



Synthesis and Characterization of Nano-Strontium Oxide (SrO) Using Erzincan Cimin Grape (*Vitis vinifera*, Cimin)

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Authors' contributions

This work was carried out in collaboration among all authors. Author AAG designed the study, performed the analysis, wrote the protocol and wrote the first draft of the manuscript. Authors HN and DDG managed the analyses of the study and the literature searches. All authors read and approved the final manuscript.

Article Information

DOI: 10.9734/CSJI/2019/v26i330092

Editor(s):

(1) Dr. Francisco Marquez-Linares, Department of Chemistry, Nanomaterials Research Group, School of Science and Technology, University of Turabo, USA.

(2) Prof. Nagatoshi Nishiwaki, School of Environmental Science and Engineering, Kochi University of Technology, Tosayamada, Kami, Kochi 782-8502, Japan.

Reviewers:

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(4) Senem Akkoç, Suleyman Demirel University, Turkey.

Complete Peer review History: <http://www.sdiarticle3.com/review-history/47662>

Original Research Article

Received 09 January 2019

Accepted 21 March 2019

Published 28 March 2019

ABSTRACT

Aims: Synthesis of Strontium oxide (SrO) is very remarkable for its applications in electrodes for gas sensors, lithium-ion batteries, solar cells, doped dye-sensitive solar cells, transistors, catalyst supports, super capacitors and semiconductors. This study aims to synthesize SrO nanoparticles using a new method, as they will find important and specific uses.

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Experimental Design: Synthesis of SrO nanoparticles (NPs) was planned to be obtained by bio-reduction method using the plant extract of Erzincan Cimin grape (*Vitis vinifera*, Cimin).

Place and Duration of Study: The experimental steps of the study were carried out in the laboratories of Atatürk University, Erzurum Vocational College, Department of Chemical and Chemical Processing Technologies.

Methodology: In this study, the synthesis of SrO NPs was made by using aqueous extract of Erzincan Cimin grape (*Vitis vinifera*, Cimin). Biosynthesis medium containing 0.1 M $\text{Sr}(\text{NO}_3)_2$ was added to Cimin grape extract to synthesize SrO NPs.

Results: The structural characterization of the SrO NPs that were obtained as a result of the reaction was analyzed using SEM, EDAX and FTIR techniques. Using the X-ray diffraction pattern in the study, the mean particle size was calculated to be 28.6 nm. SEM analysis revealed that particles having a porous nanostructure in 20-50 nm of size were synthesized. It was determined that the maximum absorbance of SrO nanoparticles was at 203 nm. From the experimental data, it was determined that for SrO nanoparticles, the optimum synthesis rate was reached at 90 minutes, 40°C and pH 8.

Conclusion: The newly synthesized nanomaterial is expected to find applications in many different areas thanks to its physical and chemical properties.

Keywords: Erzincan Cimin Grape (*Vitis vinifera*, Cimin); strontium oxide NP; green synthesis.

1. INTRODUCTION

The developments in the synthesis approaches of nanomaterials are distinguished by the acquisition of new chemical and physical properties. Today, with the use of green synthesis reactions, It has gained a significant speed in the synthesis of many metal oxide nanoparticles. The synthesis of nanoparticles from oxides of transition metals such as strontium oxide (SrO) is significant due to the structural diversity and wide range of applications. For a long time, SrO NPs have been investigated for their promising applications in manufacture of gas sensors, lithium-ion batteries, solar cells, doped dye-sensitive solar cells, transistors, catalyst supports, supercapacitors and electrodes for semiconductors [1-5]. Some studies reported the sol-gel synthesis method of SrO NPs and investigated the optical and thermal properties of nanoparticles. In another study, Nemade and Waghuley reported that they produced strontium oxide nanoparticles using a one-step chemical precipitation method [6]. In all known synthetic methods, extreme conditions (high temperature, organic solvents, high-precision precursors, toxic reducing agents, and special atmospheric media) are used and there are many disadvantages such as requiring multiple steps to perform an experiment. In addition, many of the chemical reducing agents and solvents used in these methods carry a toxicological risk and are harmful to the environment. There is very little literature on the synthesis of SrO NPs in currently used methods, and these methods have their own limitations.

