

A Critical Analysis on Green and Low-Temperature Methods for the Production of Carbon Nanoparticles

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Carbon nanoparticles (CNPs) gained a growing attention for a wide multiplicity of applications. CNPs offer the basic advantage of being well tolerated by living organisms and they are much more environmental friendly than many other inorganic NPs for analogous purposes. In this short review, several techniques are described and compared, enlightening their strengths and drawbacks in terms of easy of deployment, possibility of transfer on a large scale, plant safety and environmental soundness. The latter aspect is now one of the basic paradigms for the chemical production tout court and this target can be achieved by acting on different fronts, namely energy saving, cleaner processes and reagents substitution. Some techniques are still confined to a laboratory scale and they need deeper investigations before being transferred to an industrial production, while some other have reached a technological maturity to make them competitive on the market. The methods for the synthesis of CNPs have been grouped according to a scheme different from the usual classification in top-down and bottom-up processes. A greater emphasis has been given to the simplicity of unit operations and to the corresponding energy burden.

1. Introduction

Nanotechnology has assumed an emerging role in many aspects of science and technology, thus creating new interdisciplinary links between Materials Science, Physics and Engineering [Reverberi et al., 2018]. Nowadays, many nanostructured objects have common applications in real life, owing to the unusual physico-chemical properties of nanostructured materials with respect to a bulk solid having the same chemical composition. The generally adopted definition of nanoparticle (NP) in physics literature refers to a generic particle having diameter <100 nm. In the specific case of CNPs, as a first rough classification according to their size and properties, they include carbon nanodots (CND) and carbon quantum dots (CQD) whose average diameter is smaller than 10 nm in both cases [Rahmani and Ghaemi, 2019]. Many review papers exist, with extended and thorough discussions about the applications of CNDs and CQDs in biology [Wang and Qiu, 2016], biomedicine [Hola et al., 2014], optoelectronics, imaging [Li et al., 2020], catalysis [Sharma and Das, 2019], sensing [Pan et al., 2019], energy harnessing [Khodabakhshi et al., 2020] and environmental decontamination [Kalantari et al., 2019]. The impressive growth of research about CNP is mainly related to its good biocompatibility with mammalian cells in vitro with respect to other inorganic NPs, even though the studies concerning toxicity in vivo are somewhat limited in number. For these reasons, the safety restrictions to be fulfilled during the manufacturing process of carbon-based nanomaterials are far less stringent than ones typical of inorganic compounds or elements [Fabiano et al., 2019], with a considerable improvement in attractivity and sustainability of the global process [Pasman and Fabiano, 2021].

The methods adopted for the synthesis of such CNPs may be based on a process of disaggregation of a starting bulk material made of elemental carbon or by particle nucleation and grow from carbon-containing molecules. In the first case, CNPs are produced by purely chemical, electrochemical mechanochemical or laser-assisted etching of the precursor [Sharma et al., 2018]. Such technique is generally affected by some

drawbacks, as the process requires a relatively long time and the CNPs tend to keep memory of the physical characteristics of the pristine material [Sciortino et al., 2018]. On the opposite, the latter technique, corresponding to bottom-up processes, allows obtaining of a wide variety of carbon-based NPs and it is generally preferred in case of highly specific end-uses of the CNPs, as in cases of sensors and detectors for cations or polluting agents [Sivasankarapillai et al., 2020]. The relevant process of synthesis relies upon carbonization of organic molecules, according to different techniques like chemical dehydration, thermolysis, microwave heating, pyrolysis, plasma-assisted, ultrasonic-assisted processes, which can be carried out in solid, liquid or gaseous phase.

Bare CNPs, that is non-functionalized carbon nanoparticles, have scarce properties in optoelectronics as photoluminescent materials, but there are exceptions [Lim et al., 2015]. According to the excellent survey by Chen et al. [2019], functionalization may be realized by heteroatom doping and surface modification. The former is usually based on a precursor containing atoms other than carbon, like non-metallic dopants and metallic dopants. The latter is usually based on specific ions that form coordinate binds with electro-donors located at the CNP surface. Surface passivation, namely a surface treatment with agents like polymers protecting the surface from contaminating compounds, can be considered itself as a form of functionalization, often beneficial in terms of quantum yield (QY).

