Post-synthesis heating, a key step to tune the LPR band of gold nanorods covered with CTAB or embedded in a silica shell

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Received: 27 April 2022 / Accepted: 29 July 2022 / Published online: 8 August 2022 © The Author(s) 2022

Abstract

The transitioning of nanotechnology from laboratory to industrial-scale manufacturing poses various challenges in nanoparticle realization. From this perspective, beside the conventional synthetic procedure, based on the seed-mediated growth approach, a reshaping thermal strategy has been investigated to improve the control on gold nanorods aspect ratio, with the aim to point out a potential and encouraging way to better manage the scalability and reproducibility of nanoparticles. For this purpose, nanorods covered with CTAB and nanorods enclosed within a silica shell of tuned thickness have been synthesized and submitted to a post-thermal treatment at various temperatures, up to 300 °C for CTAB recovered gold nanorods (AuNR@CTAB), and up to 500 °C for silica-shell embedded gold nanorods (AuNR@SiO2). For AuNR@CTAB, through accurate temperature control, the longitudinal plasmonic band can be moved very close to the transversal one upon slight reduction of their length. Instead, for AuNR@SiO2, owing to the fully inorganic shell, a higher temperature of treatment can be reached leading to the possibility of reshaping the nanorods into spheres without the observation of any by-products.

Keywords Gold nanorods · Plasmon resonance · Thermal reshaping · FWHM

Introduction

The realization of nano-scale systems has been produced numerous benefits in everyday life in materials, electronics, medicine, energy conservation, and sustainability. At the bottom of this technological revolution, there are the nanoparticles and the processes that lead to their production [1–3]. As well known, metallic nanoparticles (MNPs), with a size smaller than the wavelength of the incident radiation, show resonance phenomena with the electro-magnetic field collectively called localized surface plasmon resonance (LSPR) [3–6]. LSPR properties are extremely interesting because they depend on, and consequently can be controlled by, the nature of the metal, the size and shape of the nanoparticles as well as the environment in which they are collocated [7–16]. In this way, an accurate synthetic method capable of controlling both the size and shape of the MNPs can allow tuning nanoparticle plasmonic properties [17, 18]. During the last decades, a variety of nanoparticles with specific shapes have been synthesized [19–21]. In particular, gold nanorods (AuNRs) have received vast attention due to their applications in biomedical technologies, plasmon-enhanced spectroscopies, optical and optoelectronic devices [22–25]. Indeed, the rod-like shape for its peculiar geometry with respect to other nanocrystals shows two plasmonic modes: a transversal and a longitudinal one. Actually, by tuning the length of the AuNRs during synthesis, the plasmon wavelength of the longitudinal mode can rather easily be adjusted in a wide spectral range, from the visible to the near-infrared region of the electromagnetic spectrum [26, 27]. Since their first discovery in 1991, for their wide perspective of applications, many synthetic protocols have been developed with the aim of improving the control over the yield of the synthesis, the uniformity in shape and dimension, but in particular on the most tricky part: the precise control of aspect ratio (the ratio between the length and the diameter), which mostly determines the features (shift and width) of the longitudinal band [28, 29]. To date, the most
effective way to control the shape of AuNRs has been achieved using the seeded growth method [30–32]. The basic principle of this shape-controlled method involves two steps: first, the preparation of small-size spherical AuNPs, and second, the growth of the prepared spherical particle in a rod-like micelle environment. To finely tune the morphology of the resulting nanorods, inorganic or organic additives can be added to the reaction media such as AgNO₃ [33–38] or 5-bromosalicylic acid [39–41].

In addition to chemical strategies, physical approaches based on thermal [42–44] and ultrafast laser-induced heating [45, 46] have also been proposed to modify the AuNR morphology. Indeed, since nanoparticles do melt at a lower temperature than the bulk of the relative element [46–50], heating can be employed to switch from the NRs to a thermodynamically more stable spherical shape. Furthermore, it has been found that the nanoparticle melting temperature is also greatly influenced by the nature of their capping agent or the medium in which they are dispersed [42, 48, 49]. Indeed, for this reason, and to efficiently employ AuNRs in specific applications, such as photothermal therapy, it is crucial to protect them from heat-induced shape deformation. In this regard, several methods were adopted to increase the stability of the core particles when applying heating, and in particular, enclosing AuNRs in a thick silica shell has been revealed to be a valid approach [35, 51–53].

