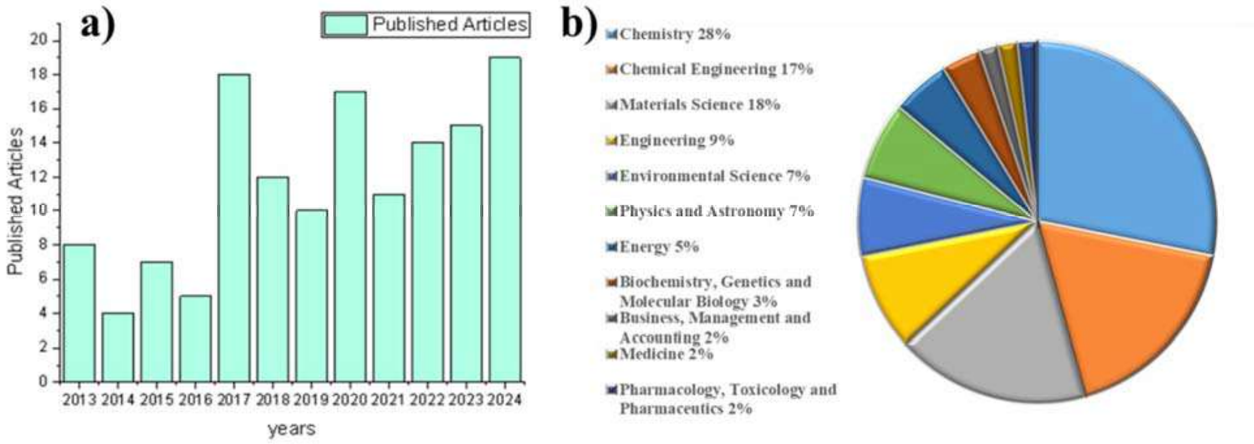

Fig. 1. (a) Recent published articles about Co_3O_4 NPs from 2013 to 2024, and (b) Recent publications of Co_3O_4 NPs in a wide range of fields. (Data retrieved from the 'Scopus' database on 20th Dec 2024 using the terms 'Co_3O_4 NPs and solution combustion synthesis' to search within article title, abstract, and keywords).

in Fig. 1b, recently many scientists and researchers had focused on, many researches and articles in a wide range of fields published, due to the unique properties and many proses of Co_3O_4 NPs.

4. Results and discussion

4.1. Synthesis

SCS method involves an exothermic redox process between cobalt oxidizing precursors such as cobalt nitrate hexahydrate oxidizer and a fuel such as glycine, citric acid, or urea to synthesize Co_3O_4 NPs. The experimental process (as shown in Fig. 2) for this synthesis includes the following steps and materials; cobalt nitrate hexahydrate ($Co(NO_3)_2{\cdot}6H_2O$), citric acid as fuel, deionized water, ethanol and crucible [9]. First, a precursor solution is distilled by weighing the F/O ratio concentration of cobalt nitrate hexahydrate relative to the assembler size of Co_3O_4 NPs. The compound is then dissolved in deionized water to make clear aqueous solution depending on the amount needed for subsequent experiment. Subsequently, the calculated amount of citric acid is then added as the fuel over the cobalt nitrate solution and the reactions mixture is stirred until it is fully dissolved.

The combustion reaction is started by putting the crucible with the precursor solution over a hot plate. The formation of the Co_3O_4 NPs is through exothermic reactions when the solution is heated. At the end, the combustion is complete then the reaction can be quenched by adding ethanol to hot solution. The particles are then pelleted by the help of centrifugation or filtration and washed with ethanol and distilled water to remove any unreacted chemicals. Finally, the collected nanoparticles are then reduced under a moderate temperature. In order to improve the crystallinity and structure rigidity of the dried nanoparticles can also be subjected to annealing. Such an approach also helps in getting the format of the Co_3O_4 NPs that are functional for several applications [27].

