Review Article

Exploring Quantum Dot Materials And Synthesis Methods: Current Trends And Future Prospects

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ABSTRACT

Quantum dots (QDs) have numerous applications in the fields of optoelectronics and quantum physics. Their emission wavelengths can be tuned based on size, they have high quantum yields and excellent photostability. These characteristics offer advantages over previous-generation materials such as organic dyes. Unfortunately, QD synthesis requires extensive precision and relies on processes that are difficult to replicate on industrial scales. This review summarizes the different materials and methods of QD synthesis in order to examine their current limitations and potential areas of improvement. It also emphasizes potential routes to improve the scalability of existing synthesis processes. As demand for QDs in optical applications continues to grow, an understanding of their manufacturing will be necessary to enable their continued development.

Keywords: Quantum dot; Nanocrystals; Semiconductors; Synthesis; Hot-injection; Microfluidics; Optoelectronics

INTRODUCTION

Quantum dots (QDs) are semiconductor nanocrystals that are small enough for their properties to exhibit quantum effects. Their electronic and optical properties make them useful for various types of optoelectronic devices (1). Today, they are used in display technologies like televisions, monitors, and smartphones. They also have the potential to increase energy intake in solar cells, improve the efficiency of medical x-ray technology, and more. The global market for quantum dots is currently valued at $10.6 billion USD and is projected to increase to $20.32 billion USD by 2032 (2). This evidently highlights the potential growth of QDs in various technologies and their relevance going forward in consumer products.

Understanding how to best exploit the properties of quantum dots requires an understanding of how they function. Electrons within QDs can be artificially excited from the valence band to the conduction band via an electronic source. It remains in this higher energy level before dropping back down into the valence band and emitting energy, usually in the form of different light wavelengths. This energy can be harnessed and manipulated, opening up enormous potential for the future of physics and chemistry.

Quantum dots have significant advantages over other materials that achieve similar goals. QDs have longer fluorescence lifetimes — on the order of picoseconds

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and nanoseconds — compared to organic dyes, which typically fluoresce on femtosecond timescales. For this reason, QDs have a higher quantum yield ratio of photons emitted to absorbed and are generally easier to study. A narrow excitation spectrum is one of the factors behind this occurrence; macroscopic materials emit at particular wavelengths based on factors such as their interatomic spacing, while QD emission wavelengths can be specifically tuned and adjusted by changing properties like crystal size (4). Additionally, many organic dyes do not maintain stable properties upon exposure to light due to their organic composition, whereas the synthetic composition of QDs permits them to be used in biological models without such sensitivity.

Synthesizing QDs is a complicated and tedious task that requires extreme precision. Regardless of the method, specific procedures must be followed to ensure safe and correct growth of these nanocrystals. Procedures must be started and stopped at specific times to facilitate proper crystal aggregation without interfering or hindering the chemical processes that ensue. This review compares different methods of synthesizing quantum dots, including their limitations, advantages, use cases and future applications, and describes each process. Summarizing these syntheses is important to understand how different QD materials are processed differently. This can unlock innovation in QD materials and synthesis, which can potentially mitigate the problems associated with them.

SEMICONDUCTOR QUANTUM DOTS

QDs composed of semiconductors offer the most versatility across their applications. They can be tuned so that their emission spectra lie within the visible and infrared range (5). A phenomenon known as quantum confinement, where the behavior of particles such as electrons change due to confinement, results in quantized (discrete) energy levels, allowing for this manipulability (3). The energy gap, or the amount of energy required to excite an electron within a certain material, increases as the size of the nanoparticle increases. Size-dependent energy gaps also change the absorption and emission wavelengths of the material, altering the optical properties of the quantum dot. This attribute makes them extremely useful in systems and semiconducting solids, which require extremely precise energy outputs. Core-shell QDs are a variation on this nanocrystal — in these QDs, the core is coated with different transition metal layers, such as cadmium (Cd), selenium (Se), lead (Pb) and zinc (Zn), indium (In), and arsenic (As) (5). Adding layers enhances the QD quality by protecting it against external damage from phenomena such as corrosion, improving quantum yield and widening the range of emission wavelengths (5). The transition metals are ionically bonded to anions in a crystalline or lattice structure. The molecular shape of the QD can differ depending on the composition of the core and ligands as well as the types of bonds connecting these two parts. This is frequently done through a process known as 'surface passivation' which involves the careful manipulation of the QD structure in order to enhance its properties to extend shelf life. For example, integrating organic ligands into a QD could reduce electron and hole trap states, which lower fluorescence emission.

