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Study of the morphological characteristics of nanoparticles synthesized by biogenic method

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Abstract*– The green nanotechnology (biogenic method) is recognized for its broad advantages in different branches of the scientific field. In recent years, has been defined as an innovated field, related to the development of methodologies with little environmental contamination. In this research, the PRISMA methodology was applied, considered some selection criteria (Open access, language, quartile), obtaining a total of 31 investigation. This research concludes the direct influence of the extract and the size of the nanoparticles, and in that this factor is highly variable due to the dependence of its properties on their phytoconstituents and the territory. furthermore, a trend in the formation of spherically shape nanomaterials was identified. and finally, it is recognized that through the biogenic synthesis the formation of nanostructures was crystalline, obtaining a considerable level of purity in each nanoparticle considered.

Keywords*—Biogenic synthesis, Green synthesis, Nanoparticles, X-Ray Difracction- Characterization, Meta-analysis.

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I. INTRODUCTION

Nanotechnology is recognized for its broad advantages in different branches of the scientific field, related to the promising opportunities offered by its structures (<100 nanometers). Which allows it to interact extensively with other materials improving its durability and driving qualities. [1]–[3]. In this way, with technological innovation and new environmental needs, it has been optimizing.

In recent years, nanotechnology has been defined as an innovated field, supported by original investigations. A lot of these related to the development of methodologies with little environmental contamination, referring exactly to these as "green synthesis", "biosynthesis" "biogenic synthesis" due to the use of plants, algae, fungi and cyanobacteria as reducing or stabilizing agents [4]–[6], highlighting the advantages of using plants for their phenolic compounds [6]–[9] [6,7,8,9]. These nanostructures can be applied in different fields due to their physical, biological and chemical potential. [10], in addition to its biocompatibility in medicine [11] .

Despite this, its potential depends on the morphological characteristics and stability [4], [11], [12]. The reference [13] obtained different results applying *Phragmites australis* (Carrizo) root and rhizome extract. Agreeing that during the synthesis nanomaterials depend on the physicochemical properties of the resource [14], [15], in addition to pH [16], [17].

The aforementioned highlights the fundamental role played by the biological resource in the synthesis, and the influence of that factor on the final morphology characteristics of the nanostructures, so it is essential to analyze the different observations in the literature.

Therefore, this meta-analysis is focused on answering the research question: What are the morphological characteristics of the nanoparticles synthesized by the biogenic route? To provide an answer, this article aims to analyze the qualities of the nanostructures synthesized by the green route.

II. METHODOLOGY

In this research, the PRISMA methodology was applied, due to the structure in which it develops the compilation and selection of the bibliographic review, increasing the quality, which allows obtaining reliable results during the search [18].

Initially, the databases used had a remarkable reliability value, among them were: Cell Press, IOPScience, Multidisciplinary Digital Publishing Institute (MDPI) and Science Direct.

In the selection of the literature, the following selection criteria were considered, such as open access articles, focused on green synthesis or biogenic route, language (English), structure IMRD (Introduction, Methodology, Results and Discussion) and by last only considered silver (Ag NPs), iron (FeO NPs) and zinc (ZnO NPs) nanoparticles. In this way, those investigations that did not meet the previously described criteria were discarded. Likewise, the journal of the selected article had to present significant relevance (Q1/Q2) indexed in Scopus and Web of Science (WoS).

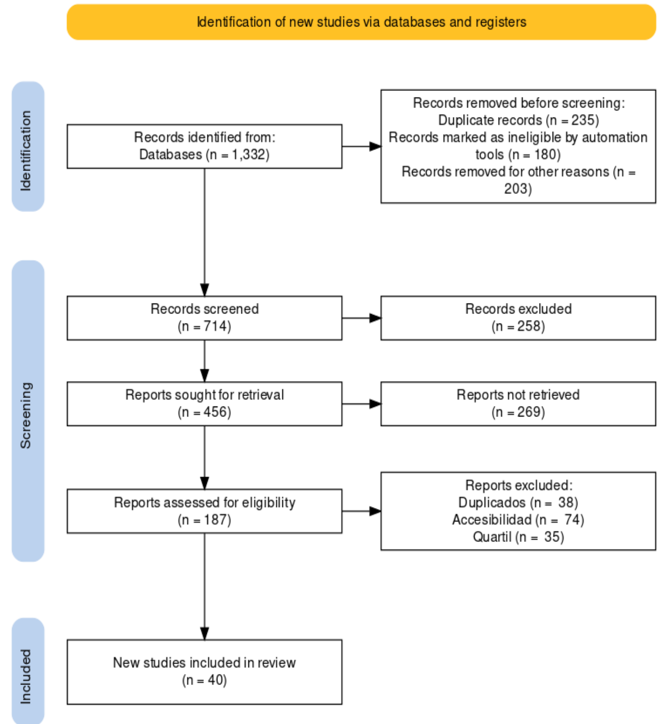

Fig. 1 Flowchart in the search and selection of Literature Diagram obtained from PRISMA 2020 Statement.