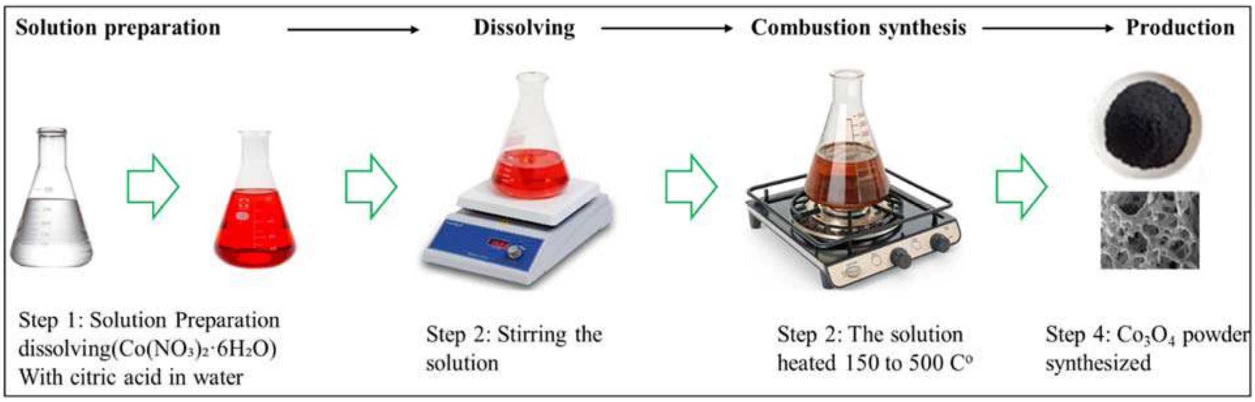

Fig. 2. Experimental setup for Co_3O_4 NPs synthesis by SCS method.

4.2. Mechanism of Solution Combustion Synthesis

The cobalt oxide synthesis through combustion requires various stages which start with precursor thermal decomposition followed by fuel oxidation and an exothermic reaction leading to cobalt oxide nanoparticles. The starting point of this method involves heating cobalt nitrate hexahydrate which results in formation of intermediary cobalt oxide compounds (1). The primary role of citric acid in this reaction consists both as the main fuel component and a reducing agent. The citric acid molecule becomes oxidized as part of the reaction process to generate carbon monoxide gas along with the additional reaction byproducts (2). The carbon monoxide production in this stage proceeds to react with oxygen byproducts that decompose the cobalt nitrate precursor. This leads to an extremely heat-releasing combustion reaction (3). The heat generated from combustion creates an ideal environment for Co_3O_4 NPs production and subsequent growth resulting in their development with desired properties (4).

$$2Co(NO_3)_2 \cdot 6H_2O \xrightarrow{Heat} 2CoO + 4NO_2 + 12H_2O + O_2 \quad (1)$$

$$2C_6H_8O_7 + 6O_2 \xrightarrow{Combustion} 6CO_2 + 8H_2O + 6CO \quad (2)$$

$$6CO + 6NO_2 \xrightarrow{Heat} 6CO_2 + 3N_2 + 3O_2 \quad (3)$$

$$6CoO + 4O_2 \xrightarrow{High\,Temperature} 2Co_3O_4 \quad (4)$$

4.3. Characterization

Powder X-ray diffraction (XRD) and Raman spectroscopy are used for study of structural characteristics of Co_3O_4 NPs as shown in Fig. 3. First, the morphology of Co_3O_4 NPs has been characterized using XRD pattern which revealed that the Co_3O_4 NPs have a crystalline structure. In Fig. 3a the X-ray Diffraction pattern indicates seven peaks at 19°, 31.3°, 36.8°, 38.7°, 44.8°, 55.8°, and 59.4° degrees corresponding to (111), (220), (311), (222), (400), (422), and (511) planes respectively [5]. These positions allow us to confirm the cubic spinel crystal structure with the space group Fd3m. The calculated unit cell parameter (a = 8.085 Å) is in close agreement with the standard value observed for Co_3O_4 NPs. From the analysis of Gaussian broadening of the peaks using Scherrer formula, the average size of the crystallites was estimated to be approximately 40 nm. Raman spectroscopy analysis also confirms the results of XRD in (Fig. 3b) five distinctive Raman active vibrational modes: F2g3 at 184 cm^{-1}, Eg at 464 cm^{-1}, F2g2 at 506 cm^{-1}, F2g1 at 601 cm^{-1}, and Eg at 670 cm^{-1} are identified unambiguously [30]. The band at 670 cm^{-1} corresponds to CoO symmetric stretching vibration of octahedral CoO_6 whereas the band 184 cm^{-1} is relevant to tetrahedral sites CoO_4. Other peaks are related to the mixed motion of oxygen at tetrahedral and octahedral sites [31].