Cadmium alloy-based QDs, such as CdSe, CdS and CdTe, are the most commonly used for research due to their exceptionally high quantum yield of over 50% (7). They usually emit light between wavelengths of 460-645 nm, making them ideal for observation and imaging purposes.

Semiconductor QDs have been shown to be more effective contrast agents than the standard gadolinium in magnetic resonance imaging (MRI) (6). This results in clearer images and, in turn, more accurate diagnoses. Semiconductors, due to their naturally longer lifetimes, could offer a more cost-effective alternative to fluorescent dyes, which decay much faster and have to be replenished on a more frequent basis.

However, multiple concerns have been raised regarding the toxicity of semiconductor QDs. The cation used is often a heavy metal such as mercury (Hg), lead (Pb) or cadmium (Cd), which poses numerous challenges. The coating used to protect these QDs can often wear off, revealing the more toxic core (8). Use of these metals within biological systems can cause significant harm in vivo, as they can last for longer periods of time without an established method of safe removal. Harmful effects include, but are not limited to, unwanted DNA mutation, organ failure, and irreversible tissue damage (8).

METALLIC QDS

Metallic QDs differ from semiconductor QDs since their conduction and valence bands exhibit continuous overlap, allowing for a constant supply of electrons. Because metallic lattice structures have a "sea" of free-flowing electrons between them, metallic QDs exhibit plasmonic properties; the electrons often oscillate and cause a phenomenon known as localized surface plasmon resonance (LSPR), making them ideal for sensing and imaging (9). Semiconductor QDs, due to their band gap, are not as efficient conductors of electricity, so they do not exhibit this plasmon resonance. However, metallic QDs are often categorized under semiconductor QDs because pure singular metal nanocrystals are difficult to manufacture and stabilize.

Metallic QDs are often incorporated into high resolution displays and liquid crystal panels. Their conductive properties make them ideal candidates for use in light-emitting diodes and solar energy conversion (10). Unlike semiconductor QDs, they possess a wider emission spectrum, which is potentially more useful for situations that require varying spectra.

The properties that make precious metals such as gold and silver ideal for usage in electronics also make them prime candidates as building blocks of specialized quantum dots. Aside from its high conductivity, gold is resistant to corrosion while being quite malleable, making it durable and fairly easy to manipulate during manufacturing (11). These properties ensure that quantum dots can be utilized in harsher conditions.

However, being heavy metals, gold and silver tend to be extremely toxic for in-vivo uses, and accumulation within living organisms can have fatal complications. Financial costs and processing obstacles pose further threats since these rare-earth metals are quite expensive and safe processing involves proper management of waste, another economic challenge (10).

CARBON QDS

Carbon QDs provide their own unique set of advantages. They are typically made from quasi-spherical carbon nanoparticles, with a core of sp^2 and sp^3 hybridized carbon atoms and a shell of functional groups such as hydroxyl, amino, and carboxyl groups. Typically, electrons remain within the s, p, d, or f orbitals of an atom. However, the orbital overlap in hybridized atoms facilitates stronger and more precise bonding, leading to structured electron distribution and improving the properties of the QD (12). Unlike with other types of quantum dots, the exact composition of these QDs depends on the synthesis method used, which also impacts the properties of the produced QD.

The primary benefit of using carbon over metals is its biocompatibility and widespread availability. Being a significantly less toxic material than heavy metals, it can be easily integrated within biological systems without repercussions (28). Use of an organic material enhances the ability to deliver drugs and opens up a new domain of prospective nanomedical technology. For example, Chiu *et al* leveraged this biocompatibility to design carbon QDs for a dual chemotherapeutic and in vivo fluorescence bioimaging (29). Additionally, variations of carbon structures, such as graphene, carbon fibers, and sheets, are shown to be resistant to pH changes. This makes these QDs effective buffers in largely acidic or alkaline conditions. In many situations, carbon quantum dots have been shown to exhibit similar or higher levels of photostability and emission spectra range as organic dyes or fluorescent material (30).