Many precursors can be used as a carbon source; among them, several organic wastes as by-products of industrial activities have been proposed. In this context, the production of CNPs represent a successful example of waste recycling and conversion into a valuable and high-value material. Sharma et al. [2017] proposed an exhaustive summary concerning the synthesis of CNDs from food and bakery products, vegetables, spikes, beverages and even from animal and human derivatives as carbon sources. This trend is consistent with the recent paradigms of green and eco-friendly nanotechnology [Reverberi et al., 2017], which are progressively getting more and more stringent owing to the recent targets of multiobjective optimization, where energy saving, risk minimization and reagent substitution are now essential goals. On that note, it is worth reminding that functionalized CNPs may require multistep processes, with a considerable waste in energy, high fixed costs of apparatuses and safety concerns related to human and environmental protection. Consequently, recent efforts are directed to combine one-pot and low-energy synthesis processes, nevertheless maximizing the target properties of the final product according to its end-uses. As an example, minimization of photocorrosion is one of the primary targets for a photocatalytic material, while it has a different role in a photoluminescent material. For the above reasons, this short review paper has been organized adopting a scheme somewhat different from the aforementioned typical and well-known classification of top-down and bottom-up methods. Here, instead, the techniques are grouped according to a different target, namely depending on a plant operation scheme, on thermal conditions and energy requirements needed for the synthesis of CNPs. This choice is instrumental to evidencing the combined trends underlying the current research in CNP synthesis.

2. One-pot chemical methods for CNPs synthesis

According to this scheme, the functionalization is achieved by mixing a precursor with a compound acting as a donor of a doping heteroatom entering the inner structure of the CNPs. Non-metallic heteroatoms like S, N, F, P, B and Si are adopted more frequently than metallic dopants. In many cases, the doping element is already present in the chemical structure of the precursor itself, thereby making unnecessary the presence of a donor molecule mixed with the precursor. This rule is valid also for oxygen, obviously present in carbohydrates as usual precursors.

Hydrothermal carbonization is a typical and high-yield process adopted in this context. In some cases, the heat is supplied from the outside by an autoclave (exogenous heat), allowing to reach temperatures even exceeding 200° at a price of higher equilibrium pressures. The reaction time is widely variable, spanning from 5min to several hours in case of exogenous heat, while it is generally shorter in case of endogenous heating. Zhang et al. [2010] were among the first in obtaining fluorescent CNPs of 2 nm diameter by heating ascorbic acid in autoclave at 180°C for 4h using both water and mixtures of water with different alcohols. Despite the absence of heterogeneous doping elements mixed with the precursor, the QY was unexpectedly high in water-ethanol medium and this phenomenon was ascribed to a possible double effect of capping and surface functionalization, related to oxygen-containing groups. Likewise, Shen et al. [2013], using several carbon sources containing both sulfonic and amide groups, prepared functionalized fluorescent CNPs of 5-30 nm diameter at 140°C in autoclave for 12h using water in all experiments. Their investigations by Fourier transform infrared spectra (FTIR) showed that carboxyl =C=O and hydroxyl –OH groups were detected at the surface of the CNPs even in the absence of such groups in the precursor, thus proving that hydrothermal carbonization triggers such kind of functionalization. Rahmani and Ghaemi [2019] performed a one-step green synthesis of N-doped CD starting from gum tragacanth mixed with ethylene diamine as N source by

hydrothermal carbonization at 180°C in pressurized vessel for 10h. The particles were particularly small, with diameters in the range 1-2.5 nm, and they exhibited a strong fluorescent emission with a maximum QY of 66.74%. Such value changed according to the pH of a dispersing solution and to the wavelength of the exciting source. The as-prepared CD were tested as detectors for Au³⁺ ions by fluorescence quenching and the relevant selectivity was investigated in the presence of other 20 common cations. Hydrothermal dehydration in strongly basic environment is less common, but an example is offered by a study on benzoxazine dissociation at 200°C in water containing NaOH for the synthesis of N-doped CNPs used as Cr(VI) sensors by fluorescence quenching [Fang et al., 2018]

In hydrothermal carbonization by endogenous heating, the thermal effect comes from an exothermic reaction triggered by dehydrating agents almost always having acid properties like H₂SO₄ and P₂O₅. This process is generally carried out at atmospheric pressure and hence it does not require expensive pressurized vessels. By the way, Zuo et al. [2014] obtained fluorescent CNPs in a simple process by mixing a solution of glucose, as a carbon source, with P₂O₅. The temperature (100°C) and the reaction time (10 min) were considerably lower than required in case of exogenous heating. Other authors used variants of such a self-heating technique and obtained functionalized CNPs by adding water to a solid mixture of a N-S-bearing precursor with P₂O₅ [Yang et al., 2014], or adopting acetic acid as liquid precursor subject to dehydration with the same carbonizing agent [Fang et al, 2012]. Interestingly, the heat released made the solution boil and the bubbles promoted the formation of hollow or solid CNPs depending on the heat capacity of the initial mass of water, thus acting as a tuning parameter for particle shape.

