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Sahar N. Rashid [1,*]
Ishraq S. Jaafer [1]
Maha A. Shaker [1]
Sumaya Z. Khalaf [2]

[1] Department of Physics, College of Science, University of Tikrit, Tikrit, IRAQ
[2] Department of English, College of Arts, University of Tikrit, Tikrit, IRAQ
* Corresponding author email: sahar83@tu.edu.iq

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Characterization of Cobalt Nanoparticles Synthesized by Two Sustainable Approaches

This work focused on two nanosynthesis methods due to their importance, as they are environmentally friendly, especially in the basics of manufacturing and characterization. Cobalt nanoparticles were synthesized by these two methods and characterized optically and structurally. The results showed that the cobalt nanoparticles synthesized by the two methods had a polycrystalline hexagonal phase, and their average crystal size was 20.52 nm and 33.54 nm when synthesized physically and biologically, respectively. They had more regular shapes when synthesized physically, but with a higher concentration than those synthesized biologically. This work presents a simple comparison of the similarity, uniqueness, and complementarity of two sustainable synthesis processes, with a proposal for their development to produce a low-cost and sustainable product in the future.

Keywords: Bottom-up synthesis; Laser ablation; Cobalt nanoparticles; Sustainable synthesis

1. Introduction

Nanomaterials are characterized by their wide-ranging applications across various sectors, such as industry [1-3], agriculture [4,5], and medicine [6,7]. This versatility stems from the ability of nanoparticles (NPs) to significantly enhance the properties of materials. These enhancements may be thermal, mechanical, or otherwise [8]. Furthermore, the precise control over the composition and structure of many NPs allows for a virtually limitless range of applications and technological advancements [1]. Undoubtedly, the size, shape, and structure of NPs have a significant impact on their physical properties [9,10]. Methods for producing metal NPs can be broadly classified into two main categories: top-down and bottom-up approaches. The key difference between these two approaches lies in the starting materials used. Top-down methods utilize bulk materials as starting materials, which are then transformed into NPs using various physical processes [11]. In contrast, bottom-up methods use atoms or molecules as starting materials [12]. Figure (1) shows the difference between the two approaches [13].

Pure and reactive NPs can be produced using pulsed laser ablation in liquid (PLAL), a top-down technique [14,15]. Laser ablation of solid targets in a liquid medium generates NPs with valuable properties, such as metastable structures, simple or complex structures (including doped nanocrystals or core-shells), and is easy to use [16]. At the same time, this technique is environmentally sustainable. It does not require specific conditions [17,18]. Figure (2) illustrates the process of pulsed laser ablation in liquid [19]. Research on laser-fabricated NPs in liquid environments focuses on characterizing NPs, particularly their size and shape. Size and shape directly influence their properties, such as catalytic performance and surface activity. Understanding how to control the shape and size of NPs will be a major focus of future research [14]. The mechanism of nanoparticle synthesis during the PLAL process can be summarized as the interaction of the surface particles of the solid material with the laser beam, which leads to their removal to form a colloidal solution of this material, which represents the liquid medium containing the nanoparticles of the target solid material [20-22].

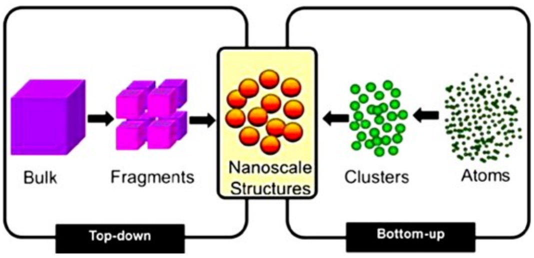

Fig. (1) Top-down and bottom-up synthesis of nanofabrication [13]

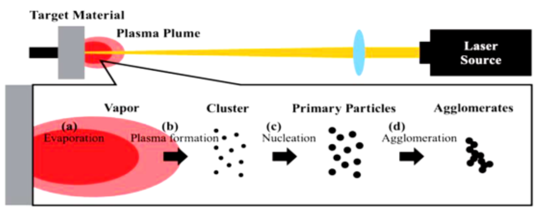

Fig. (2) Laser ablation process [19]

