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Green Simple Synthesis and Characterization of Cerium Nanostructures: Investigation of the Extract Effect on the Nanostructures Properties

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ABSTRACT

Metal oxides based on cerium structures have become the focus on new functional materials due to their good electrical, optical and other properties. In the present study, a simple method based on green chemistry has used for the synthesis of cerium nanostructures. For achieving the purpose, barberry extract has applied for the synthesis of the nanostructures. Then, various techniques were used to characterize the synthesized nanostructures. Therefore, at first Fourier transform infrared (FT-IR) spectroscopy was used for initial characterization. Then, the structural characterization was accomplished by X-ray diffraction spectroscopy (XRD) technique. Also, morphologic studies was investigated by field emission scanning electron microscopy (FE-SEM). In following the effect of barberry extract on the nanostructures properties was accomplished using electrochemical experiments. The data showed the presence of extracts in the synthesis route can be create an enhanced properties in the synthesized nanostructures.

INTRODUCTION

Recently, nanotechnology began to expand rapidly and entered many fields such as electronics, materials science, chemistry, life sciences, mechanics, and optoelectronics [1-3]. By focusing on reducing dimensions, it is possible to create almost controlled and desired changes in materials. Nanostructured compounds have a high surface area to volume ratio, which is an important feature of them, which has caused their surface behavior to overcome bulk effect. Therefore, the laws governing ordinary materials are not applicable as a result, quantum laws are used to study the behavior of nanomaterials [4]. Nanostructures were used in the manufacture of electrodes to improve the electrochemical signal due to their high surface area to volume ratio. The increase in surface area to volume ratio that occurs gradually with decreasing particle size causes the behavior of atoms located on the particle surface to prevail over that of internal atoms [5]. This phenomenon affects the properties of the particle in isolation and its interactions with other materials and greatly increases their reactivity. For example, in the case of metal nanoparticles, they are rapidly oxidized upon exposure to air. A large surface area is a key factor in the functioning of catalysts and electrode structures. This property can be used to effectively improve the efficiency of chemical catalysts or in the production of nanocomposites using these particles, stronger chemical bonds are established between the substrate and the particles and their strength is greatly increased [6].

Different types of nanoparticles with different sizes and compositions can play different roles in electrochemical sensors such as enzyme sensors, safety sensors, and biological sensors [7]. Among them, metal nanoparticles such as cerium nanostructures with the best conductivity and catalytic properties are the best choice as electronic connectors to enhance electron transfer between the redox center and the electrode surfaces and also as catalysts to enhance electrochemical reactions [8]. The use of nanoparticles as electrode modifiers gives remarkable features to the electrode, including effective catalysis, rapid mass transfer, improved signal-to-noise ratio,

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increased sensitivity, facilitated and accelerated electron transfer kinetics, reduced overpotential, and increased electrode surface area [9].

Many methods, including the sol-gel process, plasma method, mechanical alloying or high-energy ball impact, neutral vapor condensation and electrochemical methods are common methods for producing nanoparticles [10]. Although all the methods mentioned are used to produce large volumes of nanomaterials, the sol-gel method has a higher popularity and industrial application than other existing methods. The sol-gel method can produce high-quality nanoparticles (producing particles of the same size) in a large volume. Sol-gel is one of the types of bottom-up synthesis methods that is used to produce homogeneous metal oxides with high purity of ceramic particles. This method is one of the common methods for producing nanostructures in the liquid phase due to the ease and variety of products formed and the lack of the need for special equipment. Sol is derived from the word solution, which means a solution, meaning that a colloidal mixture contains solid particles suspended in a liquid phase. A colloid is actually a suspended mixture containing a phase distributed in a very small proportion, in which case the gravitational forces, which are weak and lead to the random movement of particles in the solution, can be ignored [11,12]. Generally, gel is one of the reaction products in the hydrolysis process of sol and has a continuous structure of organometallic molecules in an elastic state. The sol-gel method is classified into two categories based on the nature of the precursors, including inorganic precursors (nitrate, chloride, and sulfide) and alkoxide precursors [13,14]. On the other hand, by mixing the synthesis precursors of two or more different metals or metal oxides in certain proportions, we will be able to synthesize alloy products in one step. Also, there are other methods can do this, such as the plasma method, electrochemical methods, and phase condensation. Still, it should be noted that none of them can compete with the sol-gel method on an industrial scale. The sol-gel method can also provide the possibility of manufacturing very homogeneous and very high-purity composites with 99.99% purity. This method is also capable of producing ceramic and metallic nanomaterials at much lower temperatures compared to the common methods that have a very high temperature range [15,16].

