

Recent advances (2016 - 2020) in green synthesis of metal oxide nanoparticles: An overview

Avanços recentes (2016 - 2020) na síntese verde de nanopartículas de óxidos metálicos: Uma visão geral

Avances recientes (2016 - 2020) en la síntesis verde de nanopartículas de óxidos metálicos: Una descripción general

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Abstract

The development of products and processes which are more ecological has been a source of interest in research in recent decades. Thus, researchers have developed several methods to improve the synthesis of oxides and metals in a more ecological way. Green synthesis has become a method that is being widely used for presenting low temperature, low cost and great availability of raw material. Some biological sources such as fungi, bacteria and plants are used in this route, but plant extracts are the most used in the literature. Various parts of the plant such as the roots, bark, flowers, fruits, stem, seeds, and leaves are rich in secondary metabolites such as: flavonoids, phenols, steroids, terpenoids, alkaloids, saponins and phenolic compounds, which play an essential role in the complexation, polymerization, capping and stabilization processes that generate oxides. The methodology used in the synthesis process significantly interferes in some properties and characteristics of the same oxide as its morphology. In this sense, this paper presents a review on green synthesis in obtaining oxides and their morphologies.

Keywords: Green chemistry; Ceramics; Synthesis route; Nanomaterials.

Resumo

O desenvolvimento de produtos e processos que sejam mais ecológicos têm sido motivo de interesse de pesquisas nas últimas décadas. Dessa forma, os pesquisadores têm desenvolvido diversos métodos para aprimorar a síntese de óxidos e metais de forma mais ecológica. A síntese verde tem se tornado um método que está sendo bastante utilizado por apresentar baixa toxicidade, baixo custo e grande disponibilidade de matéria prima. Nesta rota são utilizadas algumas fontes biológicas como fungos, bactérias e plantas, porém os extratos de plantas são os mais relatados na literatura. Várias partes da planta como raízes, casca, flores, frutos, caule, sementes e folhas são ricas em metabólitos secundários como: flavonoides, fenóis, esteroides, terpenóides, alcaloides, saponinas e compostos fenólicos, que desempenham um papel essencial nos processos de complexação, polimerização, capeamento e estabilização na síntese verde de óxidos. A metodologia utilizada no processo de síntese interfere de maneira significativa nas propriedades e características dos óxidos, especialmente em sua morfologia. Neste sentido, este trabalho apresenta uma análise dos avanços registrados entre 2016 e 2020 no campo da síntese verde na obtenção de óxidos e suas diferentes morfologias.

Palavras-chave: Química verde; Cerâmicos; Rota de síntese; Nanomateriais.

Resumen

El desarrollo de productos y procesos más ecológicos ha atraído mucha atención en los últimos años. Así, los investigadores han desarrollado varios métodos para mejorar la síntesis de óxidos y metales de una forma más ecológica. La síntesis verde se ha convertido en un método muy utilizado por su baja toxicidad, bajo costo y alta disponibilidad de materia prima. En esta ruta se utilizan algunas fuentes biológicas como hongos, bacterias y plantas, pero los extractos de plantas son los más utilizados en la literatura. Varias partes de la planta como raíces, corteza, flores, frutos, tallo, semillas y hojas son ricas en metabolitos secundarios como: flavonoides, fenoles, esteroides, terpenoides, alcaloides, saponinas y compuestos fenólicos juegan un papel esencial en el proceso de polimerización, cableado y estabilización que generan los óxidos. La metodología utilizada en el proceso de síntesis interfiere aproximadamente en algunas propiedades y características del mismo óxido como su morfología. En este sentido, este trabajo presenta una revisión de la síntesis verde en la obtención de óxidos y sus morfologías.

Palabras clave: Química verde; Cerámica; Ruta de síntesis; Nanomateriales.

1. Introduction

Science and technology at the nanometric scale have attracted a lot of attention from researchers due to its use and its varied applications. Many advanced devices rely on nanostructured materials. Thus, nanotechnology has emerged as a new research field regarding the preparation of nanomaterials and nanoparticles (NPs) because of its multifunctional properties and efficiency. Nanometric materials with unique optical, magnetic, electronic and catalytic properties, as well as distinct structure, composition and morphology characteristics have drawn attention to different areas such as chemistry, physics, materials science, and engineering, etc.