In the present study, we first carefully investigated the role of the molecule/nanoparticle interaction in the seed-mediated growth approach by synthesizing gold nanorods covered with CTAB (AuNR@CTAB, sample A), and others covered with a silica shell of different thickness (AuNR@SiO₂, samples B₁, B₂, B₃). In a second phase, all prepared AuNRs have been exposed to an unconventional reshaping thermal treatment. The aspect ratio of the treated AuNRs has drastically changed thus allowing fine-tuning of the plasmonic response. While AuNR@CTAB were treated up to a temperature of 300 °C to avoid thermal degradation of CTAB organic the surfactant, AuNR@SiO₂ were exposed to temperature up to 500 °C. Upon temperature increase, an aspect ratio reduction was observed for all NRs, ultimately leading to the change of morphology from rod to sphere. In the case of AuNR@SiO₂, according to the thickness of the protective silica shell, this reduction of aspect ratio can however be attenuated. The homogeneity in shape and size of the nanoparticles has been ascertained, in each step, by TEM and photophysical analysis.

**Results and discussion**

**AuNR@CTAB**

Gold nanorods covered with CTAB, AuNR@CTAB, have been synthesized and dispersed in water according to the seed-mediated growth approach proposed by Scarabelli et al. [39, 54].

To prepare nanoparticles with anisotropic shape, it is necessary to use additional specific reagents acting as “template-mediated shape” such as CTAB itself or “shape directors” such as Ag(I) ions. Specifically, the synthetic mechanism proceeds through a physical separation of the nucleation process (seed formation solution) from the growth process (growth solution), allowing a more effective control of the final shape. Both solutions include a reductant, HAuCl₄ as gold source, and CTAB. In presence of CTAB, AuCl₄⁻ ions undergo metathesis reactions ultimately leading to the formation of AuBr₄⁻, which can be observed by the passing from a yellow solution to an orange one [31]. In the first step (seed solution), the strong reducing agent, NaBH₄ reduces Au(III) to Au(0) in presence of CTAB that covers the spherical seeds to prevent them from coalescence high-quality seed solution is necessary to obtain high-quality nanorods. Ideally, the seeds should be monodisperse and should also display the same crystallographic habit, which in practice, is achieved by adding the strong reducing agent in excess as fast as possible and under vigorous stirring. When the seeds are not monodispersed, the transversal band can also appear broadened (Fig. 1) for the presence of by-products (such as spheres or cubes) into the sample.

In some cases, the longitudinal band can also appear broadened (Fig. 2) as this is due to the co-presence of different sizes of nanorods in solution. By centrifugation, it is sometimes possible to separate the nanorods by size: the heavier will fall at the bottom of the tube while the smaller will remain in the supernatant. This separation is generally achieved by centrifuging at least at 4500 rpm for 10 min.

![Fig. 1 Extinction spectrum of an AuNR@CTAB sample with the presence of by-products derived from an inhomogeneity of the seed size](image-url)
Many actors are in play in the second step (growth solution) with regard to the growth processes of anisotropic gold nanoparticles and we tried to understand in depth their role, in particular, define which molecule/nanoparticle interactions are primarily responsible for shape control. In the preparation of the growth solution, we use CTAB and 5-BrSA in adequate proportion for which rod-like micelles are formed [40], these latter act as compartments of nanometric size, able to confine the nanocrystals growth, control and physically restrain their enlargement. We rationalized the mechanism underlying the process, taking into account that 5-BrSA acts as a weak reducing agent, reducing $\text{Au}^{\text{III}}$ to $\text{Au}^{\text{I}}$ (as showed by the bleaching of the starting $\text{AuBr}_4^{-}$ orange solution), and probably leading to the formation of a gold(I) complex [55–57]. This complex intercalates into the CTAB bilayer, stabilizing the structure and localizing $\text{Au}^{\text{I}}$ into the rod-like nanoreactor. To obtain rod-shaped, however, addition of an $\text{AgNO}_3$ water solution is necessary. Also, in this case, 5-BrSA is susceptible to complex $\text{Ag}^{\text{I}}$ [58] and, likewise to $\text{Au}^{\text{I}}$ ion, contribute to accumulate silver ion into the CTAB micelles. At this stage, seed solution is added to the growth solution just after ascorbic acid addition; the little spherical seeds, coated by CTAB, diffuse into the rod-like micelles. Silver ions form a monolayer on the seed surface, preferentially on the anisotropic faces that are eventually present. $\text{Au}^{\text{I}}$ ions, also deposited onto seed surface, suffer the following disproportion reaction:

$$3\text{Au}^+ \rightarrow 2\text{Au}^{0} + \text{Au}^{3+}$$  \hspace{1cm} (1)