Biosynthesis (green synthesis), a bottom-up technique, of metal NPs has become a popular field in nanotechnology in recent years. It is also considered a sustainable and environmentally friendly method. The metal salts used as raw materials are reduced by plants containing some bioactive chemicals. Their reducing agents can be based on bacteria, fungi, yeast, and microorganisms [14,24]. Figure (3) illustrates the process of green synthesis [25]. The biological process of nanoparticle synthesis, specifically plant-mediated synthesis, involves the reduction of mineral salts used as raw materials in the manufacture of nanoparticles from metals by plants containing some biologically active chemicals. Flavonoids, ketones, amino acids, phenols, aldehydes, and vitamins, present in the chemical composition of plants, have the ability to reduce various metal ions [26-28]. These two promising methods can be used to synthesize NPs from various metals, including cobalt (Co), which has attracted the interest of researchers due to its diverse potential applications, for example, in medical sensors, plastics, coatings, nanowires, nanofibers, high-performance magnetic recording materials, as well as in military applications [29,30]. This work aims to synthesize cobalt nanoparticles (CoNPs) by two sustainable physical and biological methods: pulsed laser ablation in liquid and green method using plants, respectively, and then characterize the resulting NPs in an attempt to determine the significance for choosing the appropriate method for potential applications.

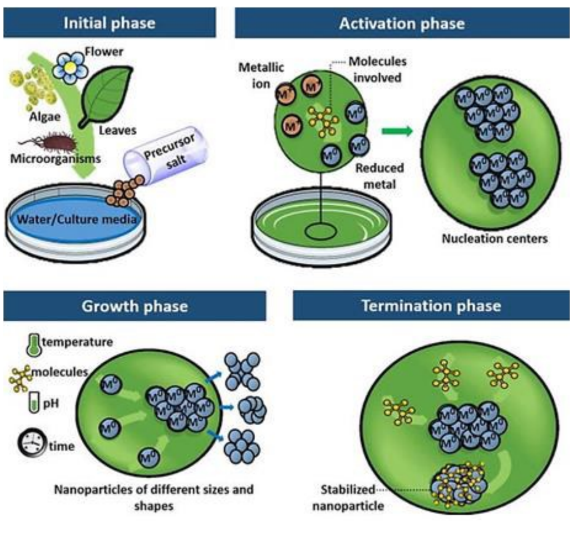

Fig. (3) Green synthesis of NPs [25]

2. Experimental Part

Cobalt nanoparticles (CoNPs) were synthesized using two methods: the first method involved pulsed laser ablation in a liquid medium, using a pure cobalt sheet of dimensions 1×1× 0.2cm immersed in 3 mL of deionized water, with a liquid level above the sample of 5 mm. The sample was irradiated with 500 pulses of Nd:YAG laser at a pulse energy of 100 mJ, a repetition rate of (5 Hz), a pulse duration of (9 ns), and a wavelength of (1064 nm). This process took less than (2 min). The second method is a green, biological approach using saffron (*Crocus sativus*) plant extract as a reducing agent for cobalt nitrate. This extract was prepared by dissolving (0.5 g) of *Crocus sativus* in (20 mL) of deionized water, heating it on a magnetic stirrer at a temperature between (70-80 °C) for (6 min), and then filtering the resulting solution. A (1 M) solution of cobalt nitrate was then prepared by dissolving (0.291 g) of aqueous cobalt nitrate in (50 mL) of deionized water, heating it on a magnetic stirrer at (50 °C) for (15 min). Finally, (5 mL) of the plant extract solution was added dropwise to the cobalt nitrate solution. Three colloidal solutions of CoNPs resulted: two prepared using a physical method and one using the biological method. The structural and optical properties of the NPs prepared in these solutions were determined using X-ray diffraction (XRD), transmission electron microscopy (TEM), and UV-visible spectrophotometer (UV-Vis) techniques.

3. Results and Discussion

The results of XRD of the two samples, which were synthesized by two sustainable methods, physical and biological, confirmed the formation of hexagonal CoNPs with polycrystalline phases with different degrees of crystallization and preferential orientations, as in Figs. (4) and (5), respectively. They also differ in their interatomic distances (d) and their average crystal size (D) calculated from Equation (1), which represents the Debye-Scherrer equation [31], where it equals (20.52 nm) for the NPs synthesized physically, while the average was greater for those synthesized biologically, where it equals (33.54 nm).

$$D = \frac{0.9\,\lambda}{\beta\cos(\theta)} \quad (1)$$

where λ represents the wavelength of the X-ray and β represents the full width at half maximum of the diffraction peaks. These differences are due to the nature and conditions of the two synthesis methods used in terms of the time taken, temperature, and raw materials used in the synthesis, as they affect the nucleation, crystallinity, and crystal sizes of NPs. Table (1) shows the data of the XRD pattern analysis of CoNPs synthesized by the two methods and the Miller coefficients (hkl) corresponding to the diffraction angles, which match JCPDS (card no. 96-901-2885).