Nanoparticles are generally obtained by two methods: “top-down” and “bottom-up”. The first approach is based on fragmenting larger structures (bulk) using ablation, grinding and cutting techniques until reaching a nanometric structure. However, the bottom-up approach usually disposes of the chemical properties of atoms and molecules to attract each other and agglomerate to form an organized structure of nanometric size. Some synthesis routes that use this approach include: sol-gel method, chemical vapor deposition, Pechini, microemulsion, and green synthesis, etc. (Aarthy *et al.*, 2021).

Due to environmental concerns, the development of products and processes which are more ecological has gained a lot of attention in recent years. In this sense, researchers have developed several methods to improve the synthesis of metals and oxides using more ecological routes. The biosynthesis or green synthesis method presents itself as an alternative route to avoid the use of chemicals, reactions and processes that are dangerous to human beings and/or the environment, in addition to presenting a low production cost and being able to obtain a product with a high yield. In this synthesis, nanomaterials are obtained from extracts of plants, microorganisms, fungi, yeasts, algae, and so on (Aarthy *et al.*, 2021; Abinaya *et al.*, 2021), which generally makes it a low-cost process, in addition to wide availability and using less toxic reagents. Fatty acids, polysaccharides, proteins, phenolic compounds, secondary metabolites present in plants and microorganisms help in the synthesis process of oxides and metals. Plant extracts are the most found in the literature among the alternative media used due to their rapid synthesis and wide availability (Moraes *et al.*, 2021). Another potential point in green synthesis is the possibility of infinite combinations of extracts to manipulate the structure and morphology of nanoparticles.

Aiming to explore the potential of green synthesis, the objective of this work is to present recent studies (2016 – 2020) on this method in producing metal oxides, with an emphasis on the effects of the synthesis process on the morphology of the obtained nanoparticles.

2. Methodology

This work consisted of a review integrated to the literature, conducted in the second half of 2021. According to Fink (2019), the use of this type of research is an organized and reproducible way which provides quick access to results on a given subject, such as also carrying out a mapping of what the literature presents about some given content, helping to delimit

research and to show how studies differ and progress in relation to the knowledge already carried out in the area (Lüdke *et al.*, 2012).

This literature review was carried out by searching the SCOPUS, Scielo, Google Scholar, CAPES Periodicals, and Science Direct digital databases, covering the period from 2016 to 2021 and using the following descriptors: green synthesis, oxides, fungi, plants, and microorganisms. Exclusion criteria were: articles published more than 6 years ago, articles in abstract format, monographs, Master's dissertations, abstracts of works and doctoral theses.

3. Results and Discussion

3.1 Plant extract use

There are several biological sources used in this type of synthesis, however the use of plant extracts are the most reported in the literature, as plants are true “biological factories” of nature and present huge metabolic composition that will help in the formation of nanoparticles, in addition to being a faster synthesis compared to that using micro-organisms (Aarthy *et al.*, 2021; Bibi *et al.*, 2019; Iravani, 2011).

Various parts of the plant are used for synthesis such as roots, flowers, seeds and stems. But leaves are the most widely reported in the literature. Plant extracts have several components that can act as reducing, complexing, polymerizing and stabilizing agents due to the combination of molecules present in the extract, and thus help in the synthesis (Shamaila *et al.*, 2016). Among these, the following stand out: amino acids, flavonoids, phenols, citric acid, terpenoids, bioactive polyphenols, and alkaloids, among other organic compounds such as alcohols, aldehydes and amines (Iravani, 2011; Shamaila *et al.*, 2016). Polyphenol phytomolecules have antioxidant capacity which are easily absorbed by nanoparticles that help to reduce the nanoparticle size, for example (Aarthy *et al.*, 2021). Table 1 shows the main works found in the literature (2016 – 2020) on oxides synthesized using plant extracts and Figure 1 makes the correlations between the characteristics.