Scanning electron microscopy (SEM) and transmission electron microscopy (TEM) were widely used for the observation of their surface morphology and particle size (Fig. 4). It was concluded that the cobalt oxide particles measuring 12 to 60 nm, with an average size of about 36 nm

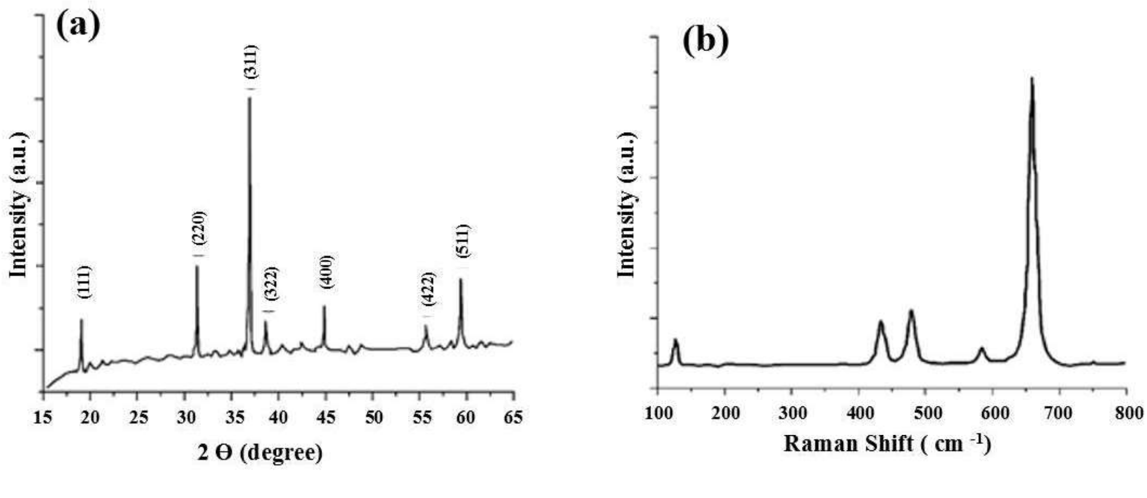

Fig. 3. (a) XRD pattern of Co_3O_4 NPs obtained by SCS method from cobalt nitrate- glycine mixture at ϕ=1.5 and (b) Raman spectrum for Co_3O_4 powder. (Adapted from Michalska et al., Beilstein J. Nanotechnol., 2021, 12, 424–431, under CC BY 4.0) [32].

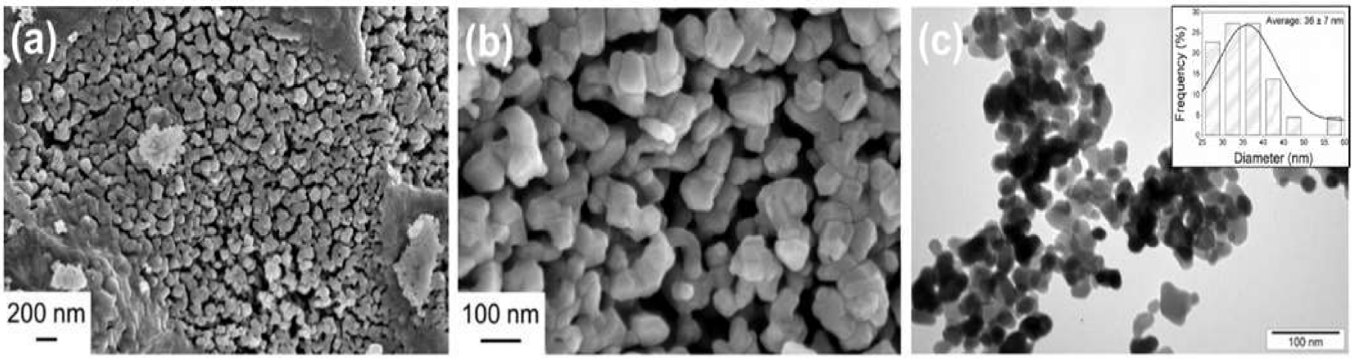

Fig. 4. SEM and TEM images of Co_3O_4 NPs: (a) uniform and porous particle arrangement, (b) faceted morphology with minimal agglomeration, and (c) TEM image with an average particle size of 36 nm. (Adapted from Michalska et al., Beilstein J. Nanotechnol., 2021, 12, 424–431, under CC BY 4.0) [32].