To overcome this disadvantage, many researchers nowadays use various plant (or herbal) extract, such as Pomegranate (*Punica granatum*), Basil (*Ocimum sanctum*), Papaya (*Carica papaya*) and Cabbage (*Brassica oleracea*) to synthesize nanoparticles. A great interest has been shown for the development of novel and simple approaches to nanoparticle synthesis that use plant compounds as reducing agents. Diverse morphology and nanoscale size have a crucial impact on improved physical and electrical properties of synthesized nanoparticles. In addition, under moderate conditions, green chemical synthesis pathway for the preparation of SrO NPs has distinguished with a moderate approach such as room temperature and neutral pH. Researchers have begun to find and develop new, economic and environmentally friendly methods for reasons such as environmental pollution factors and cost increase in the synthesis of nanoparticles.

For this purpose, Erzincan Cimin grapes (*Vitis vinifera*, Cimin) were used as reducing agents in the synthesis of SrO NPs. It is a variety of black grape grown in Erzincan province, Turkey. In the past, the grape and its products were used among the public for treatment of diseases. Today, Erzincan Cimin grapes are known to be used in food and health sector [7]. It is considered that grape extract may be used as a reducing and stabilizing agent in green nanosynthesis thanks to the presence of important components such as sugar derivatives, alkaloids and terpenoids [8,9]. Investigations showed that there are no other research that uses Erzincan Cimin grape in synthesis of SrO

NP. Therefore, SrO NPs were aimed to be synthesized and characterized in more efficient, economical and moderate conditions using Cimin grape.

2. MATERIALS AND METHODS

2.1 Chemical and Reagents

An endemic species of Cimin grape (*Vitisvinifera* cv. Cimin) was obtained from Erzincan province in September 2017. $\text{Sr}(\text{NO}_3)_2$ is 99.995% pure and other chemicals (Sodium acetate (NaCH_3COO), Tris ($(\text{HOCH}_2)_3\text{CNH}_2$), Sodium carbonate (Na_2CO_3), Sodium hydroxide (NaOH), Hydrochloric acid (HCl)) are analytical purity from Sigma-Aldrich GmbH (Sternhe I Germany). All solutions were prepared using deionized water. All the materials to be included in the experiments were disinfected in the oven and sterilized to ensure a clean environment.

2.2 Preparation of the Reaction Medium

The Cimin grapes (*Vitisvinifera* cv. Cimin) obtained from Erzincan were washed with deionized water. They were then rinsed, dried and stored in a freezer at -20°C until they were employed. An aqueous extract of grapes was prepared, and used in the green synthesis reaction medium. In a simple procedure, 100 grams of Cimin grape (*Vitisvinifera* cv. Cimin) was blended with 250 mL of deionized water for 30 minutes at 300 rpm until a homogeneous mixture was obtained. The homogenate was then filtered through a filter paper, then the filtrate was centrifuged at $3000 \times g$ to remove heterogeneous moieties. The resulting Cimin grape (*Vitisvinifera* cv. Cimin) aqueous extract was used as the reaction medium for synthesis [10,11].

2.3 Green Synthesis of SrO NPs

The prepared 50 mL grape extract was added carefully to the solution prepared with the water-soluble salt of 0.1 M Sr metal (500 mL) (Fig. 1). The green synthesis reaction was monitored by UV-Vis spectrophotometry for 72 hours under normal atmospheric pressure and room conditions under stirring at 300 rpm. The conversion of the Sr^{+2} ion to the SrO NPs in the reaction medium was monitored at 200-1000 nm using spectrophotometry and the absorbance increase at the 203 nm wavelength was observed with the formation of the SrO NPs [6]. This determined wavelength was used in the

next optimization process. The effects of time, pH, temperature and metal ion concentration parameters on nanoparticle synthesis were determined separately for optimization [6].