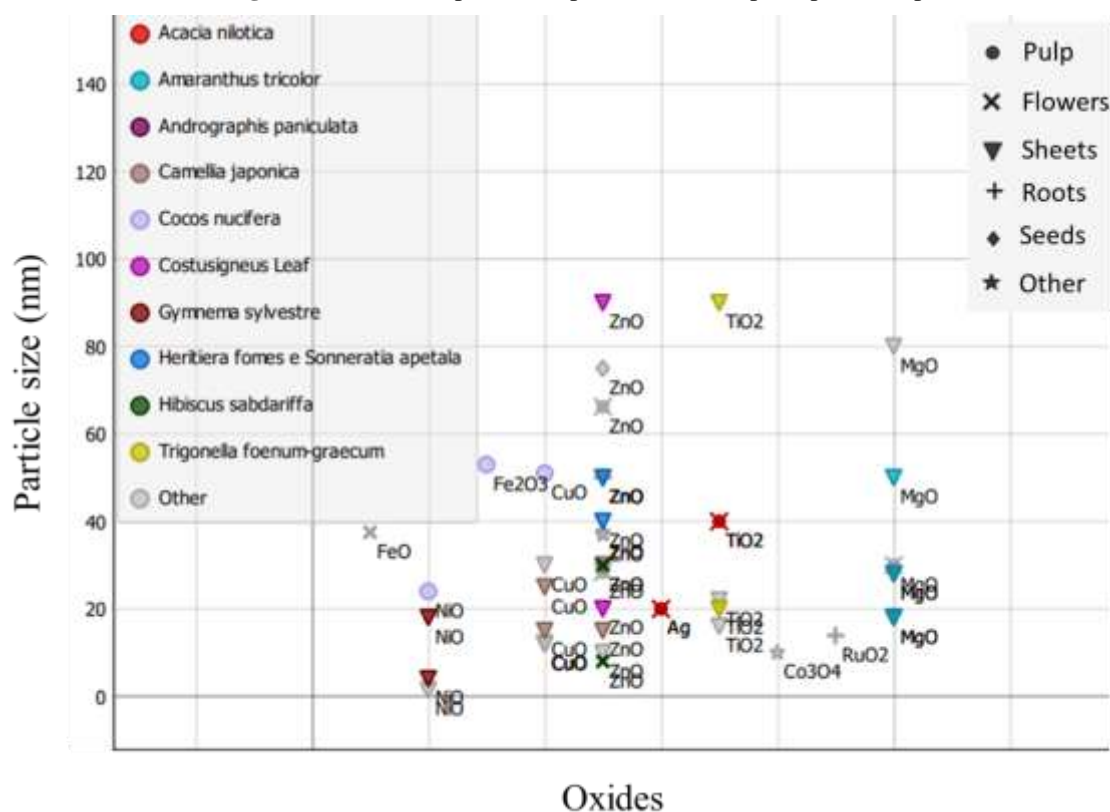
Table 1. Oxides synthesized by the green synthesis method using plant extracts.

Plant	Part of the plant used in the synthesis	Oxide obtained	Particle size (nm)	Morphology	Reference
<i>Punica granatum</i>	seeds	Fe ₂ O ₃	-	Spherical	(Bibi <i>et al.</i> , 2019)
<i>Avicennia marina</i>	flowers	FeO	37.5	Spherical	(Karpagavinayagam <i>et al.</i> , 2019)
<i>Gymnema sylvestre</i>	leaves	NiO	4-18	Spherical	(Ramalingam <i>et al.</i> , 2019)
<i>Ananas comosus</i>	leaves	NiO	1.41 – 1.76	Monodispersible	(Olajire <i>et al.</i> , 2020)
<i>Cocos nucifera</i>	Pulp	CuO;	51	Spherical	(Muthuvinothini <i>et al.</i> , 2019)
<i>Cocos nucifera</i>	Pulp	NiO;	24	Cubic monodispersed	
<i>Cocos nucifera</i>	Pulp	Fe ₂ O ₃	53	Distorted spherical	
<i>Heritiera fomes and Sonneratia apetala</i>	leaves	ZnO	400-500	-	(Thatoi <i>et al.</i> , 2016)
<i>Linnium usitatissimum</i>	seeds	ZnO	75	Nano leaves	(Moghaddas <i>et al.</i> , 2019)
<i>Costusigneus Leaf</i>	leaves	ZnO	20-90	Spherical	(Chinnasamy <i>et al.</i> , 2018)
<i>Musa spp.</i>	bark	ZnO	370	Sphere/globular	(Dutta <i>et al.</i> , 2019)
<i>Stevia (adoçante natural)</i>	leaves	ZnO	10-50	Retangular	(Khatami <i>et al.</i> , 2018)
<i>Trianthema portulacastrum</i>	-	ZnO	28.4-66.2	Aggregated	(Khan <i>et al.</i> , 2019)
<i>Averrhoa bilimbi (L)</i>	fruit	ZnO	37.5	Spherical	(Ramanarayanan <i>et al.</i> , 2018)