and loose arrangement featuring several empty spaces from the SEM and TEM imaging results. Low-magnification SEM image (Fig. 4a) reveals particles displaying uniform distribution across the surface and forming a loosely packed structure that contains observable pores [32]. The SEM image with high magnification in (Fig. 4b) demonstrates that synthesized nanoparticles show faceted morphology and feature some aggregation although distinct boundaries between particles remain observable. The TEM image in (Fig. 4c) shows distinct nano sized structures that exist separately from each other without forming compact clusters. Results from Fig. 2c demonstrate particle dimensions between 12 and 60 nm with an average size of 36 nm that reflects the crystal size determined through XRD analysis using the Scherer formula. Moreover, solution combustion synthesis enables the production of porous fine powders that develop characteristic microstructures during rapid exothermic reactions [27]. The small particles together with porous object structure enables higher surface area that results in improved electrochemical and catalytic properties [8, 29].

BET analysis enables the measurement of specific surface area together with porosity characteristics in Co_3O_4 nanoparticles as these parameters play a key role in determining their electrochemical and catalytic performance [28]. The characteristics of these materials depend strongly on synthesis method along with precursors and synthesis temperature as presented in Table 1 additionally, Mn-doped Co_3O_4 showed enhanced surface area and catalytic activity for oxidizing the CO/CH_4 gases to CO_2 and H_2O [23], while Ag-Co_3O_4 and porous Co_3O_4 demonstrated improved multifunctionality and supercapacitive

Table 1. A comparison of the BET Specific surface area (SSA) of pure and doped Co_3O_4 NPs with synthesis conditions in SCS method and applications

No	Method	Precursors	Catalyst agent	Material	SSA m^2g^{-1}	Synthesis temperature	Proposed Applications	Ref.
1	SCS	$Co(NO_3)_2·6H_2O$ and urea	$NH_2)_2CO$	Co_3O_4 nanoparticles	39	800	Catalysis	[20]
2	SCS	$C_4H_6O_4Co·4H_2O$ and D-(+)-glucose	$(C_6H_{12}O_6)$	Spinel-structured Co_3O_4	3	700	Lithium-ion batteries	[32]
3	SCS	$Co(NO_3)_2·6H_2O$ and urea in deionized water	-	Nanocrystalline Co_3O_4	10	600	Gas sensing	[34]
4	SCS	$Co(NO_3)_2·6H_2O$ and urea	-	Co_3O_4	12.8	350	Supercapacitor	[34]
5	SCS	$Co(NO_3)_2·6H_2O$ + varying urea/fuel ratios	-	Porous Co_3O_4 Doped Co_3O	91.5	300	Supercapacitor (enhanced)	[33]
6	SCS	Co precursor + reducing agent	-		52.9	450	High-capacity supercapacitor	[35]

performance due to increased porosity [7, 33]. This indicates that dopants and optimized synthesis conditions can effectively enhance the surface features of Co_3O_4 for advanced applications.

4.4. Application

Co_3O_4 NPs have a wide range of applications, some very abundant applications are as follows: in biomedical science (antibacterial, antifungal, antiviral, antileishmanial, medications, anticancer, and drug delivery), gas sensors, solar specific absorbing materials. In energy strorage and conversion (anode materials in lithium-ion batteries, supercapacitors), pigments and dyes, electromagnetic field-emitting materials, capacitors, diverse catalysis, magneto-resistive devices, and electronic lightweight films. Zhao et al. used laser-induced graphene to incorporate Co_3O_4 NPs for a flexible and highly sensitive enzyme-free glucose biosensor, highlighting their versatility in sensing applications [36]. These nanoparticles have also widely found applications in environmental remediation. As recently discussed in Anele et al. investigation of recent trends in the environmental remediation of bacteria in wastewater using Co_3O_4 NPs [24]. Thanks to the unique properties of Co_3O_4 NPs which have been taken advantage of in environmental remediation applications, such as the degradation of dyes, dye waste, and antibiotics, including the photocatalytic degradation of hazardous dye waste in wastewater using Co_3O_4 NPs.

Recently publications related to Co_3O_4 NPs focused on the better capacity performance, recommended for energy storage and batteries. Mahmood et al. investigated Co_3O_4 NPs as anodes in Li-ion batteries, due to their capacity about 890 mAg^{-1} much higher than graphite 370 Ag^{-1}. large volume changes during repeated lithiation and delithiation processes is one of the countable problems of Co_3O_4 NPs as an anode material [9].

