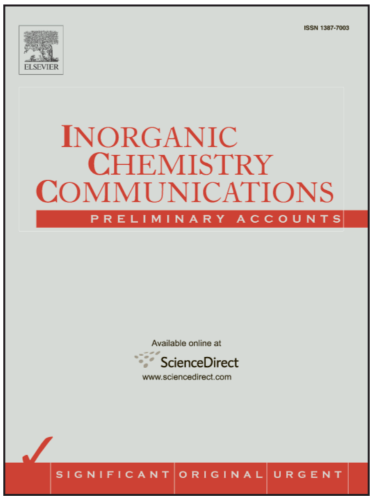

Synthesis of Graphene Quantum Dots Stabilized CuNPs and Their Applications in CuAAC Reaction and 4-Nitrophenol Reduction

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Synthesis of Graphene Quantum Dots Stabilized CuNPs and Their Applications in CuAAC Reaction and 4-Nitrophenol Reduction

Yu Huang,[a] Weifeng Chen,[a] Jialu Shen,[a] Yanlan Wang,[b] and Xiang Liu [a]*

Abstract: Here we report the synthesis of graphene quantum dots (GQDs) stabilized copper nanoparticles in neat water. Construction of six **CuNPs 1-6** nanocomposites are accomplished by mixing GQDs and $NaBH_4$, and followed by the addition of different amounts of copper sulfate at 0 ℃ in water, respectively. Among them, **CuNP-3**, with the size of 2.6 nm, exhibits excellent catalytic performance in the CuAAC reaction over than other **CuNPs**. Moreover, the **CuNP-3** has also been successfully applied for the reduction of 4-nitrophenol with excellent catalytic activity.

1. Introduction

Click chemistry was first reported by Sharpless, Kolb and Finn in 2001, as a general concept for the high-efficiency assembly of organic molecules.[1] Among the various click reactions, the copper catalyzed azide-alkyne cycloaddition (CuAAC) reaction has attracted wide attention, because its selectively corresponding product 1,4-disubstituted 1,2,3-triazole, one kind of important *N*-containing heterocycles,[2] has been widely used in medicinal chemistry, fluorescent materials, corrosion-retarding agents, agricultural products, dyes and chem-sensing.[3] In general, the CuAAC reaction requires stoichiometric quantities of Sharpless-Fokin Cu^I catalyst for obtaining high yield and avoiding the byproduct of 1,5-disubstituted 1,2,3-triazoleis.[4] However, this “click” chemistry is always restricted in the field of electronics and biomedicine due to the difficult removal of the large quantity of copper ions.[5]

Graphene quantum dots (GQDs), a new kind of graphene fragments nanomaterial, have been extensively investigated in biomedical, optoelectronic, fluorescent sensor, catalysis and energy related fields because of their high intrinsic fluorescence, high biocompatibility, high photostability, low toxicity, chemical inertness, large surface area and surface functional groups (including carbonyl, carboxyl, epoxy and hydroxyl groups).[6] In addition, GQDs have also been successfully developed as a novel stabilizer for transition metal nanoparticles. For example, GQDs stabilized AuNPs for DNA damage detection,[7] H_2O_2 electrochemical detection,[8] and Pb^{2+} detection.[9] In fact, our group has long term interest in the synthesis of transition metal nanoparticles and their applications.[10] In the present work, we first developed a facile and novel approach for graphene quantum dots stabilized copper nanoparticles (CuNPs) and their applications in CuAAC reaction and 4-nitrophenol reduction. In this work, GQDs A with the size from 2.25 to 3.50 nm has been synthesized from only commercial starch and water by our previously reported method (**Fig. 1**).[6a] Construction of the CuNPs have been carried out by here direct coordination of different amounts of Cu(II) ions into GQDs A solution followed by $NaBH_4$ reduction in neat water (**Table 1**). GQDs A is very stable and soluble in water, allowing for stabilizing the CuNPs in a controlled fashion to avoid the aggregation. Then these six CuNPs are investigated in the CuAAC reaction in neat water. Among them, CuNPs-**3** exhibits excellent catalytic activity over than other CuNPs. The **3** has also been characterized by UV-vis spectroscopy, transmission electron microscopy (TEM) and X-ray photoelectron spectroscopy (XPS). In addition, CuNPs-**3** has also been measured for the reduction of 4-nitrophenol with high activity.

