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Article

Morphological analysis and its effect on the optical properties of carbon nanospheres as a function of synthesis time

Análisis morfológico y su efecto en las propiedades ópticas de las nanoesferas de carbono en función del tiempo de síntesis

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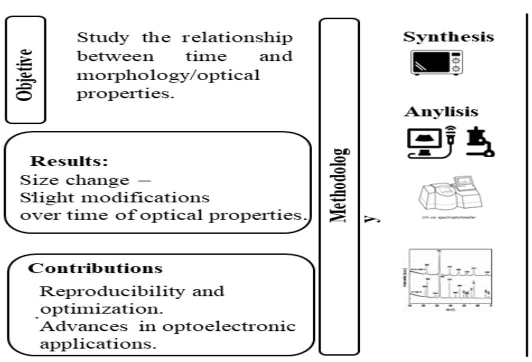
Abstract

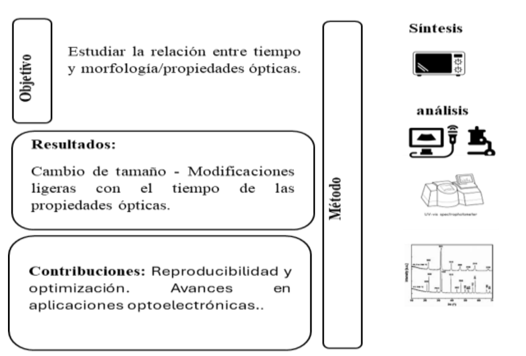
This work presents the effect of synthesis time on morphology and its influence on the optical properties of microwave-assisted carbon nanospheres. To analyze the correlation between the synthesis time and the growth in size of the nanospheres, the morphology of the nanostructures was characterized by scanning electron microscopy (SEM) and X-ray diffraction (XRD). Their optical properties were analyzed by UV-Vis spectrophotometry, and the Tauc method was used to estimate the optical band gap. The results reveal that the time variation in the synthesis does have a significant influence on the size, shape, and amount of sample obtained. The optical properties presented slight modifications. With these results, we can provide an easy reproducibility of simple and inexpensive methods to optimize the growth of carbon-based nanoparticles, which can bring advances in the synthesis of materials and improvements in their optoelectronic applications.



Nanospheres, Morphology, UV-Vis

Resumen

El efecto del tiempo de síntesis en la morfología y su influencia en las propiedades ópticas de las nanósferas de carbono asistidas por microondas se presenta en este trabajo. Para analizar la correlación entre el tiempo de síntesis y el crecimiento en tamaño de las nanósferas, se caracterizó la morfología de las nanoestructuras mediante microscopía electrónica de barrido (SEM) y difracción de rayos X (XRD). Sus propiedades ópticas se analizaron mediante espectrofotometría UV-Vis, y se utilizó el método de Tauc para estimar el ancho de banda óptico. Los resultados revelan que la variación del tiempo en la síntesis tiene una influencia significativa en el tamaño, la forma y la cantidad de muestra obtenida. Las propiedades ópticas presentaron ligeras modificaciones. Con estos resultados, podemos proporcionar una fácil reproducibilidad de métodos simples y económicos para optimizar el crecimiento de nanopartículas a base de carbono, lo que puede generar avances en la síntesis de materiales y mejoras en sus aplicaciones optoelectrónicas.



Nanoesferas, Morfologia, UV-Vis

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Introduction

The continuous search for simple, inexpensive, and innovative methods to obtain carbon-based nanostructures such as: carbon nanotubes, fullerenes, nanoonions, etc. (Ordoñez et al., 2013, Ordoñez et al 2019), has been a key driver in this research. In addition, carbon nanostructures have great potential in different applications, covering areas such as materials science, biomedicine, and nanotechnology.

In recent years, the alternative use of the microwave oven around materials science has proven to be a valuable tool for reducing synthesis costs (Adeola, et al.,2023).

Microwave-assisted syntheses offer advantages over conventional heating techniques by avoiding temperature gradients and long reaction times. The effectiveness of microwaves in materials synthesis is attributed to the ability of some chemical compounds to absorb energy in the microwave range and convert it into heat, thus accelerating the generated reactions (Pawelski D et al., 2023).