III. RESULTS

Applying the exclusion and inclusion criteria previously described, the bibliography consisted of 31 investigations. Table 1 describes some of the most relevant characteristics, categorizing it by time and type of document, documenting its frequency (F) and with this the percentage was acquired.

TABLE I
QUALITIES ARTICLES

Type of document	F	%	Year of publication	F	%
Articles	31	100.0	2017	2	6.7
Review	-	0.0	2018	2	6.7
			2019	2	6.7
			2020	4	13.3
			2021	7	23.3
			2022	14	43.3
TOTAL	31	100	**TOTAL**	31	100.0

Likewise, Table II discloses the structure that was applied to organize the information of each article, thus facilitating data collection. The following table only considers only some documents due to its extension if all the investigations were considered.

TABLE II
LITERATURE SCHEMATIZATION MODEL

N°	Title	Database	Journal	Q	Rf
1	Facile green silver nanoparticles synthesis to promote the antibacterial activity of cellulosic fabric.	Science direct	Journal of Industrial and Engineering Chemistry	Q1	[16]
2	Facile synthesis of silver nanoparticles using *Tribulus longipetalus* extract and their antioxidant and antibacterial activities.	Taylor & Francis	Internationa l Journal of Food Properties	Q2	[19]
3	Green synthesis of silver nanoparticles mediated by *Daucus carota L.*: antiradical, antimicrobial potentials, in vitro cytotoxicity against brain glioblastoma cells.	Taylor & Francis	Green Chemistry Letters & Reviews	Q2	[20]
4	Green synthesis of silver nanoparticles using *Ilex paraguariensis* extracts: antimicrobial activity and acetilcolinesterase modulation in rat brain tissue	Science direct	Green Chemistry Letters & Reviews	Q2	[17]
5	Green Synthesis of Silver Nanoparticles Using *Olea europaea* Leaf Extract for Their Enhanced Antibacterial, Antioxidant, Cytotoxic and Biocompatibility Applications	MDPI	Internationa l Journal of Molecular Sciences	Q1	[21]
6	Green synthesis of silver nanoparticles using *Rheum palmatum* root extract and their antibacterial activity against *Staphylococcus aureus* and Pseudomonas aeruginosa	Taylor & Francis	Artificial Cells, Nanomedici ne, and Biotechnolo gy	Q1	[22]
7	Green synthesis of silver nanoparticles using *Ipomoea aquatica* leaf extract and its cytotoxicity and antibacterial activity assay	Taylor & Francis	Green Chemistry Letters and Reviews	Q2	[23]
8	Unraveling the Active Biomolecules Responsible for the Sustainable Synthesis of Nanoscale Silver Particles through Nuclear Magnetic Resonance Metabolomics	ACS	ACS Sustainable Chemistry and Engineering	Q1	[7]
9	Biosynthesis and antibacterial activity of silver nanoparticles using *Flos Sophorae* Immaturus extract	SPRINGER LINK	Heliyon	Q1	[24]
10	Grape Pomace Extracted Tannin for Green Synthesis of Silver Nanoparticles: Assessment of Their Antidiabetic, Antioxidant Potential and Antimicrobial Activity	MDPI	Polymers	Q1	[25]

Next, Table III characterizes the chosen articles of silver (Ag NP), Iron oxide (FeO NP) and Zinc oxide (ZnO NP) nanoparticles according to the extract used (plant), Size, Shape and characterization by X-Ray Diffraction (XRD).