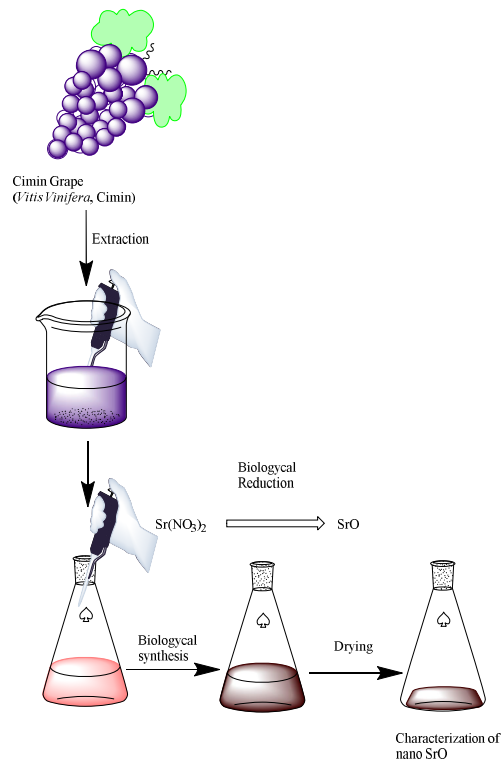


Fig. 1. Synthesis of Nano Strontium Oxide (SrO) from Erzincan Cimin Grape (*Vitis vinifera*, Cimin)

2.3.1 Interaction time

From the reaction medium prepared with the solution of $\text{Sr}(\text{NO}_3)_3$, the samples were taken at 3-minute intervals and the SrO formation was measured by the absorbance changes at 203 nm and the reaction was monitored with the spectrophotometry during a total reaction time.

2.3.2 Optimum pH

The most efficient pH for the synthesis of SrO NPs was investigated by using different buffer solutions. For this purpose, synthesis reaction media of SrO NPs were prepared using phosphate buffer for reaction media of pH: 2-3, pH: 4-6, and pH 7-8, and carbonate buffer for pH: 9-11. The formation of SrO NPs and the changes in absorbance at 203 nm were monitored through UV-Vis spectrophotometry.

2.3.3 Optimum temperature

In order to determine the temperature at which the SrO NPs were more synthesized, the reactions were carried out separately at temperatures between 10-90°C (at intervals of $10 \pm 2^\circ\text{C}$). All measurements were made to keep the pH and metal ion concentration constant so that the only variable was the temperature of the reaction medium.

2.3.4 Optimum metal ion concentration

Different concentrations of solutions (0.05 M, 0.1M, 3M, 5M and 7M) were prepared for the synthesis of SrO NPs. All measurements were made to keep the pH and temperature constant so that the only variable was the metal ion concentrations of the reaction medium. The effect of metal ion concentration on the nanoparticle synthesis reaction was determined spectrophotometrically (at 203 nm).

After all conditions were optimized, the obtained SrO NPs were collected, dried and then characterized.

2.4 Characterization of SrO NPs

The synthesized SrO nanoparticles were characterized by a UV-VIS spectrophotometry (Epoch Nanodrop UV-VIS spectrophotometer), scanning in the 200-1000 nm range. The topographic analysis of SrO nanoparticles was performed by SEM (Scanning Electron Microscopy analysis). The ratios of structural components of SrO NPs were determined by Energy Dispersive X-Ray Analysis (EDAX) and Fourier Transform Infrared spectrophotometry (FTIR). So, the interactions between the organic phase and SrO NPs were determined and structural characterization of the synthesized NP were made [12,13].

3. RESULTS AND DISCUSSION

Strontium is a compound having thermal, electrical and electro catalytic activity. It is also used in dentistry and dental applications [1]. Strontium (Sr) has been reported to be one of the important trace elements that strengthen bones in the human body and prevent caries [14]. In previous studies for the synthesis of SrO NPs, various chemical and physical methods have been employed, such as sol-gel, co-precipitation, hydrothermal and spray pyrolysis. Conventional hydrothermal methods have also been used for

the synthesis of SrO nanoparticles. In particular, by means of microwave hydrothermal synthesis, high temperature heat treatment which can lead to particle growth and agglomeration can be avoided. However, the use of large amounts of strontium precursors and low yield are two major disadvantages of the current methods [15]. In another study, it has been reported that Strontium oxide (SrO) NPs are synthesized by hydrolysis of single source molecular precursor $\text{SrCl}_2 \cdot 6\text{H}_2\text{O}$ with potassium hydroxide whereafter the structure, morphology and properties of the particles were characterized using XRD, TEM, Raman and UV-VIS spectroscopic techniques [15].