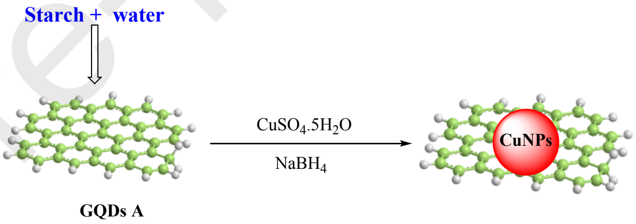

Figure 1. The synthesis of CuNPs.

2. Results and Discussion

2.1. Synthesis of the CuNPs 1-6

First, GQDs A with the size of from 2.25 to 3.50 nm has been synthesized from only commercial starch and water.[6a] The synthesis of GQDs-stabilized **CuNPs 1-6** was conducted by using GQDs A and sodium borohydride dissolved in water, and followed by the addition of different amounts of $CuSO_4·5H_2O$ (5.0×10^{-3}, 2.0×10^{-3}, 1.0×10^{-3}, 5.0×10^{-4}, 2.5×10^{-4}, 1.0×10^{-4} mmol) at 0 ℃, respectively, provoking the formation of yellow color (CuNPs), corresponding to the reduction of the Cu^{2+} ions to the Cu(0) metal and **CuNPs 1-6** formation (**Table 1**). Cu^{2+} ions are first bonded by -OH and -COOH groups of the GQDs, then reduced by $NaBH_4$, further as-synthesized CuNPs has been stabilized by these -OH and -COOH groups. As a comparative experiment, the commercial starch has also been used as a substitute for GQDs A in the same above condition. The result shows that starch is insoluble in water at 0 ℃ and does not stabilize **CuNPs.**

2.2. Compared catalytic activities of CuNPs in the click reaction

These six CuNPs compared catalytic performance in click reaction of phenylacetylene and benzyl azide has been scrutinized. As shown in **Table 1**, the reactions were carried out with phenylacetylenes (0.5

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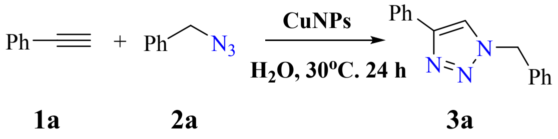
Table 1. Optimization of the reaction condition.[a]



Entry	Cu (Ⅱ) ions (mmol)	CuNPs	CuNPs (mol)	Yield[b] (%)
1	5.0×10^{-3}	**1**	0.01%	11.7
2	2.0×10^{-3}	**2**	0.01%	26.1
3	1.0×10^{-3}	**3**	0.01%	86.0
4	1.0×10^{-3}	**3**	0.02%	90.2
5	1.0×10^{-3}	**3**	0.05%	95.1
6	1.0×10^{-3}	**3**	0.10%	92.4
7	5.0×10^{-4}	**4**	0.01%	83.5
8	2.5×10^{-4}	**5**	0.01%	5.5
9	1.0×10^{-4}	**6**	0.01%	28.0

[a] Reaction conditions: 1a (0.5 mmol),2a (0.6 mmol), H_2O (2 ml), 30 ℃, 24 h.

[b] Isolated yield.