TABLE III
CHARACTERIZATION OF THE LITERATURE

Biological Resource	Experi. Cond.	Average Size (nm)	Shape	Characterization XRD	Rf
Ag NP					
Natural polysaccharide (BSEPS) produced by *Bacillus subtilis*	$AgNO_3$ (10mM) BSEPS al 2% y pH de 6	15–50	Spherical	Face-centered cubic (fcc) structure	[16]
Leaves of *T. longipetalus*	5 mL extract alcoholic (2% polar) : 0.01 M $AgNO_3$	15	Spherical	Crystallographic planes of face-centered cubic structure (fcc)	[19]
Leaves of *Daucus Carota L.*	400 mL (AgNO3,1mM) : 80mL (extract)	16.81 ±7.72	Spherical	Face-centered cubic (fcc) crystalline	[20]
Ilex paraguariensis	50 mL of Extract : 50 mL of silver nitrate	12.09	Varied	Face-centered cubic (fcc) and crystalline nature	[17]
Leaves of *Olea europaea*	1mM ($AgNO_3$) : 25 mL extract	8	Spherical, with uniform distribution	Intense peaks with high degree of crystallization	[21]
Roots of *R. palmatum*	5ml Extract : 100 ml (AgNO3 2mmol)	121.5	Spherical and hexagonal	Cubic crystal phase of Ag metal	[22]
Ipomoea aquatica leaf	10mL of Extract : 100 mL $AgNO_3$ (1mM)	18.81	Spherical	Cubic structure of silver	[23]
V. corymbosum L.	2.5 mL extract : 50 mL 1mM $AgNO_3$	20+- 5	Spherical	Monocrystalline	[7]
Flos Sophorae Immaturus	16 mL extract : 40 mL $AgNO_3$ (4mM)	36.5	Spherical	Face-centered cubic silver	[24]
Grape pomace (GP)	1000ppm extract : 10 mL $AgNO_3$ (1mM) ph 7	15 - 20	Spherical uniformly distributed	Face centered cubic (FCC) crystal structure.	[25]
FeO NP					
Olea europaea	20 mL extract :200 mL FeCl3 (1 Mm)	30	Spherical and polydispers e	Face centered cubic (FCC)	[26]
Carica papaya leaves	1 papaya leaves : 1 Ferric chloride hexahydrate (0.1M)	21.59	Uniform and agglomerat ed	Crystalline nature	[27]
Chlorophytum comosum leaf	9 ml leaf extract (5%) : 1ml of FeCl3?6H2O (0.1 M) at pH 6	>100	Spherical and amorphous	Insufficient distinctive diffraction peaks	[28]
Sida Cordifolia plant	5 ml of extract : 10 mL of Iron nitrate (0.01M) Ratio: (1:2)	16	Spherical nano clusters	uniform dispersion	[29]

Psidium guajava leaf extract,	50 mL of extract : 50 ml $FeCl_2.4H_2O$ (1M) and 50 mL$FeCl_3·6H_2O$ (2M)	21.75	Not uniform	Cubic crystallinity with crystalline nature	[30]
S. rebaudianawas (SRLe) obtained from Sugar Crops	4 mL extract :40 mL Ferric Cholride (2mM)[($FeCl_3·6H_2O$)]	19.60 ± 3.8	Spherical and aggregation	Matching nicely with the crystal structure of α-Fe_2O_3 NPs	[31]
The *P. obtuse* fresh leaves	100 mL extract : 50 mL ferrous acetate (3mM) $Fe(C_2H_3O_2)_2$)	50	Spherical shape	Pure phase of FeNPs, crystallographic planes	[32]
Centaurea solstitialis leaves	250 mL extract methanol : 100 mL $FeCL_3$ (0.1M)	Small	Spherical	Small uniform size	[33]
Withania coagulans. Berries	2 0 mL extract : 100 mL (1 M $FeCl_3.6H_2O$: 2 M$FeCl_2.4H_2O$)	16 ± 2	Irregular clusters with aggregation	Highly crystalline nature	[34]
Roots of *Picrorhiza kurroa*	3 mL aqueous extract: 50 mL solution of aq. ferric nitrate at 80 °C.	26	Hexagonal Rhomb-Centered geometry	-	[35]
Terminalia bellirica (TL)	0.5 M of Fe-salt : 1 extract	21.32	Single spherical particles and irregular shaped	The percent composition of C and O was observed	[36]
ZnO NP					
Moringa Olifeira	1:1 ratio (v/v) of zinc nitrate hepta-hydrate and the Gum sample	60	Small uniform distribution	Clear sharp peaks nature crystalline	[37]
The root extract of the *Saussurea lappa*	50 ml extract : 50 ml (hexahydrate zinc nitrate. 0.1M)	26+- 1	Hexagonal shape	Hexagonal wurtzite structure with slight agglomeration.	[38]
Citrus limon (L.) Burm. F. seeds	25 ml extract : 50 ml Zinc acetate (1 mM)	150	spherical-shaped particles	Face-centered cubic (fcc) are mentioned as crystal structured ZnO NPs with the hexagonal phase	[39]
Costus Pictus	50 ml $Zn(NO_3)_2·6H_2O$ (0.1M) : 10 mL of extract	40	elongated, hexagonal and rod-shaped some agglomerated	Crystalline size measured as 29.11 nm	[40]
Pyrus pyrifolia fruits	250 mL $Zn(NO_3)_2.6H_2O$ (0.1M) : 50 mL Aqueous fruit extract	-	Cubical, spherical uniform and agglomerated	Hexagonal phase of the wurtzite ZnO structure	[41]
Calotropis gigantea	-	80–100	Irregular spherical and agglomerated	Purity of ZnO nanoparticles by calcination	[42]
Waste peels of pomegranate (Pp)	50 mL extract : 25 mL zinc sulphate heptahydrate ($ZnSO_4·7H_2O$)(1.0 M)	38	Spherical-type hierarchical structure	Crystalline nature hexagonal wurtzite and crystalline phase	[43]