<i>Typha latifolia.L</i>	leaves	ZnO	-	Nano flowers	(Kumar <i>et al.</i> , 2019)
<i>Hibiscus sabdariffa</i>	flower	ZnO	8- 30	Semi-spherical	(Soto-Robles <i>et al.</i> , 2019)
<i>Codonopsis lanceolata</i>	roots	ZnO	500	Flower	(Lu <i>et al.</i> , 2019)
<i>Scutellaria baicalensis</i>	roots	ZnO	50	Spherical	(Chen <i>et al.</i> , 2019)
<i>Calliandra haematocephala</i>	leaves	ZnO	-	Flower shape	(Vinayagam <i>et al.</i> , 2020)
<i>Camellia japonica</i>	leaves	ZnO	15-30	Spherical	(Maruthupandy <i>et al.</i> , 2017)
<i>Acacia nilotica</i>	-	Ag / TiO ₂	20-40	Spherical	(Rao <i>et al.</i> , 2019)
<i>Espania grandiflora</i>	leaves	TiO ₂	160-220	Spherical	(Srinivasan <i>et al.</i> , 2019)
<i>Trigonella foenum-graecum</i>	leaves	TiO ₂	20-90	Spherical	(Subhapiya <i>et al.</i> , 2018)
<i>Calotropis procera</i>	latex	Co ₃ O ₄	10	Spherical	(Dubey <i>et al.</i> , 2018)
<i>Anacyclus pyrethrum</i>	root	RuO ₂	13.9	Almost spherical	(Nisha <i>et al.</i> , 2020)
<i>Azadirachta indica</i>	leaves	CuO	12	Spherical	(Rehana <i>et al.</i> , 2017)
<i>Hibiscus rosa-sinensis</i>	leaves	CuO	12	Esférica	
<i>Murraya koenigii</i>	leaves	CuO	12	Spherical	
<i>Moringa oleifera</i>	leaves	CuO	12	Spherical	
<i>Tamarindus indica</i>	leaves	CuO	12	Spherical	
<i>Camellia japonica</i>	leaves	CuO	15-25	Spherical	(Maruthupandy <i>et al.</i> , 2017)
<i>Thymus vulgaris L.</i>	leaves	CuO	30	Spherical	(Nasrollahzadeh <i>et al.</i> , 2016)
<i>Acanthospermum hispidum</i>	leaves	CuO	-	Spherical	(Gowri <i>et al.</i> , 2019)
<i>Amaranthus tricolor</i>	leaves	MgO	18-28	Hexagonal	(Jeevanandam <i>et al.</i> , 2017)
<i>Amaranthus blitum</i>	leaves	MgO	50-80	Spherical	
<i>Andrographis paniculata</i>	leaves	MgO	18-28	Spherical	
<i>Solanum trilobatum</i>	-	MgO	30	Spherical	

Source: Authors.

Figure 1. Relationship between particle size and plant part and species.



Source: Authors.

These extracts also behave as a capping agent which controls the particle shape and size in the synthesized material. A very important fact in this type of route is that each plant component presents a variation in its composition or phytochemical concentration according to the tissue or the conditions it may be in, such as temperature and humidity, for example (Narendhran *et al.*, 2019). Thus, both the phytochemical composition and the selected plant part will influence the efficiency of the extract in forming the desired nanoparticles (Shamaila *et al.*, 2016). One of the criteria for selecting the plant is its geographical disposition, an unprecedented report, in addition to its phytochemical composition (Das *et al.*, 2013).

