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Title: Synthesis and characterization of carbon-based quantum dots for use in Biotechnology

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INTRODUCTION

Quantum dots (QDs) are small semiconductor nanoparticles (NPs) of 2 to 10 nanometers (nm) in diameter. They are composed of various materials, including semiconductors, metals and carbon. They typically contain only 100,000 atoms and have a well-defined crystalline structure, allowing them to reveal unique optical and electronic properties (Tao et al., 2019).

They possess qualities such as electron confinement, energy quantization and the ability to absorb and emit light at different wavelengths depending on their composition and size. Furthermore, due to their tiny size, QDs suffer several quantum effects such as the discretization of their energy bands. Under these characteristics, QDs can interact with light and matter differently than materials on a larger scale; this is due to the quantum confinement effect, which occurs when particles are so small that electrons behave differently due to energy quantization. Quantum confinement occurs when the diameter of the crystal is smaller than its Bohr radius and influences the properties of the QDs to be quite different from those of macroscopic materials (B.H. Juárez, 2011).

An attractive property of these NPs is that they show confinement in the three directions of space; this is because the electrons are restricted to move in extremely small regions, less than 10 nm.

QDs can be considered nanocrystalline due to their crystalline structure and nanometric size; and since they are made up of semiconductor materials, they have a valence band (saturated with electrons) and a conduction band (empty energy band) separated by an energy difference called a gap.







INTRODUCTION

The luminescent process in quantum dots occurs through the emission of light when electrons relax from a higher energy state to a lower energy state. This process consists of four stages, excitation, relaxation, emission and recombination.

The excitation phase, the QDs absorb the energy of incident light, which excites the electrons in the valence band and leads them to a higher energy in the state conduction band, leaving gaps in the valence band.



The relaxation; electrons in the state of higher energy are relaxed toward the lowest energy state, releasing the excess of this in the form of light generated as radiative combination between the generated electrons and holes.



The emission phase, where the emitted light has a specific wavelength, which depends on the size and composition calculated by the separation between the two energy levels.



The recombination phase is reached. in which electrons and gaps recombine. releasing excess energy in the form of light (Cui et al., 2018).







BACKGROUND

Semiconductor quantum dots were discovered in 1980 by two independent groups, one in Russia by Alexei I. Ekimov in a glass array; and the other in the United States of America by Louis E. Brus and Alexander Afros who obtained them in colloidal solutions (Akimov & AMP; Anshchenko, 2023).

Currently, they are being researched for electro-optical applications such as photovoltaic devices (such as dyes absorbing sunlight), light-emitting diodes (already with commercial applications such as their use in QLED televisions, for example), photosensing, photocatalysis and bioimaging (as an alternative to traditional stains for fluorescent microscopy) (Bera et al., 2010; Kairdolf et al., 2013; Martynenko et al., 2017).

The range of applications for quantum dots is wide, however, there are concerns about the effect on health and the environment with the use and disposal of these materials which include metal ions such as Cd2+, Pb2+ as well as some non-metals such as As3-, Se2-, and Te2- considered toxic (Filali et al., 2020; Hardman, 2006). From this point lies the interest in less dangerous alternatives, such as carbon-based quantum dots (CQDs). These were discovered in 2004 when researchers purified the soot residue of arc flash by synthesizing carbon nanotubes and noticed unexpected fluorescent properties (Xu et al., 2004). Since that study to date, there has been great progress in the scientific community that seeks to replace inorganic quantum dots with carbon quantum dots that can provide similar properties with simple syntheses, at low cost, using widely available precursors, with easy waste management, lower toxicity and greater biocompatibility, for use in areas of medicine and energy mainly.





SYNTHESIS OF CQDs

The synthesis of CQDs tends to include a breakdown, polymerization, and carbonization of molecules. Normally this process occurs in some aqueous medium, so the final functional groups on the surface of CQDs are hydrophilic (Cayuela et al., 2016).

In the present paper the CQDs were synthesized using an organic precursor; piloncillo, this contains carbohydrates such as sucrose, glucose and fructose. This means that it has functional groups such as -OH and -CO; these groups can dehydrate at high temperatures, which is why it was decided to carry out this synthesis by ultrasound and microwave. This process by which synthesis is carried out is called sonication, and the type of chemistry used in this technique is known as Sonochemistry (Dong et al., 2013).









METHODOLOGY

The synthesis of quantum points of carbon was carried out using a green synthesis taking as a precursor the glucose from the piloncillo, a base of Sodium Hydroxide (NaOH), Hydrochloric Acid (HCl) and an ammonia base (NH3), thus obtaining a homogeneous solution (Figure 1).



Figure 1. Homogeneous solutions with piloncillo as a precursor and base of NH3 (right vessel), NaOH (medium vessel) and HCl (left vessel)

The sample is crushed with the help of a mortar until a fine powder is obtained from it, subsequently 3 solutions were prepared in which they were mixed at 1.0 M of the respective powder, using this unique concentration for the 3 different solutions with 30 mL of distilled water each. consequently they were stirred to obtain a homogeneous mixture with the piloncillo respectively. Once prepared the solutions with distilled water and the sample were subjected to the ultrasonic cube for a period of 30 minutes (Figure 2).