Pleurotus ostreatus	25 mL ZnNO3 (0.2M) : 10 ml of muchroom extract	7.50	Spherical and agglomerated	-	[10]
The bark of *Acacia caesia (L.) Willd.*	50 ml of extract : 5 mL of Zn (NO3)2.6H2O (10 mM)	32.32	Spherical, hexagonal and agglomerated	Hexagonal and crystal planes	[44]
Guava Plant Extract	100 mL of guava extract : Zinc chloride (ZnCl2)	50	Hexagonal well-define	Polycrystalline nature	[14]

IV. DISCUSSION AND CONCLUSIONS

As Table III shows, the Ag NPs are within a range of magnitude between 8-50 nm [16], [17], [21], [23], [24] which presented an adequate and uniform distribution, agreeing with what was indicated by Neme, K et al, since for the "nano" scale the results must have a proportion less than 100 nm [1]. Despite this, investigations such as that of Arokiyaraj, S. et al do not meet that magnitude [22]. Therefore, to identify the qualities of nanostructures it is vital to abide by the established range and thus evaluate within the same criteria.

Regarding the FeO NPs, the majority had sizes smaller than 50 nm, however, they presented agglomerations [27], [30], [31], [33], [34], which can be attributed to the predominant bioactives of the plant extract. This agrees with what was pointed out by [26] who concluded that oleuropein, tyrosol and antioxidant compounds have an essential role during the synthesis, specially in the regulation of nanoparticles [32], [36], which can lead to them to obtain a high aggregation capacity [28].

For ZnO NPs, variable sizes greater than 30 nm were obtained, which is why they were more prone to agglomerate [38], [40]–[42], [44]. Although Mkhize, S. et al obtained a smaller size, the nanostructures were agglomerated [10]. The reference [45] mention the influence of the precursor salt,
especially with the use of ZnSO4 on the size and shape. Despite this, the most variable factor in the synthesis is the influence of the extract, since it is subject to the phytoconstituent chemical properties in the extracts.

Due to the aforementioned, [20] attribute the variability of the size of the nanomaterials to the proportionality of the extract. This coincides with the results of [7], [17] since they identified that, with greater volume, the magnitude of the nanoparticle also increased.

Table III also shows us the shape. In the literature, spherical nanoparticles [10], [20], [23], [25], [26], [31]–[33], [39], [43], as hexagonal [22], [35], [38] were more frequently obtained [37], [40], [44] and finally cubic [23], [28], [30] in all the nanomaterials considered in this review. Likewise, nanoclusters [29], [34] are mentioned in FeO NPs and amorphism [27], [28], [42] particularly in ZnO NPs and Feo NPs. Coinciding with the research by [11], stating that the biogenic route has the potential to develop defined nanostructures with various shapes, which determine the type of behavior with the membrane or enzymes of different microorganisms, which increases their potential. of application in different fields [46].

The XRD results in this review allowed us to identify the crystalline state of the metal, so for the Ag NPs they presented a crystalline centered cubic structure [17], [19], [24], [25], the FeO NPs mostly obtained a cubic crystalline structure [26], [30], [32], [34] and geometric hexagonal centered in the rhombus [35]. Regarding the ZnO NPs, a crystalline nature of wurtzite hexagon was obtained [37]–[39], [41], [43]. Therefore, it is attributed to the stable formation of nanostructures synthesized by biogenic route applying plant extracts, agreeing with [11] that indicates the effectiveness of its agent to affect the size and shape of nanomaterials.

V. CONCLUSIONS

By virtue of this, the article concludes that the proportionality of the biological extract applied directly influences the size and shape of the nanoparticles. However, these have a high level of variability due to the constituent properties of each resource, which depend on the territory.

Likewise, it was identified that the nanostructures synthesized by the biogenic route have a tendency to form spherical structures, while the formation of other structures (hexagonal, nanoclusters) is recognized despite being moderately agglomerated.

Finally, the formation of crystalline nanostructures through biogenic synthesis is recognized, as well as the purity of the nanomaterials obtained thanks to X-Ray Diffraction analysis.

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