Carbon QDs have the ability to act both as photosensitizers and energy donors for other photosensitizers (13). Photosensitizers are light-sensitive catalysts, with numerous applications in polymer and materials chemistry. This light-triggered activity can be leveraged in photodynamic, or light-based, therapy to combat cancer and numerous other illnesses. Their high solubility in water also allows for extremely efficient drug delivery (31).

HOT INJECTION

Hot injection is one of the most common methods of quantum dot synthesis. It is known as a "bottom-up approach" in which nanoparticles are formed by injecting precursor molecules into a heated solvent.[14] This method has multiple advantages over others, the primary one being the manipulation of quantum dot nuclei growth via a process called nucleation. Factors such as chemical concentration and temperature can be carefully controlled throughout the experiment to control the size of each quantum dot, which in turn determines its optoelectronic properties (15). Additionally, the nucleation process can be completely separated from the growth stage, allowing for even more precision when adjusting the QD sizes and compositions. This method is often known as organometallic synthesis due to the presence of both organic molecules and metals within the material.

In an experiment conducted by Mahajan *et al*., $Cd(CH_3)_2$ is mixed with elemental Se into a trioctylphosphine solution to form CdSe QDs, where dimethylcadmium is the organometal,a molecule consisting of at least one metal-carbon bond, and trioctylphosphine (TOP) acts a stabilizing agent with a high boiling point of around 320°C (14). In certain situations, octadiene can be replaced for TOP and cadmium oxide could replace dimethyl cadmium to form different compounds.

However, it is necessary to address the limitations that are associated with hot injection (Figure 1). The high temperatures used in hot injection pose a problem for larger-scale implementations. Energy consumption for maintaining these conditions will surely become an issue, and efficiency will be a leading problem in the use of hot injection (15). The question of concentration gradients also arises with the injection of the precursor. Due to its cool temperature relative to the solvent, injection will likely cause clumps or uneven particle distribution throughout the container, leading to more potential problems (16). These include, but are not limited to, varying size distributions, reduced fluorescence, and decreased electron transfer. All of these impact the performance of QD applications, which require consistency. Finally, the dimethylcadmium has been found to be extremely toxic, and hot injection often has numerous byproducts. Disposal of these chemicals needs to be done in a safe and efficient way, which hinders it from being used at larger commercial scales (16).

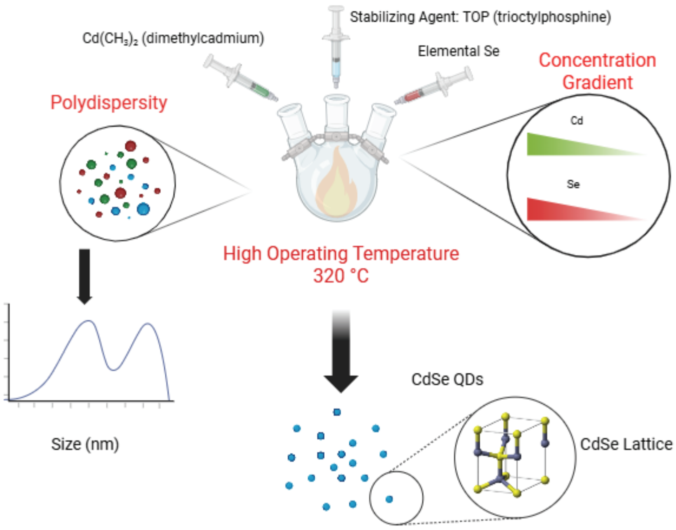

Figure 1. Limitations of hot injection QD processing. The concentration gradients in reactants result in polydispersity, while the high processing temperatures limit scalability. However, hot injection has been successfully used to generate QDs for many chemistries, including CdSe.

MICROFLUIDICS

Microfluidics involves the study and manipulation of fluids and particles within microtubes, which range from tens to hundreds of micrometers wide (17). Different pump systems regulate the flow of fluids within these channels and are controlled by intricate computer chips or simple syringe pumps. The utilization of microfluidic reactors is another example of a "bottom-up" approach which separates the nucleation and growth stages quite effectively. Figure 2 highlights the benefits of microfluidics over hot-injection processes in multiple aspects. They allow for the formation of more complex QDs and provide better quality assurance due to high levels of controllability (17).