Although the formation of agglomeration and clustering in Figs. (6) and (7), which represent the results of TEM for NPs synthesized by physical and biological methods, respectively, it was found that the NPs synthesized by the physical method (laser ablation) have more regular shapes, as they are spherical and semi-spherical, while those created by the biological method range between needle-shaped and elliptical. Their average size was (30 nm) and (48 nm) for those created by the physical and biological methods, respectively. The small dimensions of NPs, the strength of chemical bonds, and the strength of surface energy lead to the emergence of more clusters and agglomerations [32] that, which was observed in NPs synthesized by ablation compared to those created by biological methods. However, on the other hand, these clusters may lead to delayed effectiveness in potential biological applications, for example, when used as an alternative to antibiotics.

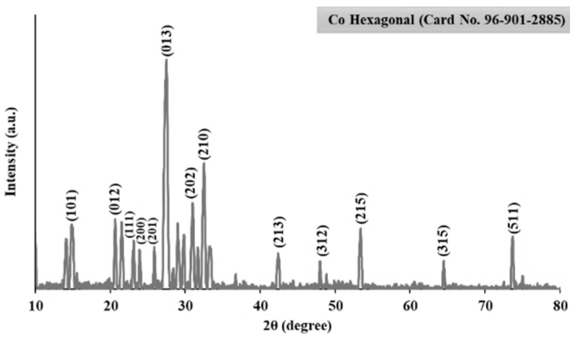

Fig. (4) XRD pattern of CoNPs synthesized by laser ablation

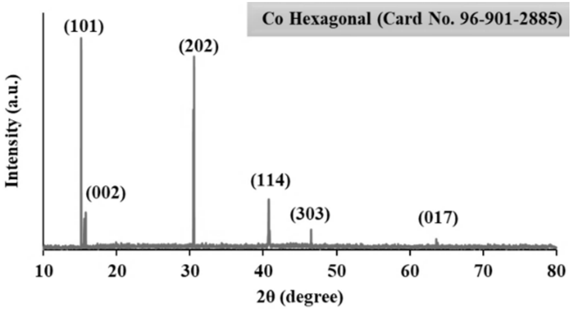

Fig. (5) XRD pattern of CoNPs synthesized by biological method

Table (1) XRD data of CoNPs synthesized by physical and biological methods

Method	2θ (deg)	hkl	β (deg)	d (nm)	D (nm)
Physical	14.8	101	0.6	0.597847	13.34649
	20.5	012	0.4	0.432722	20.17497
	23.2	111	0.8	0.382936	10.13347
	24	200	0.3	0.37035	27.06202
	26	201	0.3	0.342297	27.16694
	27.6	013	0.8	0.322806	10.22155
	31	202	0.4	0.288132	20.60228
	32.4	210	0.6	0.275994	13.78258
	42.4	213	0.5	0.212928	17.03527
	48	312	0.3	0.189312	28.97573
	53.4	215	0.4	0.17137	22.22255
	64.4	315	0.3	0.144499	31.28204
	73.6	511	0.4	0.128543	24.79357
Biological	15.7	101	0.1	0.563772	80.16316
	17.4	002	0.12	0.509054	66.946924
	30.45	202	0.1	0.29321	82.30053
	40.18	114	0.2	0.224166	42.27844
	46.5	303	0.1	0.195063	86.4309
	63.6	017	0.12	0.146122	77.86468