mmol) and benzyl azide (0.6 mmol) in the presence of **CuNPs 1-6** (0.01 mol%) in H_2O (2 mL) at 30℃ for 24 h, respectively (**Table 1**, entry 1-3 and 7-9). Among them, **CuNP-3** provides the highest yield of the desired product **3a** (86.0 %). Next, we investigated the different loadings of **CuNP-3**, we found the amount of 0.02 mol %, 0.05 mol% and 0.10 mol % obtained 90.2, 95.1 and 92.4 % yields, respectively (**Table 1**, entries 4-6). Thus, 0.05 mmol % **CuNP-3** in H_2O is chosen as the optimal conditions for this click reaction. Then TEM and UV-vis. spectrum have been measured to confirm the morphology and size of the **CuNP-3**. As shown in **Fig. 2a and 2b**, we found the size of **CuNP-3** is 2.6 nm. However, the characteristic peaks of **CuNP-3** did not appear in the UV-vis spectrum, due to its small size (**Fig. 2c**). The representative high-resolution transmission electron microscopy (HRTEM) image of **CuNP-3** exhibits the Cu (111) lattice fringe distance of 0.209 nm in the inner region (**Fig. 2d**). Furthermore, the crystalline nature of the **CuNP-3** was determined using X-ray diffraction (XRD), only graphene (002) was observed in **Fig. S1**, due to too little of CuNPs. To further verify the formation of **CuNP-3**, the XPS of **CuNP-3** has been recorded. Cu 2p is fitted with two pair of peaks for the Cu $2p_{3/2}$ and $2p_{1/2}$ doublets in the XPS. The Cu $2p_{3/2}$ peak positions are around 932.5 and 934.0 eV. These positions are assigned to Cu(0) at 932.5 eV and to Cu(II) at 934.0 eV, respectively.[11] Obviously, **CuNP-3** has been partly oxidized to Cu(I) and Cu(II). All of these results showed that the **CuNP-3** was in its classic Cu^I state for click reaction.

Under the optimized reaction conditions, we extended the study with different acetylenes and benzyl azides for the synthesis of the corresponding 1,4-disubstituted 1,2,3-triazoles. As shown in **Table 2**, a variety of alkynes and organic azides are well suited for this "click" reaction with the **CuNP-3** as the catalyst. For instance, either the electron donating substituted or withdrawing substituted acetylenes including Ph-C≡CH, 4-Br-C_6H_4C≡CH, 3-F-C_6H_4C≡CH, 4-Cl-C_6H_4C≡CH and 4-MeO- C_6H_4C≡CH with benzyl azide obtaining the corresponding 1,4-disubstituted 1,2,3-triazole derivatives **3a-e** in good to excellent yields (72 to 95 %). Similar excellent isolated yields (71 to 88 %) of 1,4-disubstituted 1,2,3-triazoles **3f-h** are afforded when ethynylbenzene is reacted with variously electronically substituted R^2-N_3 organic azides (such as 4-F-$C_6H_4CH_2N_3$, 4-CN-$C_6H_4CH_2N_3$ and 4-Me-$C_6H_4CH_2N_3$). However, the lowest yield of **3i** (47 %) is obtained, when the azide contains the 4-Cl group.

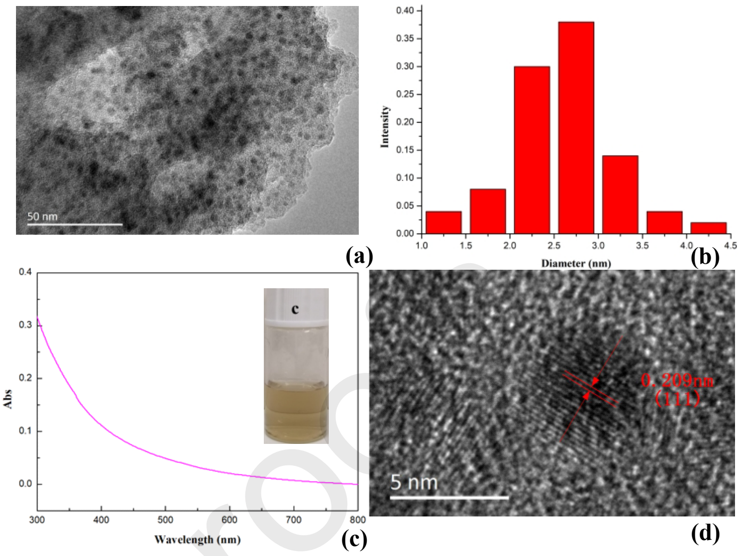


Figure 2. (a) TEM image of **CuNP**-3;(b) Diameter histogram distribution of **CuNP-3**; (c) UV-vis. Spectrum of **CuNP-3**. (d) HRTEM of **CuNP-3**.