In this study, using Cimin grape extract, SrO NPs were synthesized in high efficiency and moderate conditions. The size and purity of the nanoparticle were improved by optimizing the synthesis conditions (pH, temperature, reaction time, etc.). SrO nanoparticles showed maximum absorbance at 203 nm as a result of the screening (Fig. 2). The synthesized SrO nanoparticles showed a maximum absorption at 203 nm as a result of the screening (Fig. 2). So, the reactions were followed at 203 nm using an UV spectrophotometry. At the end of the experiments, for optimization of the method, it was determined that the SrO NPs at 0.1 M metal ion concentration, 40°C , pH 8, reached the maximum synthesis rate in 90 minutes.

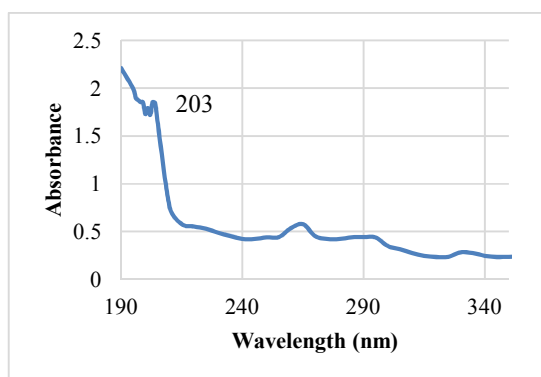


Fig. 2. UV-vis spectrum of synthesized SrO NPs

After optimizing the reaction, the characterization of the obtained nanoparticles was performed. Fig. 3 shows the SEM image of the SrO NPs obtained from the SEM analysis. It was observed that the SrO NPs consisted of small spheres with a diameter of $20-50 \pm 2.5$ nm and arranged as channels.

In Fig. 4 is EDAX analysis of SrO nanoparticles. Based on data from EDAX analysis and Scanning Electron Microscope (SEM) results support the formation of SrO nanoparticles.

Fourier Transform Infrared (FT-IR) spectrophotometer was used to obtain information about the binding of SrO NPs with the organic phase. In detail, the absorption bands at 3664 cm^{-1} originates from stretching vibration and deformation vibration of O-H. The strong broad absorption peak at about 1473 cm^{-1} is attributed to the asymmetric stretching

vibration of Sr-O, the sharp absorption bands at 956 cm^{-1} and 885 cm^{-1} can be attributed to out-of-plane bending vibration of Sr-O. The two bands at 1255 cm^{-1} are attributed to the C-O vibrations. [16-18]. The sharp absorption bands at 1083 cm^{-1} , 1317 cm^{-1} , 1473 cm^{-1} , 1610 cm^{-1} , 2979 and 3276 cm^{-1} can be attributed to bending vibration of O-H and stretching vibrations of C-O [15]. The presence of C-O, O-H, C-C, and C-N peaks indicates that all SrO NPs are formed using the Cimin grape extract as reducing agent and also Cimin grape is acting as capping agent on the surface of metal oxide nanoparticles (Fig. 5).