5. Conclusion

The synthesis of Co_3O_4 NPs using SCS method is one of the most revolutionary advancements in the synthesis of nanomaterials. The SCS method is preferred due to the high speed of the synthesis and high reaction rates to synthesize highly crystalline desired nanoparticles with related morphology and structure. The findings from recent studies highlight the critical influence of synthesis parameters, such as fuel-to-oxidizer ratios and precursor selection, on the resultant nanoparticles' porosity, surface area, and crystallinity. Furthermore, Co_3O_4 NPs exhibit a unique combination of properties, including high density, magnetic nature, and responsiveness to varying chemical environments, making them suitable for diverse applications. It has certain promises in energy storage applications including lithium ion batteries and supercapacitors, as well as in water treatment and organic pollutant removal.

Future work shall therefore focus on opening up other opportunities especially in relation to environmental uses of Co_3O_4 NPs. This mini-review also focuses on not only the evolution of these nanoparticles but also the scarcity of information regarding their application in novel environmental technologies. Further studies on the SCS method and the physical and chemical characteristics of Co_3O_4 NPs may open new doors to potential uses hence regional development in material science, nanotechnology and key sustainable development goals. Interestingly, when young scientists embrace this field, the future is bright to alleviate global challenges leveraging innovative nanomaterials.

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Синтез наночастиц Co_3O_4 методом синтеза горения раствора (СГС) и морфология их структуры: мини-обзор

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АННОТАЦИЯ

В работе представлен краткий обзор исследований, в которых рассматривается получение наночастиц оксида кобальта Co_3O_4 методом синтеза горения раствора, представляющим собой процесс, характеризующийся высокой скоростью реакции и производительностью процесса. Уникальными особенностями наночастиц оксида кобальта (II)(III) Co_3O_4 являются размер от 12 до 60 нм, а также высокая площадь поверхности и пористость, что повышает их эффективность использования в областях хранения энергии, экологии и сенсорных приложениях. Анализ различных параметров синтеза, таких как соотношение топлива и окислителя, материалы-предшественники и термические условия, позволяет определить их влияние на характеристики и функциональность частиц. Предлагаемый обзор подтверждает эффективность применения наночастиц Co_3O_4 в энергетических приложениях, однако проведение дальнейших исследований, напраленных на их экологическое применение, остается актуальным. В статье предлагаются дополнительные возможности использования наночастиц Co_3O_4 для решения некоторых актуальных экологических проблем.

Ключевые слова: наночастицы Co_3O_4, метод SCS, пористость, чувствительные сенсоры, хранение энергии

Ерітіндінің жану синтезі (SCS) әдісі арқылы Co_3O_4 нанобөлшектерінің синтезі және олардың құрылымының морфологиясы: шағын шолу

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АҢДАТПА

Бұл шағын шолудың мақсаты - реакцияның жоғары жылдамдығымен және төмен құнымен сипатталатын процесс болып табылатын ерітіндінің жану синтезі арқылы дайындалған кобальт оксиді Co_3O_4 нанобөлшектері туралы білімнің ағымдағы жағдайын көрсету. Кобальт (II) (III) оксидінің Co_3O_4 нанобөлшектерінің бірегей ерекшеліктері 12-ден 60 нм-ге дейінгі өлшемдер, сонымен қатар энергияны сақтау, экология және сенсорлық қолданбалар сияқты салаларда олардың тиімділігін арттыратын жоғары бетінің ауданы мен кеуектілігі болып табылады. Отынның тотықтырғышқа қатынасы, прекурсорлық материалдар және жылулық жағдайлар сияқты әртүрлі синтез параметрлерін талдау арқылы бұл шолу бұл факторлардың бөлшектердің сипаттамалары мен функционалдығына қалай әсер ететінін зерттейді. Бұдан басқа, ауқымды зерттеулер Co_3O_4 нанобөлшектерінің энергияны қолданудағы тиімділігін атап көрсеткенімен, олардың қоршаған ортада қолданылуына назар аударатын әрі қарай зерттеулерге және жас ғалымдарды нанотехнологиялар мен материалтанудағы инновациялық зерттеу бағыттарын іздеу арқылы жетілдірілген шешімге шақыру қажет. Шолу кейбір өзекті жаһандық экологиялық мәселелерді шешу үшін Co_3O_4 нанобөлшектерін пайдаланудың қосымша мүмкіндіктерін ұсынады.

Түйін сөздер: Co_3O_4 нанобөлшектері, SCS әдісі, кеуектілік, сезімтал сенсорлар, энергияны сақтау