In solvothermal carbonization, the embedding medium is generally represented by high-boiling liquids like glycols [Uriarte et al., 2019], organic acids or other solvents that, in some cases, may take part to the functionalization of CNPs. In analogy with hydrothermal carbonization, the heat may be exogenous or endogenous. When a microwave heating is adopted, glycols offer the advantage of having a high dielectric constant, making them good absorbents for microwaves, thus allowing short reaction times which, joined with a high viscosity, have a positive effect in hindering particle aggregation. These conditions were met in the thermolysis of cystine in glycerol to synthesize CDs useful for the detection of Hg²⁺ ions in water by enhanced fluorescence intensity ("turn-on effect") [Xu et al., 2018]. In a different scheme of synthesis [Estes et al., 2019], glycerol may serve directly as precursor subject to endogenous dehydration carried out by an excess of concentrated H₂SO₄ at different reaction temperatures. The physicochemical properties of the CNPs varied considerably with the carbonization temperature, having also influence on the distribution of –SO₃H and –COOH functional groups in the final product. Likewise, oleic acid proved to be a useful solvent when cysteine and citric acid as precursors were carbonized at 220°C for the synthesis of N-S doped CNPs further adopted as probes to detect trinitrophenol in water [Lai et al., 2020].

A single-step flame synthesis of CNPs activated by heteroatoms can be carried out by sooty combustion of organic precursors containing the same heteroatoms. Namely, the soot produced by combustion of pyrrole proved to be a valid precursor in the synthesis of N-doped CNPs for the realization of electrochemical sensors and electrodes [Karikalan et al., 2016].

3. Low-temperature CNPs synthesis

The temperature of CNPs synthesis is a basic parameter whose values may deeply condition the energy demands and the safety issues of the processes devoted to the CNPs synthesis. For this reason, the present section has the goal of surveying those processes carried on at T≤100°C. This threshold has been assumed taking into account the temperature values of the processes listed in the review of Sharma et al. [2017]. Of 65 works concerning CD synthesis from green precursors, only 8 have been carried out at T≤100°C.

3.1 Hydrothermal/solvothermal carbonization

Very few chemical processes operating at room temperature are efficient in synthesizing CNPs or CD with a good conversion, owing to obvious chemical kinetics constraints. It is intriguing to note that strong bases like NaOH allow realizing a carbonization in a single step operated at temperatures considerably lower than those typical of acid drying agents considered in the previous section, but there are exceptions [Gong et al., 2015]. On this matter, Feng et al. [2016], obtained CNPs adding NaOH to a solution of trypsin and dopamine at room temperature. The product was tested as sensor to selectively detect Fe³⁺ ions. Other authors [Reverberi et al., 2020] carried out a thermal dissociation of different sugars by NaOH-driven hydrothermal carbonization. Fructose and lactose underwent carbonization producing CNPs even at T≤50°C, while polyalcoholic sugars like mannitol or erythritol remained undissociated in the same operating conditions. Another successful use of basic dehydration was proposed by Tegafaw et al. [2018], who synthesized amorphous CNPs at T=90°C from dextrose as precursor in aqueous medium containing NaOH. An accurate characterization was performed and

the FTIR absorption spectrum revealed the presence of C=C bond, absent in the precursor, together with hydroxyl and carboxyl groups explaining the strong hydrophilicity of the as-prepared CNPs.

3.2 Non-thermal plasma-assisted carbonization

Non-thermal or “cold” plasma, namely a non-equilibrium plasma where the temperature of electrons is different from the temperature of ions by many orders of magnitude, represent an important technique to carry out chemical reactions in gas or liquid phase without reaching bulk temperatures typical of conventional pyrolysis processes. This type of plasma is generally realized by means of a low-current high-voltage discharge between electrodes located in a gas stream or in a liquid phase containing a precursor subject to dissociation. The electric potential, usually set in a range of 1-60 kV, must be high enough to break the dielectric strength of the embedding phase. The energy transferred to electrons breaks the chemical bonds of the carbonaceous precursors, with formation of unsaturated intermediates and finally producing CNPs.