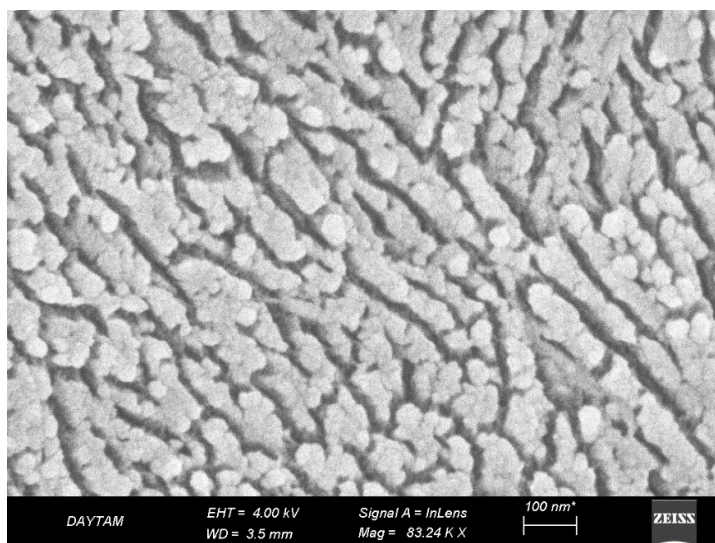


Fig. 3. SEM image of SrO NPs

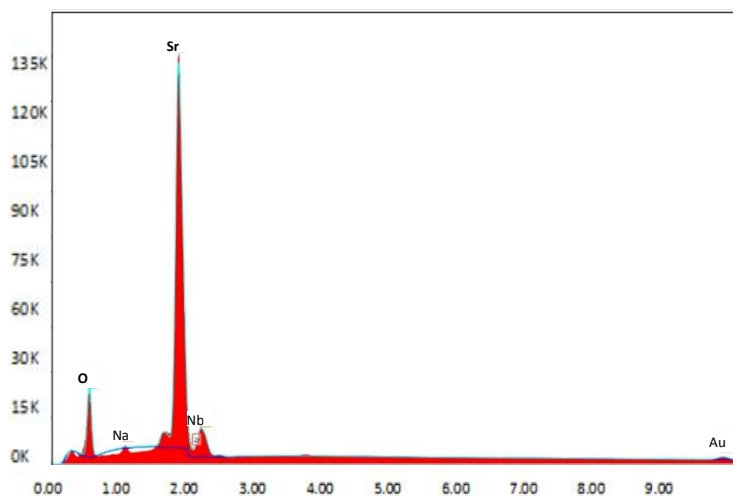


Fig. 4. EDAX analysis result

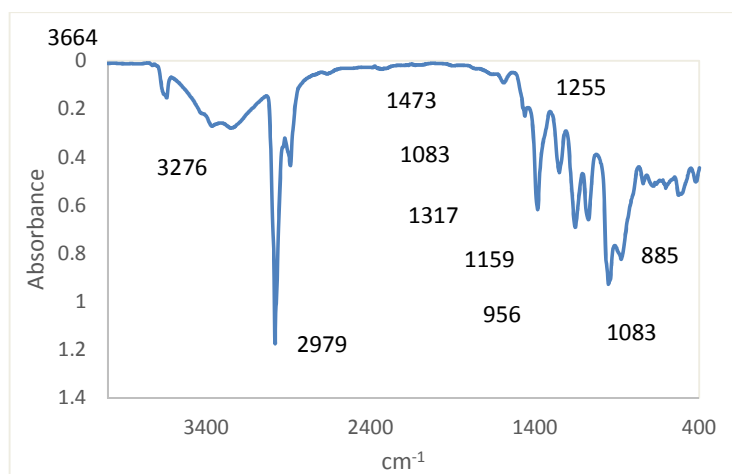


Fig. 5. FTIR spectrum of SrO nanoparticles

4. CONCLUSION

So far, various chemical methods are known for synthesis of SrO NPs, such as sol-gel, co-precipitation, hydrothermal and spray pyrolysis. Today, due to cost-reducing and environmentally friendly approaches, the use of green chemical reactions has gained significant importance in the synthesis of NPs from a large number of metal oxides. For this reason, the synthesis of SrO nanoparticles having a widespread use is an important and interesting research topic. According to the results obtained from SEM, XRD and FTIR analyses, nano-sized strontium oxide particles can be synthesized by the method used in this study. It is concluded that these synthesized nanoparticles can be used because of the advantages they gained with this production approach in many industrial areas.

COMPETING INTERESTS

Authors have declared that no competing interests exist.

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