The morphological properties are influenced by several aspects such as the pH, synthesis temperature, the extract concentration and volume. The review work by Akintelu *et al.* (2020) showed how the type of extract used in the synthesis, as well as its concentration, influenced the synthesis of copper oxide, obtaining different molds and applications. Devatha *et al.* (2018) identified and quantized the polyphenols present in the extracts using HPLC (High Performance Liquid Chromatography) and concluded that the greater the availability of these phytochemicals present in the medium, the faster their nanoparticle formation rate. Another important feature in obtaining nanoparticles is the volume and extract ratio used and the type of extract and its concentration, which was observed by Devatha *et al.* (2018) and Fazlzadeh *et al.* (2017). The authors observed that these parameters significantly influence both morphological and biological properties of nanoparticles. Devatha *et al.* (2018) found that the 1:2 ratio of iron particles had sizes 98–200 nm, while the others had particles ranging between 200 and 600 nm and these had more agglomerates. Its bacterial inhibition activity increased as the proportions increased from 1:2 to 1:5. A study by Fazlzadeh *et al.* (2017) verified that the different plant extracts interfered in the shapes, sizes and characteristics of the nanoparticles and thus altered the ability to remove aqueous pollutants.

According to Table 1, it is possible to observe that the main oxides studied are NiO, ZnO and MgO, as they are more interesting for biological, photocatalytic, catalytic, and biosensor activities (among others). It is also possible to notice that

there is a variation in morphology for ZnO from a spherical to flower type when using *Scutellaria baicalensis* and *Camellia japonica*, as well as *Codonopsis lanceolata* and *Calliandra haematocephala* plants, which is due to the fact that they present differences in the chemical composition of the extract and under synthesis conditions. However, there is no difference in morphology for CuO, only differences in crystallite size. Figure 1 shows the relationship of oxides with particle sizes smaller than 20 nm using extracts obtained from leaves, regardless of the species used to extract the phytochemicals. However, this can be directly attributed to the use of the leaf to obtain nanoparticles, since the large volume of work in the literature is using leaves combined with other process variables such as pH, calcination temperature or the extract concentration.

3.2 Bacteria use

The synthesis of metals and oxides can occur via an extra or intracellular mechanism when green synthesis occurs through using bacteria. In the first case (extracellular), studies suggest that the growth and stabilization of nanoparticles are made from enzymes and proteins secreted by bacteria (Bandeira *et al.*, 2020). Synthesis in the intracellular method begins with electrostatic interaction of the cell wall (negative charge) with metal ions (positive charge). Then these ions are captured into the cell interior after contact, and the enzymes present in the cell wall will reduce the ions to nanoparticles (Hulkoti *et al.*, 2014).

From the medium, intra- and extracellular routes obtained different morphologies of oxides with varied sizes. The use of bacterial cultures has advantages such as: low cost-benefit, easy culture, moderate experimental conditions and stability in the production of nanoparticles (Ghasemi *et al.*, 2017). Several bacteria were used as a biological source of green synthesis to obtain oxides. Table 2 presents some works which performed this biosynthesis.

Table 2. Synthesis of metal oxides using bacteria.