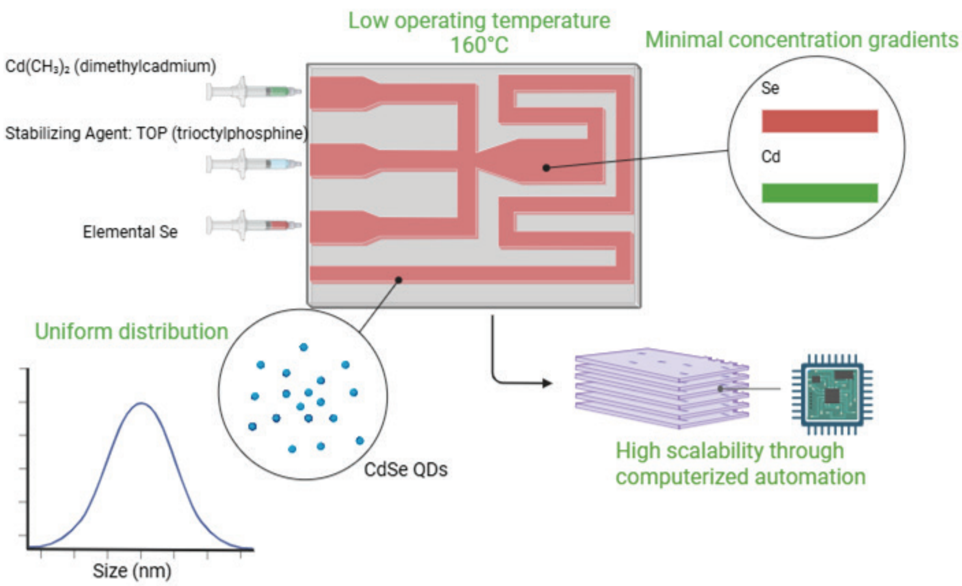

Figure 2. Advantages of microfluidic reactors. Channels provide a relatively uniform particle size distribution, and are easily scalable through computerized automation. Concentration gradients are significantly minimized.

Microfluidics consist of three primary subcategories: continuous flow laminar reactors, droplet based reactors, and segmented flow reactors (17). All involved inlets where various precursor solutions are injected, but mixing processes and end results differ greatly. The size of the QD depends on the reaction time, which can be carefully controlled through integrated computer systems. Due to large surface area to volume ratios and short diffusion distances within the channels, microfluidic reactors have high heat and mass transfer capabilities (18).

Precursor solutions move along microtubules in a smooth laminar pattern within continuous flow reactors, allowing for equal mixing and higher control over reaction temperatures. Microfluidic reactors generally operate at around 160°C, a lower temperature than the 300°C needed for conventional batch reactors. The laminar flow is easily adjusted and allows for a safe but efficient approach towards QD synthesis (18).

In droplet reactors, the majority of the fluid is contained within microdroplets. A key advantage with this is that it minimizes the risk of clogging the tubes and does not interfere with the flow. This offers a unique way to form QDs because reactions can be isolated to each microdroplet and its kinetics or reagents can be studied. However, not all materials are compatible with this design.

Unfortunately, despite the energy efficient design of microfluidic reactors, they are not scalable to industrial levels due to the unique design of the small channels. However, it is important to note that multiple smaller reactors can be used in parallel, and this is financially reasonable in certain cases. For example, the recent mRNA based SARS-CoV-2 vaccine utilized this technology for mass production in a relatively short time frame (32). Additionally, viscous drag is a major problem and causes a spread of differing fluid velocities in the channel (19). In turn, this leads to polydispersity, or a large variation in particle sizes, which is an unwanted outcome. Nonuniform QD sizes lead to different optoelectronic properties, which is not suitable for industrial purposes.

BIOSYNTHESIS

Biosynthesis offers a more economical and less toxic approach to QD synthesis. It is a bottom-up approach where source material is composed of bacteria, microbial strains, fungi and other organic resources (20). This method is particularly good for medical and biological applications as the minimal use of metallic compounds and/or rare earth metals decreases toxicity and cost significantly. Biologically synthesized QDs have higher resistance to photobleaching, which is beneficial for in-vivo and in-vitro imaging, and studies have shown that this characteristic is extremely helpful for cancerous tumor imaging (20). Furthermore, biosynthesis uses enzymes, proteins, and peptides, natural agents that act to prevent agglomeration and maintain a relatively uniform size and shape distribution.