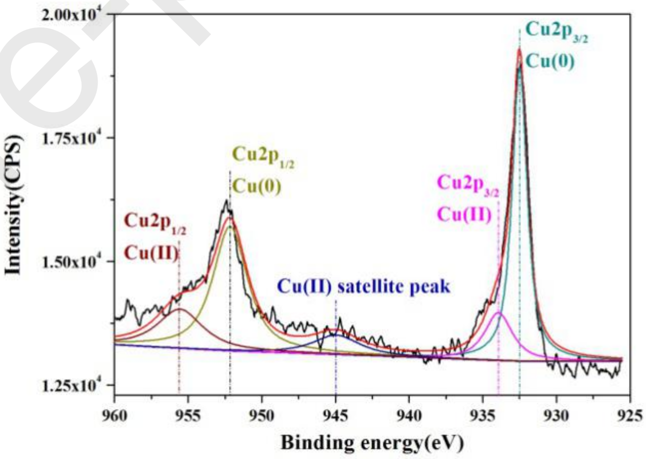
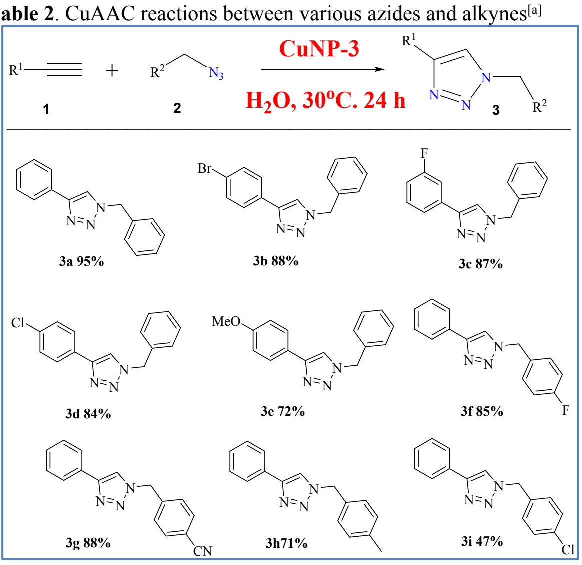


Figure 3. XPS of **CuNP-3**.

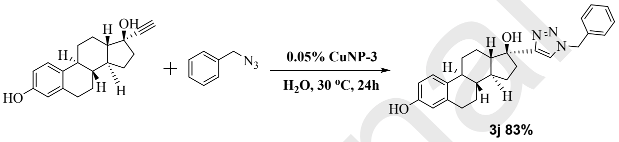
In addition, the **CuNP-3** has also been successfully applied for preparing some key functional materials. Such as"click"-triazole functionalized moxestrol (**Eq. 1**) and "click"-triazole functionalized 7-

(propargyloxy)-coumarin (**Eq. 2**). Estradiol, as the predominant sex hormone present in female, has been mainly used in hormone replacement therapy and menopausal hormone therapy in women, reproduction, nervous system, the skeletal system and pharmaceuticals. Here the CuAAC click reaction of benzyl azide and ethynyl estradiol, in the presence of 0.05% **CuNP-3** in H_2O at 30 ℃ for 24 h, provided "click"-triazole functionalized moxestrol **3j** in 83% isolated yield (**Eq 1**). On the other hand, coumarin and its derivatives are widely used in fluorescent probes and the perfume industry.[12] Thus, click-triazole-functionalized 7-(propargyloxy)-coumarin **3k** was obtained from 7-(prop-2-yn-1-yloxy)-2H-chromen-2-one and benzyl azide in water in 88 % isolated yield in the presence of **CuNP-3** (0.05 %) .

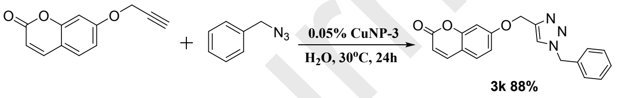
Table 2. CuAAC reactions between various azides and alkynes[a]



[a] Reaction conditions: Azide 0.6 mmol, alkyne 0.5 mmol, H_2O 2 mL, 30 °C, 24 h.