In the present work, metal nanostructures based on cerium and barium were synthesized by a simple sol-gel method. Also, an environmentally-friendly route was selected based on barberry extract. Then, for characterization of the nanostructures different techniques were used. Also, the effect of barberry extract on the synthesis route was investigated. The data were showed the enhanced properties can be achieved for the nanostructures in the presence of barberry extract.

Experimental

Chemicals and instruments

The reagents that were used in the present work containing Ce(SO4)$_2$.4H$_2$0 (99.99%), Ba(N0$_3$)$_2$ (99.999%), and others were purchased from Merck Company. The chemicals were applied as received. Deionized water was used for preparation of all solution.

Characterization of the nanostructures was performed using a Fourier transform infrared (FT-IR) Shimadzu Varian 4300. Also, -ray diffraction spectroscopy (XRD) technique (Cu Kα (λ= 1.54 Å)) was used for structural characterization. Next, for investigation of the morphological properties of the nanostructures field emission scanning electron microscopy (FE-SEM, MIRA3TESCAN-XMU) was applied. Also, a potentiostat/galvanostat SAMA 500 (Iran) was used for electrochemical studies. Electrochemical experiments were performed at room temperature using a conventional three-electrode system. A bare or the modified carbon paste electrode based on cerium nanostructures, a platinum, and an Ag/AgCl (3 M KCl) were used as working, contour, and reference electrodes, respectively.

Synthesis of the Nanostructures

In order to synthesize cerium nanostructures using the sol-gel method, cerium sulfate and barium nitrate were first reacted and barberry extract is used as a fuel source. The stoichiometric ratios of $Ce(SO_4)_2.4H_2O$ and $Ba(NO_3)_2$ salts are used similarly for all optimization experiments and other parameters affecting the synthesis method including the amount of barberry extract, pH, temperature and calcination time were optimized. The obtained gel was placed in an oven for 24 hours to dry and then calcined in a furnace at 900°C for 4 hours.

RESULTS AND DISCUSSION

FT-IR Analysis

Fig. 1 shows the FT-IR spectrum of the cerium nanostructures that were synthesized in the presence of barberry extract. This spectrum is used to identify the functional groups. The bands about 3400 cm^{-1} and 1600 cm^{-1} are attributed to the bending and stretching vibrations of the O-H groups of water molecules [17]. A strong band around 1100 cm^{-1} indicates the C-O-C stretching vibrations obtained from the synthesis using the plant extract [18]. It is clear that the absorption peaks at about 525 cm^{-1} and 610 cm^{-1} are related to the characteristic metal-oxygen stretching modes of the compound [19].

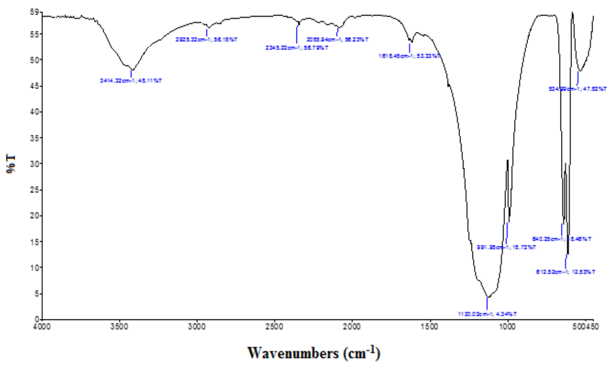

Fig. 1. FT-IR spectrum of the cerium nanostructures that were synthesized in the presence of barberry extract.

XRD studies

Fig. 2 shows the XRD pattern of cerium nanostructures. This study shows that the nanostructures are formed without impurities in the presence of barberry extract. In the present study, a natural compound containing barberry extract is used to reduce environmental pollution in the synthesis of cerium nanostructures. Also, the crystal size of cerium nanostructures that were synthesized in the presence of barberry extract was obtained to be about 38 nm using XRD analysis and the Scherrer equation. It should be noted that natural materials act as both reducing agents and fuels in the synthesis route [20,21].

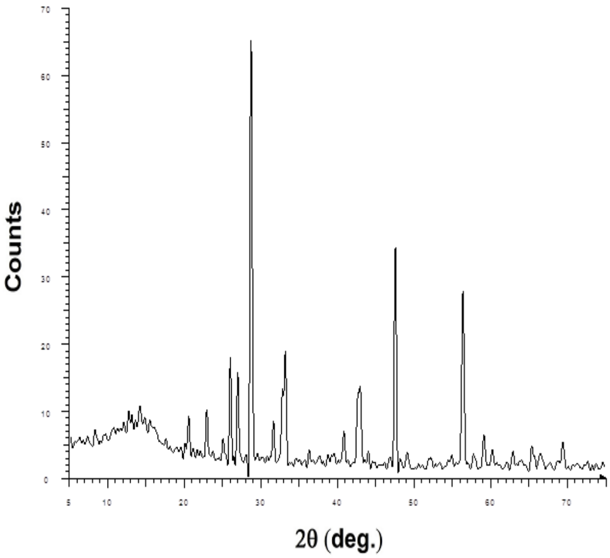

Fig. 2. XRD pattern of the cerium nanostructures that were synthesized in the presence of barberry extract.