Figure 2. Solutions subjected to ultrasonic cleaning







The solutions were subsequently prepared with NaOH, HCl and NH3. For the NaOH solution, 30 mL of distilled water and one 1.0 M solution were used, for the HCl and NH3 solution, a combination of 30 ml of distilled water and a 30% V-V solution was used, and the solutions were shaken until they were completely diluted.

To complete this process, the homogeneous mixture of piloncillo is placed in a precipitated jar and the precursor solutions are added to it (Figure 3), again the mixtures are shaken for 15 minutes, subsequently exposed for 30 minutes to the ultrasonic cube and finally subjected to microwave for 7 minutes at a power of 10 Watts.



Figure 3. Precursor solution with homogenized base solution and NH3 (right vessel), NaOH (medium vessel) and HCl (left vessel)









RESULTS



Figure 4. CQDs with UV exposure







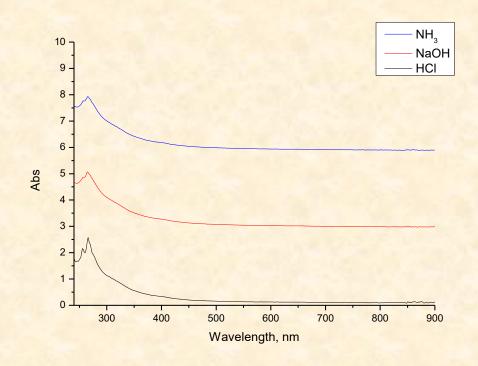
Uv-vis spectroscopy

The different solutions contained in the CQDs were exposed to ultraviolet light radiation, to visually check for luminescent properties. Figure 4 shows samples obtained from CQDs with and without ultraviolet radiation where the presence of luminescent properties is confirmed.

The aquatic solutions of quantum dots have their maximum excitation at 341 nm within the ultraviolet spectrum and an emission close to 442 nm inside the visible range in a cyan-blue color. From the spectroscopes it is observed that the intensity of luminescence depends on the increase in the concentration of organic material (Graph 1) until it reaches an over-saturation in concentration and has a decrease in intensity due to a phenomenon called cooling of concentration, on the other hand, the luminescent intensity will depend on the reaction time.

UV-Visible spectra show wide absorption at 280 nm (Graph 1) which is consistent with what is in various research (A. Mewada, 2013).

The quantum carbon dots have absorption bands at 250 and 280 nm, (Graph 1), which correspond to the bond and transition π - π * between carbon atoms C=C of aromatic domains, such absorptions are attributed to the n- π transition of the bands C=O and C = C. On the other hand, an absorption at 280 nm is observed indicating the presence of carbon nanoparticles.



Graph 1. UV-Visible Spectrum of Quantum Carbon Dots



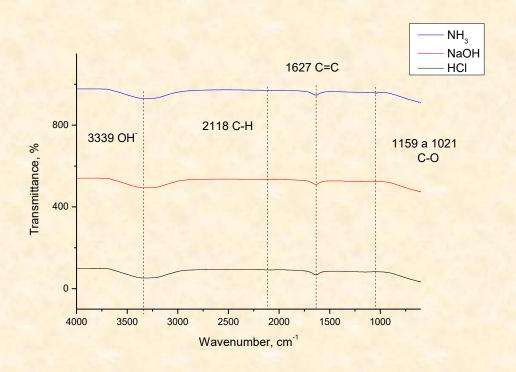
RESULTS



FT-IR spectroscopy

Infrared spectroscopy was used to identify functional groups present in the carbon quantum points of the representative samples. IR spectroscopes (Graph 2) show absorption bands present at 3339, 2118, 1627 and 1159 at 1021 cm-1, indicating the existence of functional groups OH-, C-H, C-N, C=C and C-O, C-OH, C -O-C, COOH, C = C (Valencia, 2019). From these results the quantum carbon dots obtained from piloncillo are composed of multiple functional groups which makes them highly soluble in water and makes them good candidates for their application in biotechnology.

Another variable to which the maximum intensity is attributed is to concentration; where at lower concentrations greater intensity, because at less matter, there is greater number of interactions for the formation of quantum dots. (Metha, 2014).



Graph 2. FT-IR spectrum of Quantum Carbon Dots





CONCLUSION

Quantum carbon dots were synthesized, with different solvents, obtained from piloncillo, which are cost-effective for their low cost of synthesis and environmentally friendly. Optimal emission conditions were found at a concentration of 0.1M in a time of 3 hours, presenting a maximum excitation at 280 nm, which will allow surface passivation for anchoring with biomolecules for application in biotechnology.

According to the FTIR analysis, the functional groups OH-, C-H, C-N, C = C and C-O, C - OH, C - O-C, COOH, C=C are identified as responsible for the functionalization of the surface that allowed the obtaining of luminescent properties (blue emission when excited by ultraviolet light with a wavelength of 254 nm).





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