TOP DOWN

Laser ablation is a top-down method that involves starting with extensive material, and then physically removing material in order to reach the desired QD size (21). The use of a laser is extremely advantageous for heavy metals, which require the precision and strength of a laser, but also prevents any foreign particles and impurities from entering the QD synthesis process (21). The homogeneous nature of the particles is maintained, and uniformity is relatively consistent.

Electron beam lithography is another similar method that essentially “carves” out the necessary material but uses a beam of focused electrons in order to “draw” specific molecular shapes. This permits the fabrication of more complex shapes and specific tuning for certain properties (22).

High energy ball milling involves using mechanical energy to physically grind down larger particles into fine powders occasionally with the aid of a solvent (23). This method is suitable for larger scale production, because these machines can run continuously and in batches.

THE FUTURE OF QUANTUM DOTS

QDs have significant potential in numerous scientific applications, but their synthesis comes with various drawbacks that need to be addressed. Key issues consistently present in many types of synthetic procedures include toxic waste formation and scalability to large volumes required for commercial manufacturing.

Unfortunately, owing to the extensive use of heavy metals in QDs, waste management is still an issue. “Doping” is an emerging technique that takes a more ecological approach to QD synthesis: this process imparts certain properties onto semiconductors by adding impurities during the synthesis (24). It utilizes nitrogen and carbon, which are found in biomass and biological waste, thereby lessening the environmental impact with regards to the extraction of raw material. Furthermore, it replaces heavy metals in the atomic structure of the QD, so that less of the toxic material is used. The use of "N-doping" — the introduction of nitrogen into the QD synthesis process — also enhances the capabilities of the QD. The addition of nitrogen allows for higher fluorescence intensity and increased solubility in water due to the introduction of nitrogen allowing for polar functional groups to be formed between water and QD molecules, resulting in higher levels of dispersion. It also permits the bandgap to be changed based on the amount and method of nitrogen introduction (25). This allows for greater flexibility when controlling the photoelectric attributes of the QD.

As of today, the only viable solution is to substitute the precursors with 'greener' alternatives, which would generate lower quantities of toxic impurities, or make the waste more processable. For example, Pu *et al* substituted trioctylphosphine oxide (TOPO) with octadecene (ODE), resulting in almost no loss of final QD properties (15).

An upcoming example would be QDs made of indium phosphide or those made up primarily of sulfur or phosphorus (26). Fabrication of QDs using these materials would be more cost-efficient, thereby allowing for more widespread use. Leveraging a greener organic precursor to synthesize these QDs would also help alleviate some of the environmental concerns associated with QD synthesis. These molecules would have significantly lower melting points, as covalent bonds – the bonds typically found in non-metallic compounds – have lower melting points than metallic bonds (26). This further decreases the need for high temperatures and potentially allows for larger scaling, as extreme energy demands are unlikely to be replicated within industrial corporations.

High temperatures, specific timescales and precise control are all factors that limit mass production of QDs. Higher temperatures in much larger vessels have a higher likelihood of inconsistency, leading to temperature and concentration gradients. This in turn results in an uneven size distribution of QDs, which is not ideal for mass production. Also, the timescale is hard to manage in industrial settings due to the expensive technology required, as nucleation is often controlled to the millisecond or nanosecond. One again, size control and uniformity are essential to maintain the properties of the QD, specifically, the bandgap (26). This further decreases the need for high temperatures and potentially allows for larger scaling, as extreme energy demands are unlikely to be replicated within industrial corporations.

Microfluidic reactors have shown significant potential in the realm of QD synthesis. Considerable numbers of these channels can be controlled simultaneously through computerized chips, allowing for larger production of these QDs (27). Additionally, the 160°C temperature threshold is significantly lower than the conventional hot-injection temperatures of ~300°C, making it much more feasible.

CONCLUSION

The synthesis methods of QDs offer a wide array of possibilities in fields ranging from optoelectronics to nanophotonics and beyond. However, more research is needed to develop methods that can scale to commercial levels. As these advances continue, quantum dots are expected to play a key role in various fields from solar energy to medical imaging, making their future both exciting and transformative. As various economies and markets expand, the demand for QDs continues to grow in a society where technology is becoming more and more necessary.

DECLARATION OF CONFLICT OF INTERESTS

The author(s) declare that there are no conflicts of interest regarding the publication of this article.

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