When the process is operated in gas phase, the precursor (feedstock gas) is mixed with a carrier gas (buffer gas) before entering the zone crossed by the electrical discharge [Moreno-Couranjou et al., 2009]. The CNPs are gathered in a chamber located downstream the plasma reactor. According to this technique, Lu et al. [2020] using a mix of hydrocarbons/hydrogen in different proportions as feedstock gas and argon as carrier gas at room temperature and atmospheric pressure, obtained CNPs of different geometries like spherical CNPs of 20-40 nm and graphene nanoflakes. Wang et al. [2020] discussed the role of inert and reactive buffer gases like Ar, He, H₂ and N₂ in cold-plasma dissociation of CH₄. They pointed out that the chemical characteristics of the buffer gas may strongly influence geometry, crystallinity, average diameter and inner defects of the CNPs. This aspect was particularly evident in the case of reactive gases like N₂, acting as a dopant in carbon structure with formation of C-N bonds detected by Raman and by X-ray photoelectron spectroscopy (XPS). For this reason, this low-temperature one-step technique appears to be a valid alternative to other standard thermal methods in doping CNPs.

In liquid phase, a cold-plasma dissociation is operated by dipping the electrodes, at which a high voltage is applied, within a carbon-containing liquid precursor. The latter consists of organic compounds having a high dielectric constant, needing a tiny distance in a range of 0.5-15 mm between the electrodes, generally made of tungsten wire. In this way, Panomsuwan et al. [2015] synthesized fluorine-doped CNPs using a toluene/trifluorotoluene mix in different proportions by a one-step plasma process where the temperature of the precursor never exceeded 35°C. A XPS analysis of the solid phase led them to the conclusion that the C-F bonds have a covalent, semi-ionic and ionic configuration, whose balance depends on the global fluorine content. Other examples exist where heteroatom-doped CNPs have been successfully produced, as in the study of Li et al. [2016], who carried out a cold plasma-assisted dissociation of pyrazine and acrylonitrile, forming N-doped CNPs with diameters of 24.5-35.4 nm. Seangarunthong et al., [2017] prepared CNPs with primary particles diameter in a range 20-30 nm by a solution plasma process (SPP) where a mixture of exhaust motor oil and benzene at various concentrations underwent dissociation at room temperature and atmospheric pressure. Intriguingly, it was observed that the resulting CNPs tend to shift toward a graphitic structure with higher crystallinity in the presence of an aliphatic fraction, namely for growing motor oil content in the precursor, consistently with previous investigations of Morishita et al. [2016]. A drawback common to many SPP is the presence, often revealed by XPS analysis, of small amount of tungsten carbide (WC) in the as-prepared CNPs. This compound presumably derives from etching and further reaction of tungsten electrodes with active carbon compounds present as intermediates during the carbonization steps. Cold plasma dissociation of alcohols offers interesting insights concerning a wide possibility of tuning and controlling both the characteristics of the CNPs and the composition of gases produced during the carbonization. This is the view adopted by Zhou et al. [2020], who dissociated ethanol by a non-thermal plasma in liquid phase, obtaining both CDs of 3 nm average size and gaseous products whose composition varies depending on the power delivered to the spark. For power limited in a range of 4-6 W, the released gases contain more than 90% of hydrogen and this finding may open challenging perspectives for a practical utilization of this process in the production of H₂ with competitive costs with respect to standard industrial technologies. The versatility of such ethanol carbonization by cool plasma was previously investigated in the paper of Xin et al. [2018], where the size of CNPs varied from 1180 nm to only 10.7 nm by increasing the discharge power by only 20 W.

4. Conclusions

The most important aspects discussed in this short review can be summarized as follows:

- The current trend in CDs and CNPs synthesis is addressed to methods aiming at reducing both the number of unit operations, their times and costs in terms of energy demand.

- The use of toxic and noxious compounds, often adopted in the production of inorganic NPs used in semiconductor technology, is drastically reduced in the manufacture of CNPs, with obvious advantages in plant safety and environmental protection.
- The recent low-temperature processes in CNPs manufacturing represent a challenging aspect of research, owing to their multipurpose applications in nanotechnology and hydrogen recovery.

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