Similarly, the microwave radiation synthesis technique has also become popular because of (Zhou, J., Xu et al.,2020). A pioneering study conducted by the University of Wyoming has demonstrated the effectiveness of this method in converting raw carbon powder into nano graphite, a finding that reinforces the use of microwave technology in the production of C60 and C70 fullerenes (Masi et al. 2021), as well as carbon quantum dots (De Medeiros et al., 2019), demonstrating size variation in allotropic materials.

In recent years, this technique has also been used in a wide range of materials beyond carbon allotropes, encompassing inorganic, organic compounds, and biomaterials (Siebert et al.,2019). Mainly focused on increasing the improvement of the purity and properties of the materials obtained (Kumar, A et al., 2020).

Some materials that present improvements using this technique are nanoparticles of metals such as silver (AgNP), gold and platinum (Dankovich, 2014), allowing a finer control over the size, morphology, and distribution of the nanoparticles. As well as, titanium dioxide (TiO_2), zinc oxide (ZnO) and (Fe+2) (Ashok, 2014. Zhu,2012. Pati ,2014).

Some ceramics improve their density and mechanical properties (Borrel et al., 2013), and in the case of polymers obtaining them can achieve improvements in properties (Kuo,2016).

From these previous investigations, we carried out the preparation of 4 samples with 99% purity grade graphite powder in wafer form, using as a synthesis method a conventional microwave, which emits electromagnetic radiation around 2.45 GHz.

The main approach was to vary only the synthesis time of the samples every 2.5 minutes. The results revealed a diversity of morphologies, but the presence of sphere-like nanostructures stands out.

Corroborating that the synthesis time does affect the morphology and size. With respect to the optical properties, they were analyzed by UV-Vis spectroscopy and using the Tauc method, for the study of the optical forbidden band, it is verified that any change in the surface, size and presence of impurities affects the properties.

Methodology

A conventional microwave oven with electromagnetic radiation around 2.45 GHz at an output power of 1000 W was used. As carbon source, powdered graphite with a purity of 99% was used, using equivalent amounts of 1 milligram per sample placed in a ceramic crucible, completely covered.

Four samples were prepared, varying the synthesis time at intervals of 2.5 minutes each. The initial amount per sample of graphite powder was 1.2 grams. At the end of each synthesis, they were weighed and an average decrease of .2 grams per sample was observed.

Results and discussion

SEM characterization

Figure 1 shows the SEM micrographs of the four samples using scanning electron microscope, (SU5000, Hitachi).

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Box 1

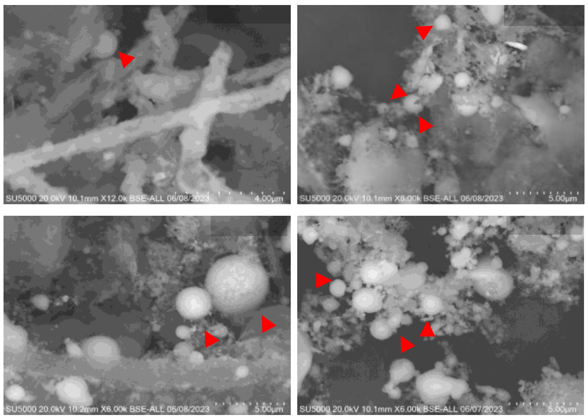

Figure 1
SEM micrographs of the 4 samples
Source: Own elaboration

As can be seen in the 4 micrographs corresponding to different synthesis times, indicated in the upper right part, several nanostructures with deformity in their upper layer are observed, but in all of them nanospheres of different sizes appear.

It can be confirmed that the amount of these structures and their shapes vary with time. For example, in the micrograph taken at 2.5 minutes, more nanotubes or nanowires than nanospheres are observed, unlike the micrographs taken at 10 minutes, where more nanospheres are present and no other form of carbons can be seen.

Another important aspect to highlight is the size of these nanostructures. In the case of micrographs taken at 2.5 minutes, it is observed that the nanospheres have an average diameter of about 380 nanometers at 1.1 microns. In the micrographs taken at 5 minutes, the nanospheres have an average diameter of about 980 nanometers at 1.68 microns.

For micrographs taken at 7.5 minutes, nanospheres with diameters of 4.66 nanometers to 1.31 microns are observed, while, at 10 minutes, the sizes vary from about 679 nanometers to 1.86 microns.