The thus formed $\text{Au}^{\text{III}}$ is reduced back to $\text{Au}^{\text{I}}$ by ascorbic acid to $\text{Au}^{\text{I}}$, and the process goes on; the presence of silver ions driving the formation of the elongated shape. Indeed, the growth will occur on the faces where $\text{Ag}^{\text{I}}$ is absent. Ultimately $\text{Ag}^{\text{I}}$ ions are dislodged (we suggest for the lower Z-potential of $\text{Ag}^{\text{I}}$ with respect to $\text{Au}^{\text{I}}$) and the gold growth proceeds anisotropically (EDX analysis Fig. 1 SI). Scheme 1 recapitulates the steps of the synthesis.

**Role of CTAB and AgNO$_3$ concentration in AuNR@CTAB synthesis**

We reproduced the previous protocol by modifying CTAB and AgNO$_3$ concentrations, in order to investigate their contribution to the formation of the shape-like rod. In particular, by reducing the CTAB concentration of the growth solution from 0.05 to 0.025 M, nanorods with high polydispersity are formed, as shown in the TEM image below (Fig. 3).

Regarding the role of AgNO$_3$, we evidenced that its concentration plays a crucial role in the obtaining the nanorod shape. In fact, by reducing its amount, in our case...
of 10 times respect the reported protocol in experimental section (“AuNR@CTAB” section), nanoparticles of elongated shape, similar to bipyramids, are formed (Fig. 4), while, in absence of AgNO₃, various shapes are then observed (Fig. 5), and preferential growth has been lost. These experimental results confirm the AgNO₃ directing power discussed above.

Furthermore, as explained by Scarabelli, there is also a correlation between the aspect ratio of AuNR@CTAB and the pre-reduction time (Au³⁺ to Au¹⁺); practically the aspect ratio is correlated to the Au⁰ amount in the growth solution when ascorbic acid is added. We monitored the pre-reduction of Au³⁺ to Au¹⁺ following the drop in absorbance of the Au³⁺ band at 396 nm recording for a shorter pre-reduction time (more Au³⁺ than Au¹⁺ in solution) an increase in aspect ratio value, and for longer pre-reduction time (more Au¹⁺ than Au³⁺ in solution) a decrease in aspect ratio value. As known, the longitudinal band shifts from red to blue with decreasing in the aspect ratio value. Figure 6 reports the results obtained by fixing the pre-reduction time when the absorbance at 396 nm was measured around 0.85, and then AA was added, obtaining AuNR@CTAB with longitudinal plasmonic peak at 794 nm, transversal plasmonic peak at 513 nm, and aspect ratio 4 (calculated from TEM images, Fig. 7a). TEM images show nanoparticles homogeneous in shape and size. The extinction spectrum in solution confirms this: the more the longitudinal band is symmetric (with a limited broadening), the more the sample results homogeneous [54]. Increasing the pre-reduction time, by adding ascorbic acid when the absorbance at 397 nm is around 0.75, the aspect ratio of AuNR@CTAB decreases from 4 to 3.3 (Fig. 7b) and, consequently, the longitudinal band blue shifts from 794 to 766 nm. Spectra in Fig. 8 have been obtained by varying, in a narrower range, the pre-reduction time.

According to our previous protocol [53], we covered pre-synthesized AuNR@CTAB, AR = 3, with a mesoporous silica shell of different thickness, that were obtained by varying the AuNR@CTAB concentration, as reported in Table 1. By increasing the AuNR@CTAB concentration, the silica thickness, around gold nanorods, is drastically reduced (Fig. 9 TEM images of samples B₁, B₂, B₃). In detail, sample B₁, which was derived from the lowest concentration of AuNR@CTAB used, showed the thickest silica shell (50 nm × 30 nm). Increasing the AuNR@CTAB concentration, the silica thickness is reduced to achieve the dimension of 10 nm × 5 nm in sample B₃. In all samples, the silica around the gold nanorods is rather homogeneous. The silica coating strategy is based on a pH-controlled condensation of tetraethyl orthosilicate (TEOS) precursor
onto CTAB-capped AuNRs. Hydrolysis and condensation of TEOS require in this case basic conditions, reached by adding to the sample NaOH. CTAB surfactant deposited onto AuNRs is a structure-directing agent that facilitates the formation of mesostructured silica shell around the NRs.