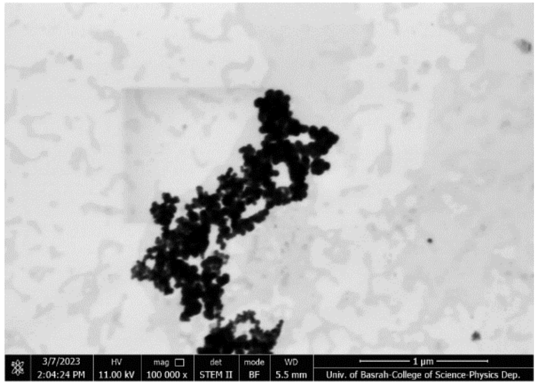

Fig. (6) TEM image of CoNPs synthesized by physical method

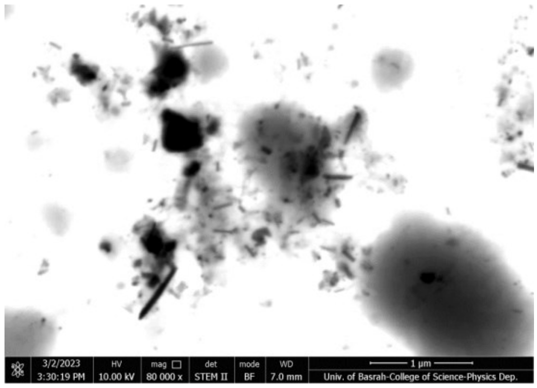

Fig. (7) TEM image of CoNPs synthesized by biological method

Figures (8) and (9) represent the absorbance and absorption coefficient spectra, respectively, resulting from UV-Vis analysis of CoNPs synthesized by the two methods. By comparing the absorbance spectra, it is noted that the absorbance of the biologically synthesized CoNPs is higher than that of the physically synthesized ones. This indicates the high concentration of NPs in their biologically prepared colloidal solution, which confirms their high effectiveness. On the other hand, it is noted that the physically synthesized CoNPs have a more regular absorption spectrum, which indicates the stability of these NPs in their colloidal solution. This result is consistent with what was obtained from the structural characterization, as the increase in the average size of the NPs and crystals of the biologically synthesized ones compared to the other method led to a shift in the absorbance peak towards the high wavelengths (red-shift). From Eq. (2) [33] through which the absorption coefficient (α) of CoNPs was calculated, which represented as a function of photon energy, it is clear that its values depend on the absorbance (A) values, and since it determines the light transmittance through a path (t) within the material before its absorption, the high concentration compared to the biologically created nanoparticles led to them having a higher absorption coefficient than those created physically.

$$\alpha = \frac{2.303\,A}{t} \qquad (2)$$

These results show that the superiority of the NPs properties produced by the two methods varies depending on the preparation conditions and materials. The work can be developed by changing the different preparation parameters and in-depth characterization with additional techniques to reach an ideal characterization.

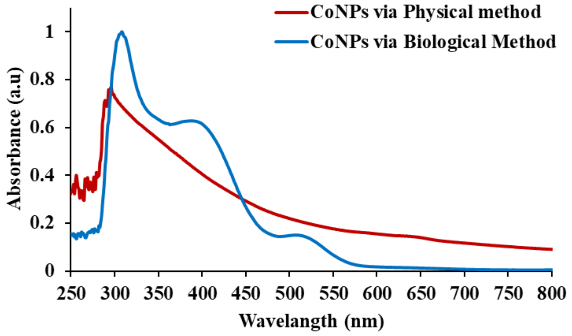

Fig. (7) Absorbance spectra of CoNPs synthesized by physical and biological methods

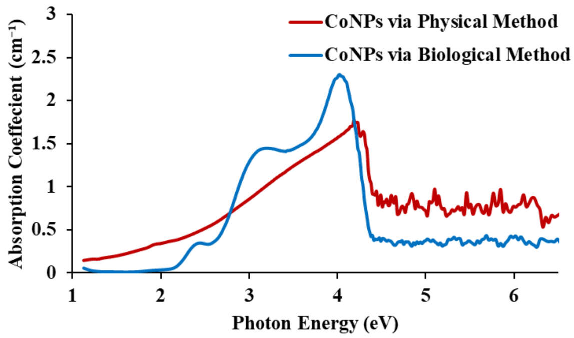

Fig. (8) Absorption coefficient of CoNPs synthesized by physical and biological methods

There is no doubt that there is a great diversity in the potential applications of the resulting NPs, each according to the specifications appropriate to the required application. In this context, researchers Mustafa and Taghried [1] presented a set of significance criteria that researchers must benefit from in choosing different nanosynthesis techniques according to the required application and taking into account environmental sustainability through a simplified description of the first letters of the word (significance) as follows: (s) selection of analysis by direct technique, (i) integration of operational and analytical processes, (g) generation of as little waste as possible, space and treatment, (n) never waste of energy as little as possible, (i) implementation of automation and simplification of methods, (f) favor of factors that can be obtained from a renewable source to preserve health and the environment, (i) increased safety of workers by choosing a simplified and safe technique, (c) conducting on-site measurements, (a) avoiding derivatives and accidental results that may be harmful, (n) note that the size and number of samples should be as low as possible, (c) choose of multi-analytical methods to suit different conditions and with multiple parameters, and (e) elimination or replacement of harmful reagents.

4. Conclusion

A comparative description of the laser ablation in liquid and plant-mediated synthesis methods, as well as the properties of the resulting nanoparticles, can be concluded, where by comparing the quantities of materials and equipment used between the two methods to synthesize cobalt nanoparticles, we find that the physical method is less expensive, with the exception of the ablation device used. It is worth noting that, at present, it is easy to obtain equipment with good specifications and operate it for long periods. Furthermore, the time and effort required to prepare samples using the physical method are reduced more than those obtained using the biological method. Through characterization of the resulting nanoparticles, the current work conditions produced a higher concentration of those synthesized by the biological method than those obtained by the physical method based on optical characterization. The structural characterization showed that the physically synthesized nanoparticles had more regular shapes and smaller size averages, but they had clusters and aggregates. This variation in characterization is useful in choosing the appropriate technique for the desired potential application.

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