We conclude that the nanostructures become larger at higher synthesis time exposure and that the nanospheres can be defined as multilayered nanobeads, since they present not very smooth surfaces and some few uniform ones (Karthik,2014), moreover, they exceed the corresponding size to classify them among fullerenes and/or quantum dots (Georgakilas,2015. Yang,2023).

This is because as the synthesis time and therefore the temperature increases, there is more time for the carbon atoms to reorganize and form a larger and perhaps ordered structure (Zhao ,2005). The ceramic crucible used still plays a significant role in reaching a higher temperature in a longer time. However, it is important to keep in mind that the relationship between the synthesis time and the size of the nanospheres is not always linear, so it is intended to optimize the synthesis technique.

X-ray diffractometer

To corroborate the type of morphology of the nanostructures, an X-ray diffractometer brand X'Pert pro PANalytical with CuKα radiation λ=0.1542 nm was used. The plane references obtained yielded C60 and C70 fullerene-type carbon nanostructures, as shown in figure (2).

The presence of planes corresponding to these two types of fullerenes may be to the similarity that exists with nanorod-like nanostructures, due to the interactions with the outer layers of these, which usually resemble individual fullerenes (Zeiger,2023).

We can observe that the planes obtained in the sample at 2.5 minutes, have lower peak intensities and increases as the exposure time increases until reaching 10 minutes, only in the case of graphite peak (2θ=26°), the intensity decreases as the synthesis time increases, this variation in intensity directly influences the crystallinity, shape, size, and structural defects (Terohid ,2018). The obtained planes confirm the presence of spherical nanostructures, possibly of multilayered nanosheets type with impurities in their outer layer, previously seen in SEM micrographs.

Box 2

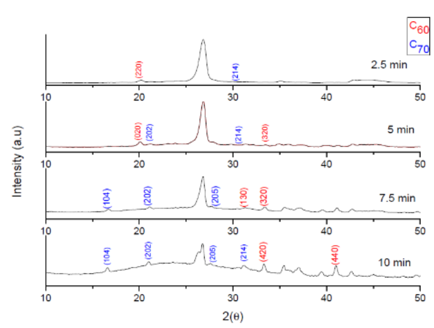

Figure 2
XDR of the 4 samples at different exposure times
Source: Own elaboration

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UV-Vis Absorption spectroscopy

Optical properties were observed using a Stellar et spectrometer with a Newport integrated sphere to obtain UV-VIS Figure 3. The absorbance of carbon nanostructures can be present in several regions of the electromagnetic spectrum, but for our analysis we will focus only on the electronic transitions π→π* (200 - 400 nm) and n→π* (400 nm -750 nm) (Orlandi,2002).

The absorbance spectra of the 4 samples present the absorption behavior of very similar nanostructures to C60 and C70 (Duarte-Ruiz,2021) which are very similar to nanorod responses.

In the region of electronic transitions π→π corresponding to the ultraviolet-visible UV-Vis region, repetitive responses were observed in the 4 samples, considering the most notable peaks at: 223,270,340,392 nm, this indicates the presence of nanostructures of different sizes (Duarte-Ruiz,2021).

In the region of electronic transitions n→π* corresponding to the visible region, the most remarkable repetitive peaks would be: 433,453,516 and 530 nm, indicating larger nanostructures. According to the literature, the similarity of responses of nanostructures such as C60 has strong absorption bands in the ultraviolet region of the spectrum, typically around 200 to 350 nm and the C70 shows strong absorption bands in the UV region (Duarte-Ruiz,2021).

With this absorption behavior of the graph, we confirm the presence of spherical nanostructures of different sizes visualized in SEM micrographs, more like nano-onions.

Box 3

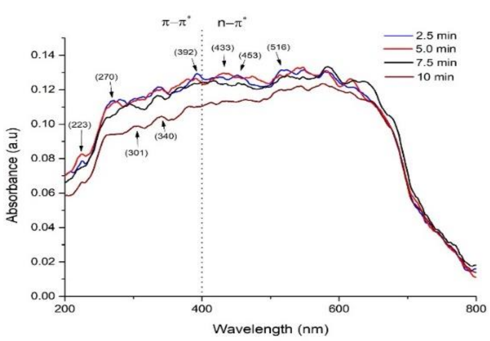

Figure 3

UV-vis absorption spectra of the four samples obtained

Source: Own elaboration

Optical band gap estimation

In Graph 4, the estimated behavior of the optical energy gap of the 4 samples obtained by the Tauc method from the analysis of the absorption data, Graph 3 is shown (Tauc,1968). It is widely recognized that this method assumes that the absorption coefficient is dependent on the photon energy (α) and can be expressed by the following equation [1]:

$$(\alpha - hv)^{\wedge}\left({}^{1}/_{\gamma}\right) = B(hv - Eg) \qquad [1]$$

where h is Planck's constant, ν is the photon frequency, Eg is the band gap energy, and B is a constant (Tauc,1968).