**Thermal treatment of gold nanorods**

The four samples A (AuNR@CTAB), B₁, B₂, and B₃ (AuNR@SiO₂), dispersed in 2 mL of distilled water, were transferred into aluminum vials and placed in the center of a muffle furnace. The samples have been heated at different temperatures as indicated in the respectively caption and were maintained at this temperature for 30 min before letting the furnace to cool down at room temperature; samples were characterized by UV–Vis and TEM. After heating the sample, the NRs were all adhered onto the aluminum walls and were retrieved by addition of distilled water to the vial under sonication.

**Sample A** Analyzing the spectra showed in Fig. 10, a significant blue shift of the longitudinal plasmonic band is observed passing from 50 to 150 °C. For samples heated from 220 to 300 °C, no significant variation is observed. However, at 300 °C, a drastic absorbance decrease of the longitudinal band is registered, which becomes only barely

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**Fig. 6** Extinction spectra of gold nanorods with aspect ratio 4 (c) and 3.3 (d) respectively correlated with the amount of Au(III) present in the growth solution, Abs 0.85 (a) and 0.65 (b) respectively

**Fig. 7** TEM image of AuNR@CTAB aspect ratio 4 (on the right, a); TEM image of AuNR@CTAB aspect ratio 3.3 (on the left, b)
visible at 320 °C. By increasing temperature, the aspect ratio of AuNRs decreases, as confirmed by the morphological and optical characterization. TEM images (Fig. 11) show, as the treatment temperature was increased, a higher tendency of the nanoparticles to get shorter and bigger, which is in agreement with the progressive blue-shift of the longitudinal band, that approaches the transversal one with increase of temperature. However, it is worthy of note, as indicated by the narrowing of the longitudinal band (Fig. 10), that the NRs have gained in homogeneity of shape and size if the temperature of the treatment is kept under 300 °C. Figure 12 shows a graphic of FWHM (size dispersion measurement) vs. temperature. Unfortunately, owing to the organic nature of CTAB, further increase in temperature treatment was not possible for its thermal decomposition, hence irremediable agglomeration of the nanoparticles [59]. To palliate to such a drawback, we studied the effect of this post-synthesis heating treatment onto the AuNR@SiO₂.

**Sample B₁**  As observed for sample A, a blue shift of the longitudinal band with increasing of the temperature is still observed (Fig. 13), but the thick protective shell avoids a severe perturbation of the elongated rod shape (Fig. 14); in fact, the nanorods aspect ratio reduces only from 3 to 2.5, in agreement with the soft blue shift of the band. The difference in electron density of silica shell at 350 °C and 500 °C (more appreciable in SI Fig. 2) suggests a silica porosity increasing due the CTAB calcination.

**Samples B₂ and B₃**  As the thickness of the silica shell is reduced, the effect of the post-synthesis heating treatment is incremented. Again, homogeneity in size and shape is observed after all the heating treatments.

Only when the temperature is probably reaching the melting point of the gold nanoparticles (ca. 500 °C) the homogeneity might be reduced. This is particularly true for sample B₂ (Fig. 15) characterized by an intermediate thickness of shell, while instead for sample B₃ (Fig. 16) homogeneity in size and dimension is repristinate since all the NRs have switched shape to adopt a full spherical conformation. As for samples A and B₁, all these geometry changes of the NRs owe to the heat treatment can be followed by UV spectroscopy, where both blue-shifting and reducing of the half-height width of the longitudinal plasmonic band can be observed (Fig. 15). Noteworthy, the UV response of sample B₁ heated at 500 °C shows a unique band without any shoulder owe to the reaching of the full spherical shape (Fig. 16).