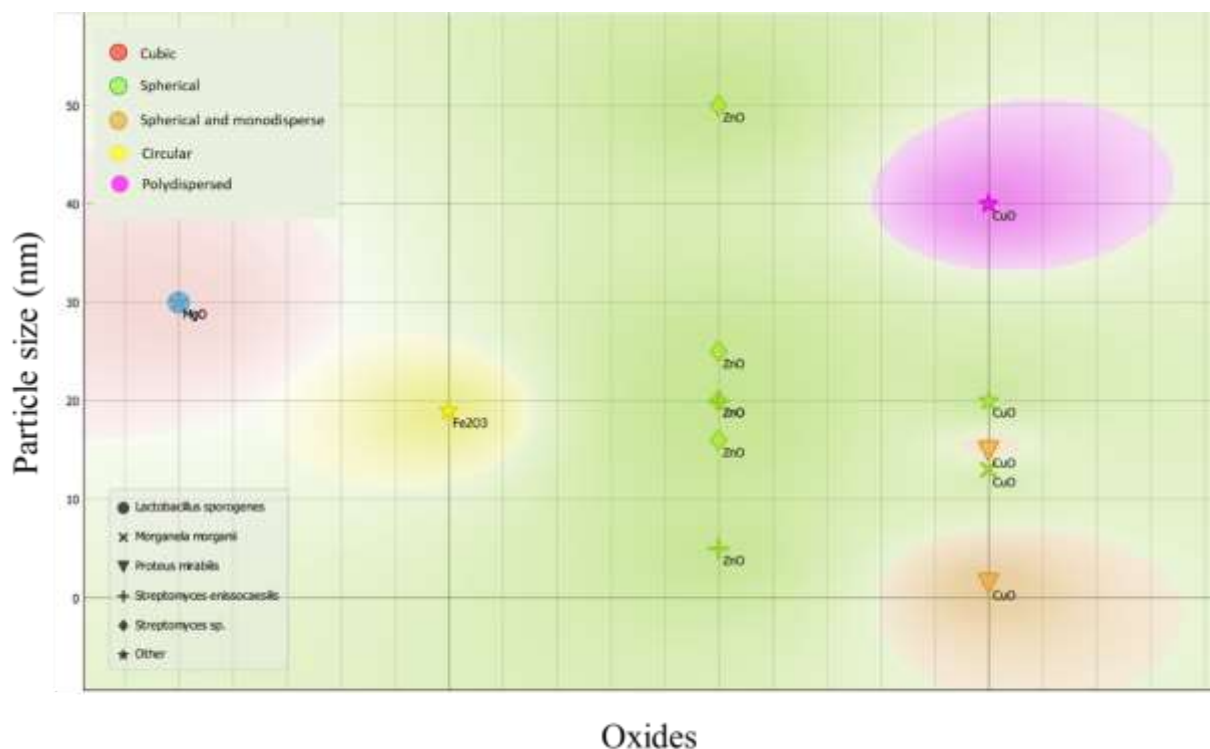
Bacteria	Oxide obtained	Particle size (nm)	Morphology	Reference
<i>Lactobacillus plantarum</i>	MgO	30	Cubic	(Mohanasrinivasan <i>et al.</i> , 2018)
<i>Lactobacillus sporogenes</i>	MgO	30	-	
<i>Desulfovibrio, cepa LS4</i>	Fe ₂ O ₃	19	Rounded	(Das <i>et al.</i> , 2018)
<i>Streptomyces sp.</i>	ZnO	16-25	Spherical	(Shanmugasundaram <i>et al.</i> , 2017)
<i>Streptomyces sp.</i>	ZnO	20-50	Spherical	(Balraj <i>et al.</i> , 2017)
<i>Streptomyces enissocaealis</i>	ZnO	5-20	Spherical	(Shaaban <i>et al.</i> , 2018)
<i>Desulfovibrio marinisediminis</i>	CuO	400	Polydispersed	(Alasvand <i>et al.</i> , 2016)
<i>Morganella morganii</i>	CuO	13	Spherical	(Ghasemi <i>et al.</i> , 2017)
<i>Proteus mirabilis</i>	CuO	1.44-14.9	Spherical and monodispersed	(Eltarahony <i>et al.</i> , 2018)
<i>Lactobacillus casei</i>	CuO	200	Spherical	(Kouhkan <i>et al.</i> , 2020)

Source: Authors.

In the work by Jacob *et al.* (2019), it was found that the particle texture and size are affected with the variation in the pH of the medium in the synthesis of iron oxide with *Streptomyces* spp. via the extracellular pathway. The pH considered ideal for this synthesis was 7 and 9 nm, and they also concluded that temperature is an important factor in the morphology of the material.

According to Table 2, it is possible to observe that the main oxides studied are MgO, ZnO and CuO due to their application in several areas such as: catalysis, semiconductor, biomaterial, environmental and so on. It is also possible to notice that there was no variation in morphology for ZnO and CuO, but only differences in crystallite size. Figure 2 indicates that the spherical morphology is related to nanoparticles smaller than 10 nm.

Figure 2. Relationship between particle size and bacterial species and morphology.



Source: Authors.