Box 4

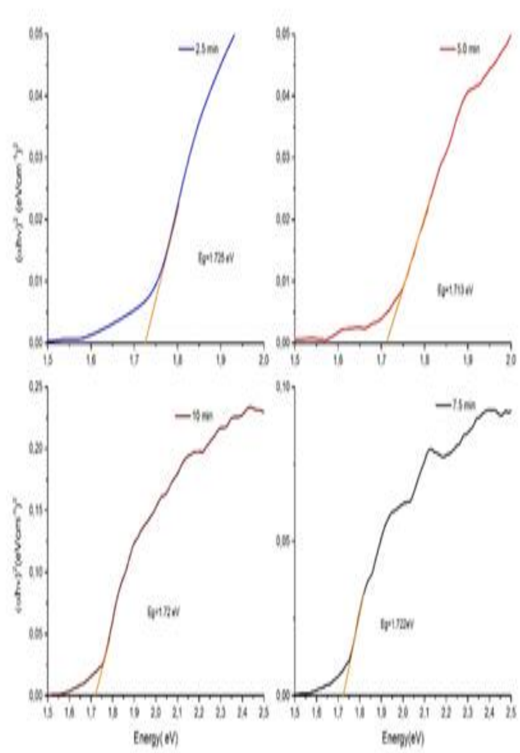

Figure 4

Optical band gap estimation of the four samples

Source: Own elaboration

It is observed in the graph that the band gaps vary very little, remaining on average at 1.70 ev. This value is consistent with the energy gaps of semiconductor powders, corresponding to unfunctionalized nanoparticles whose gap energy is around 1.6-1.7ev and corresponding to C60 and C70 carbon nanostructures (Rabenau, 1993).

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With respect to the behavior of the curves, it is evident that at temperatures above 2.5 minutes, they show a distortion in the linearity of the graph considered as extended tail, which confirms the presence of structural defects and impurities (Makuła et al.,2018).

These defects usually appear in materials that have suffered damage or modifications in their upper layer which causes additional energy states to be introduced (Makuła et al.,2018), causing the spectrum Estimation of the Optical Band Gap of the Four Samples having a similarity with the so-called Urbach tails (Migliorini,2022).

Only in the case of the time at 2.5 minutes, the graph presents a linearity without distortion, if we observe the micrograph, it is possible that it is because of the presence of more carbon nanostructure shapes (tubes, threads, spheres, etc.) and as the synthesis time was shorter, perhaps they did not obtain very little structural damage.

Conclusions

In this work, we presented the morphological study and the behavior of the optical properties of carbon nanostructures of the nanoshells, obtained by a simple and economic method, with short synthesis time variations. It was observed that the carbon nanoparticles with longer synthesis time present more structural damage in their morphology, and their optical responses showed no significant variations across different synthesis times.

We believe this work is pertinent because the method is feasible and easily reproducible, which can be optimized to produce nanoparticles with fewer structural defects for specific applications.

Declarations

Conflict of interest

The authors declare no conflict of interest. They have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this article.

Author contribution

Ordoñez-Casanova, Elsa G.: Contributed to the development of the project concept, research methodology, and experimental techniques, including the synthesis of carbon nanospheres.

Trejo-Mandujano, Héctor A: Contributed to the development of the project concept, research methodology, and experimental techniques, including morphological characterization using SEM and XRD.

Saucedo-Acuña, Rosa A: Contributed to the development of the optical analysis using UV-Vis spectroscopy and the Tauc method.

Villanueva Montellano, Alfredo: Contributed to manuscript writing, data interpretation, and the formulation of conclusions

Availability of data and materials

Data sets used or analyzed during the current study are available from the corresponding author upon reasonable request.

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Abbreviations

C60	Nanostructure composed of 60 carbon atoms
C70	Nanostructure composed of 70 carbon atoms
SEM	Scanning electron microscopy
XRD	X-ray diffraction

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