### Table 1 Correlation between [AuNR@CTAB]/M and silica thickness

| Sample  | [AuNR@CTAB]/M | Silica thickness |
|---------|---------------|-----------------|
| B₁ (36 µL TEOS) | 5.0E−5 | 60 nm×40 nm |
| B₂ (12 µL TEOS) | 1.0E−4 | 30 nm×15 nm |
| B₃ (12 µL TEOS) | 3.0E−3 | 10 nm×5 nm |

![Fig. 8](image1.png) *Extinction spectra obtained by varying the pre-reduction time in a narrower range*

![Fig. 9](image2.png) *TEM images of AuNR@SiO₂ covered with a silica thickness of 50 nm×30 nm (B1); 30 nm×15 nm (B2); 10 nm×5 nm (B3) long axis×short axis respectively (from left to right)*
Conclusions

In gold nanorods, the longitudinal plasmonic resonance band can be tuned by adjusting their aspect ratio and, for this purpose, many synthetic protocols have been developed to control this morphological parameter. Here we discussed the synthetic mechanism seed-mediated growth to obtain gold nanorods, clarifying, for the best of our knowledge, the role of each reagent in the nanoparticle formation. Accurate photophysical studies of the plasmonic properties of these metal nanoparticles have been performed, correlating them to the size and shape. This synthetic strategy has some limitations in terms of aspect ratio reproducibility: we cannot assume that the longitudinal band always falls at the same wavelength keeping the same experimental conditions and, generally, data reported in literature do not show longitudinal band under 600 nm (near the transversal one) and a narrow half-height width corresponding to a high degree of homogeneity of the nanorod sample in size and shape. Furthermore, the synthesis provides various steps and a long and delicate purification by centrifuge if often necessary. Here we propose an unconventional reshaping thermal strategy to reach a fine control of aspect ratio of gold nanorods. Compared to the traditional procedure, which makes a correlation between the longitudinal band position of gold nanorods and the pre-reduction time ($\text{Au}^{III}$ to $\text{Au}^{I}$), we tuned the longitudinal band by doing a correlation with the administered heating. In doing so, by preparing only one nanoparticle synthesis, we obtained various nanorod samples with different aspect ratio. The longitudinal band has been tuned very close to the transversal one, maintaining a homogeneity degree and better control of the aspect ratio reproducibility. We have shown that both CTAB-covered AuNRs and NRs embedded in a silica shell can be reshaped in this

Fig. 10 Extinction spectra of sample A: AuNR@CTAB at different temperatures; the FWHM (full width at half maximum) decreases increasing the temperature (110 nm = 25 °C; 65 nm = 120 °C)

Fig. 11 TEM images of sample A: AuNR@CTAB at increasing temperature: from black (25 °C), magenta (120 °C), green (150 °C) to violet (320 °C) picture frame, according to the spectra in Fig. 10
manner, but with particular care and limits. For CTAB, only temperature below 300 °C can be used, while for SiO₂-covered AuNRs, the results in terms of controlling the aspect ratio of the NRs are highly depending on the thickness of the shell.

However, since 500 °C can be reached without any degradation in this case, AuNRs can be fully switched into perfect spheres without any by-products.

Respect to the conventional synthetic strategies, thermal treatment is a faster way to tune the aspect ratio of gold nanorods, correlating the position of the longitudinal plasmonic band to the temperature. The opportunity to adjust the longitudinal mode of gold nanorods, in a wide spectral range, opens up wide fields of applications. Thus, the thermal reshaping method can be applied as a general strategy to personalize plasmon response expanding the nanoparticle applications in photothermal therapeutics and different application fields of nanophotonics. The method described here is reproducible, scalable, and the optical response of the resulting gold nanoparticles nears the theoretical limit, giving a highly appealing approach toward the increasing applications of plasmonic nanoparticles.

Furthermore, the same thermal treatment can be proposed for other metal nanorods for which synthetical pathways failed in furnishing high homogeneity in size and shape. This rising unconventional strategy could become a developing technique to produce nano-systems on a larger scale and at higher rates.

**Experimental section**

**Materials**

Hexadecyltrimethylammonium bromide (CTAB, ≥ 96%), 5-bromosalicylic acid (technical grade, 90%), hydrogen tetrachloroaurate trihydrate (HAuCl₄·H₂O, ≥ 99.9%), silver nitrate (AgNO₃, ≥ 99.0%), L-ascorbic acid (A.A. ≥ 99%), sodium iodide (99.99%), trisodium citrate (99%),

![Fig. 12](image12.png)  
**Fig. 12** Graphic of FWHM (size dispersion measurement) vs. temperature

![Fig. 13](image13.png)  
**Fig. 13** Extinction spectra of sample B₁ AuNR@Si at different temperatures