3.3 Fungi use

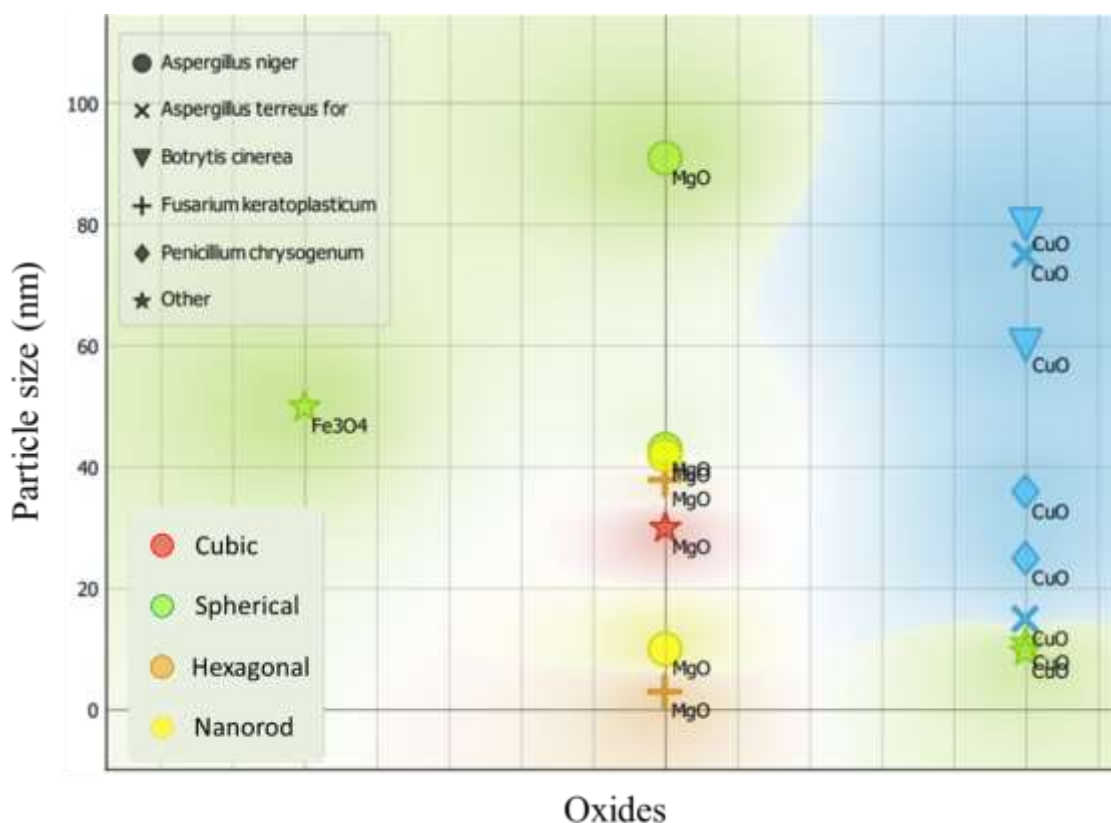
Fungi can be very promising in synthesizing nanoparticles on a large scale, as they can be easily cultivated in both the laboratory and on an industrial scale. Fungal endophytes have a great capacity for bioaccumulation and bioreduction, in addition to being sources of secondary metabolites and active biomolecules that make them effective in obtaining nanoparticles (Gupta *et al.*, 2020). Species known as *Trichoderma* manage to generate different metabolites such as pyrones, terpenes, diketopiperazine, enzymes, glycolipids and polyketides that can help in the synthesis of oxides (Nadeem *et al.*, 2020). Some fungi species such as *F. oxysporum* secrete proteins, polymers and enzymes that help in the stability and yield of the formed nanoparticles (Gupta *et al.*, 2020). Like bacteria, fungi use two pathways for synthesis: intracellular and extracellular. Works show that the intracellular synthesis route presents samples with smaller sizes and with a good dispersion compared to the extracellular route (Waris *et al.*, 2021). However, the extracellular synthesis pathway has been widely used, in which the components to be used by the synthesis are secreted by the fungi and thus the obtainment takes place outside the cellular components of the fungus. Several fungi are reported in the literature on oxide synthesis, as can be seen in Table 3. Figure 3 lists the fungus species with the particle size and morphology.