FE-SEM studies

The improvement and control of surface morphology in the sol-gel method is carried out using a certain ratio of fuel [22]. The effects of fuel to oxidant ratio are a fundamental approach in the synthesis of microstructures and provide control over size, morphology, and stable preparation. In this study, plant extracts containing large amounts of carbohydrates and proteins were used as a natural fuel. These compounds can play the role of reducing and capping agents in the synthesis of nanostructures [23]. The functional groups in these structures have the ability to form complexes with metal ions and can be used as a suitable and new template for nucleation and guidance of structures. To study the role of barberry extract on the morphology of cerium nanostructures, the FE-SEM image of the synthetic sample in the presence of the extract is shown in Fig. 3. As mentioned, barberry extract, as a rich source of acids and various organic compounds, can be a suitable alternative as an acidic fuel source in the sol-gel process. As can be seen in Fig. 3, cerium nanostructures were separated from each other in the presence of barberry extract as a reducing and capping agent and, by creating nanometric structures.

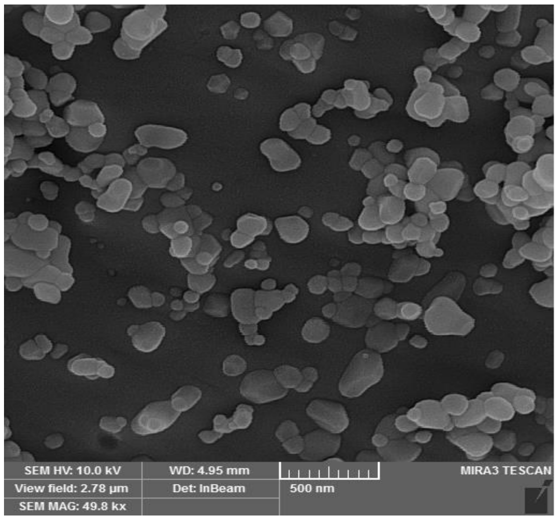

Fig. 3. FE-SEM micrograph of the cerium nanostructures that were synthesized in the presence of barberry extract.

Electrochemical studies

To prove the effect of barberry extract and performance of the synthesized nanostructures in electrochemical studies, the cyclic voltammetry (CV) spectrum of the synthesized cerium nanostructures in the absence and presence of barberry extract in the potassium hexacyanoferrate II and III probe solution was recorded. Fig. 4 (a) shows the cyclic voltammogram of the synthesized cerium structures in the absence of barberry extract, and Fig. 4 (b) shows the cyclic voltammogram of the synthesized cerium structures in the presence of barberry extract. As can be seen, in the presence of barberry extract, the peak oxidation-reduction potential of the sample decreased and the peak oxidation-reduction current increased significantly. This indicates that the synthesized cerium nanostructures with high electrochemical performance in the presence of barberry extract were synthesized. Therefore, cerium nanostructures have the ability to facilitate electron transfer and can be used as the suitable electrocatalysts in various fields such as hydrogen storage studies, electrochemical supercapacitors, and electrochemical sensors.

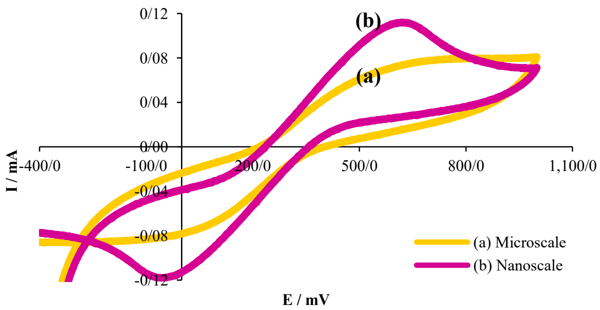

Fig. 4. Cyclic voltammograms of the synthesized cerium nanostructures in the (a) absence of barberry extract and (b) presence of barberry extract

CONCLUSION

In this research, the green synthesis of the nanostructured compounds, considering the economic efficiency and safety of the proposed method, encouraged us to use this method. In order to synthesize of cerium nanostructures using the sol-gel method, first barium and cerium salts were reacted and barberry extract was used as a fuel source. Then, the identification and study of the nanostructures were carried out by different techniques. The characterization techniques confirmed the formation of cerium nanostructures. Also, the effect of barberry extract was investigated. The results showed the samples that were synthesized in the presence of barberry extract are in nanometric scales and with the enhanced electrochemical properties. Therefore, the proposed nanostructures can be act as the potential electrocatalysts in different fields.

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