![Fig. 14](image14.png)  
**Fig. 14** TEM images of sample B₁ AuNR@Si at increasing temperature: from red (150 °C), gray (350 °C), to blue (500 °C) picture frame, according to the spectra in Fig. 13
polyvinylpolypyrrolidone (PVP K15, Mn 10000), and sodium borohydride (NaBH₄, 99%) were purchased from Aldrich and used for the preparation of gold nanorods covered with CTAB (AuNR@CTAB) as previously reported [39]. All the chemicals were used as received. Milli-Q water (resistivity 18.2 MΩ·cm at 25 °C) was used in all experiments. All glassware was cleaned with aqua regia, rinsed with water, sonicated threefold for 3 min with Milli-Q water, and dried before use. TEOS (Alfa Aesar, 99.9%), NaOH (Sigma Aldrich, 98%), and MeOH (Sigma Aldrich) were used for the SiO₂ over coating as reported in a previous work [53].

**Methods**

A Perkin Elmer Lambda 900 spectrophotometer was employed to obtain the extinction spectra. The size and the morphology of the gold nanoparticles were measured using a transmission electron microscope (Jeol JEM-1400 Plus 120 kV). The samples for transmission electron microscopy (TEM) were prepared by depositing a drop of a diluted solution on 300 mesh copper grids. After evaporation of the solvent in air at room temperature, the particles were observed at an operating voltage of 80 kV. For the heat treatment was used a muffle furnace Z 1200, GEFTRAN 1001 temperature control.

**AuNR@CTAB**

**Preparation of the seed solution** Twenty-five microliters of 5.0E−2 M HAuCl₄ water solution was added to 4.7 mL of 0.1 M CTAB water solution; 300 μL of a freshly prepared 1.0E−2 M NaBH₄ water solution was then injected under vigorous stirring. Excess of sodium borohydride was consumed by keeping the seed solution for 30 min at room temperature prior to use.

**Preparation of the growth solution** Forty-five milligrams of 5-BrSA was added to 50 mL of 0.05E−2 M CTAB water solution. After complete dissolution, 480 μL of 1.0E−2 M AgNO₃ water solution was added. The solution was mildly stirred for 15 min at room temperature, and then 500 μL of 5.0E−2 M HAuCl₄ water solution was added to the mixture, starting the pre-reduction step. At the selected pre-reduction time, 130 μL of 0.1 M ascorbic acid (AA) water solution was added under vigorous stirring, followed by 80 μL of seed solution. After 30 s, the stirring was stopped and the mixture was left undisturbed at room temperature for at least 4 h. The sample was finally centrifuged (9000 rpm, 20 min, 30 °C).
AuNR@SiO₂

An aqueous solution of CTAB (10 µL, 0.2 M) has been added to 2 mL of an aqueous dispersion of AuNR@CTAB at different concentration according to our previous work [40] (Table 1). After few seconds, 20 µL of a 0.1 M NaOH aqueous solution was added under vigorous stirring, reaching a pH value of 8.5, followed by three additions, each of 12 µL, of TEOS 20% v/v in methanol under gentle stirring, at room temperature. After 14 h, the mixture was centrifuged in Milli-Q water twice at 6000 rpm for 10 min and the sample was dispersed in 2 mL of Milli-Q water.

Thermal treatment

Two milliliters of each sample (A, B₁, B₂, B₃) was transferred into aluminum vials, placed in the center of a muffle furnace, and maintained at the chosen temperature (as indicated in the respectively figure caption) for 30 min. After this time, the same for all samples and for all temperatures tested, the temperature of the muffle was brought to room temperature and opened. Two milliliters of distilled water, under sonication, has been added to the aluminum vials thus bringing back into solution the dry nanoparticles.

Supplementary Information

The online version contains supplementary material available at https://doi.org/10.1007/s13404-022-00320-0.

Author contribution

A.C. synthesized and characterized the samples, wrote the main manuscript text; F.P. prepared the figures; G.D.M., F.S., I.A., N.G. reviewed the manuscript; M.L.D. supervised all steps, reviewed and validated the manuscript.

Funding

Open access funding provided by Università della Calabria within the CRUI-CARE Agreement. DEMETRA Project (PON ARS01_00401) supports the research funds.

Data availability

Not applicable.

Code availability

Not applicable.

Declarations

Competing interests

The authors declare no competing interests.

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