Table 3. Green synthesis of metal oxides using fungi as a source.

Fungus	Oxide obtained	Particle size (nm)	Morphology	Reference
<i>Lactobacillus plantarum</i>	MgO	30	Cubic	(Mohanasrinivasan <i>et al.</i> , 2018)
<i>Aspergillus niger</i>	MgO	43-91	Spherical	(Ibrahim <i>et al.</i> , 2017)
<i>Aspergillus niger SIM 1</i>	Fe ₃ O ₄	50	Spherical	(Abdeen <i>et al.</i> , 2016)
<i>Aspergillus niger</i>	MgO	10-42	Nanorod	(Mohamed <i>et al.</i> , 2019)
<i>Fusarium keratoplasticum</i>	MgO	3-38	Hexagonal	(Saravanakumar <i>et al.</i> , 2019)
<i>Trichoderma asperellum</i>	CuO	110	Spherical	(El-sayyad, 2019)
<i>Penicillium chrysogenum-Mediated</i>	CuO	9.70	Spherical	(El-sayyad, 2019)
<i>Botrytis cinerea</i>	CuO	60-80	-	(Kovačec <i>et al.</i> , 2017)
<i>Aspergillus terreus for</i>	CuO	15-75	-	(Mousa <i>et al.</i> , 2020)
<i>Penicillium chrysogenum</i>	CuO	25-36	-	(El-batal <i>et al.</i> , 2018)

Source: Authors.

Figure 3. Relationship between particle size and fungus species and morphology.



Source: Authors.

Kalpna *et al.* (2019) synthesized ZnO using *Aspergillus niger* as a culture and obtained particle sizes between 84-91 nm and spherical morphology, and verified its use in the removal of Bismarck's brown dye and its microbial activity. The synthesized nanoparticles proved to be effective in degradation in the treatment of Bismarck brown wastewater and an excellent microbial activity of 12 mm for *Staphylococcus aureus* and 10 mm for *E. coli*.

Fouda *et al.* (2018) synthesized ZnO with spherical morphology and an average size of 10-45 nm using the fungus *Aspergillus terreus*. Antibacterial tests and in vitro cytotoxicity of biosynthesized ZnO were performed, and concluded that the nanoparticles had better inhibitory action against *Staphylococcus aureus* and *Bacillus subtilis* (Gram positive) bacteria than *Pseudomonas aeruginosa* and *Escherichia coli* (Gram negative).

According to Table 3 and Figure 3, it is possible to observe that the main oxides synthesized using fungi were MgO

and CuO, as they exhibit excellent physical-chemical properties such as: corrosion resistance, low electrical conductivity, physical resistance, and mechanical strength, etc., and they are applied in several areas. It is also possible to notice that there is a variation in morphology for MgO from cubic to spherical, nanorod, hexagonal when using the *Lactobacillus plantarum*, *Aspergillus niger*, *Aspergillus niger* and *Fusarium keratoplasticum* fungi, respectively. This is due to the difference in the chemical composition present in the extract and also its synthesis route. However, CuO did not exhibit this variation in its morphology, only in crystallite size variation.

4. Final Considerations

The oxides obtained through conventional routes are used in several applications, but these synthesis routes are not ecological since there is the use of toxic reagents as well as the production of by-products that are harmful to the ecosystem due to their toxicity and harmful to human health. Given this scenario, green synthesis is a route that has been presenting itself as an excellent viable and ecologically friendly proposal for producing metallic oxides. Furthermore, it explores the chemical potential of plant phytochemicals in forming and improving the physicochemical properties of the synthesized products. Thus, green synthesis has shown considerable expansion due to these factors, as it provides adequate solutions to eliminate the problems (use of harmful reagents and production of toxic by-products) generated by conventional routes. As it is still a relatively new synthesis, there is a wide variety of methodologies used to obtain the material under study. Thus, this review comprehensively provides the main oxides synthesized in recent years and the use of different extract sources in the morphology and size of the particles generated. Therefore, this paper will significantly contribute to the national and international scientific literature, bringing a more specific material for researchers seeking to elaborate their projects on the green synthesis of metallic oxides.

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