Eq. 1



Eq. 2

2.3. Catalytic activities of CuNP-3 in the 4-nitrophenol reduction

The **CuNP-3** has also been successfully applied for the reduction of 4-nitrophenol, a highly hazardous and poisonous industrial pollutants, to 4-aminophenol. As a industrial intermediate, 4-aminophenol has been widely used in hair dying agents, anticorrosion lubricants and antipyretic and analgesic medicine. thus efficient **CuNP-3** catalysis of 4-nitrophenol reduction is of great significance. Besides, it is very convenient to monitor the progress of 4-nitrophenol reduction by UV-vis. spectra, due to the distinct peak of 4-nitrophenol and 4-aminophenol are at 400 and 300 nm, respectively.[13] The other key sign of the reaction progress is the disappearance of the yellow color of the solution. The 4-nitrophenol reduction has been carried out in the presence of 5% **CuNP-3** and 100 eq $NaBH_4$ in water at room temperature (**Fig. 4a**). The result shows the reduction reaction does not need any induction time and is completed in 1 h. The apparent rate constant k_{app} is 8 × 10^{-4} s^{-1} (**Fig. 4b**).

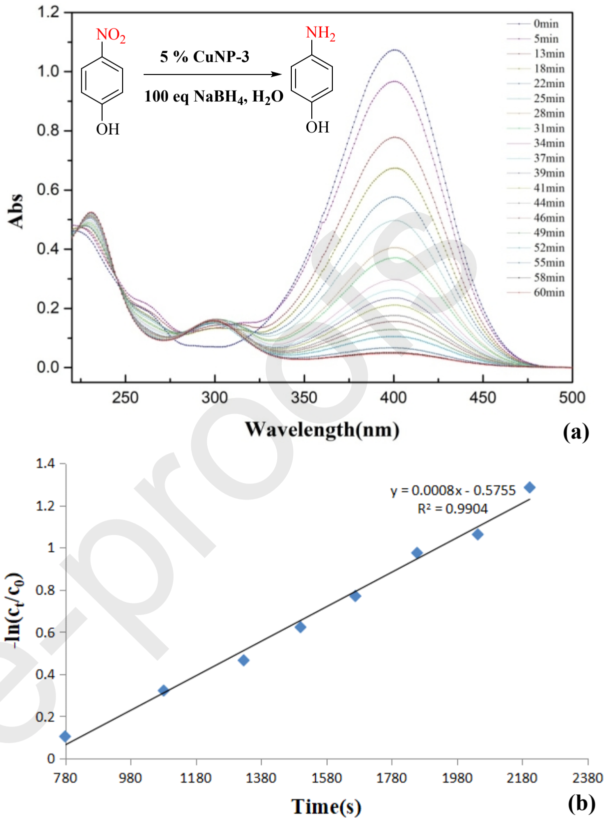


Figure 4. (a) UV-vis spectrum in which 4-nitrophenol reaction is monitored 25 ℃; (b) Consumption rate of 4-nitrophenol -ln(C/C_0) versus reaction time

3. Conclusion

A green and facile method of synthesis of graphene quantum dots stabilized CuNPs has been developed in this present work. Construction of six **CuNPs 1-6** nanocomposites are accomplished by mixing GQDs and sodium borohydride, and followed by the addition of different amounts of $CuSO_4{\cdot}5H_2O$ at 0 ℃ in water, respectively. Among them, **CuNP-3** exhibits excellent catalytic performance in the CuAAC reaction over than other **CuNPs**. The **CuNP-3** has also been characterized by UV-vis spectroscopy, TEM, XRD and XPS. Then a variety of alkynes and organic azides are well suited for this "click" reaction with the **CuNP-3** as the catalyst, leading to the regioselective formation of various 1,4-disubstituted 1,2,3-triazoles (including "click"-triazole functionalized moxestrol and "click"-triazole functionalized 7-(propargyloxy)-coumarin) in high isolated yields. The **CuNP-3** has also been successfully applied in the reduction of 4-nitrophenol with excellent catalytic activity. In addition, we are trying to immobilize our CuNPs on magnetic Fe_3O_4 for the recyclability of graphene quantum dots stabilized CuNPs. Such work will be published in the future. The graphene quantum dots stabilized CuNPs show wide potential applications in cell image, sensor, and catalysis fields.

Experimental

1. Preparation of CuNPs

First, 1 mL(0.29 g/L) GQDs are dissolved in 2 mL of deionized water in a Schlenk flask, and the solution is stirred for 10 mins at 0 ℃. Then a colorless solution of $NaBH_4$ (1 x10^{-2} mmol in 1 mL water is added to the solution of GQDs. And the solution is stirred for 30 mins. A 1-mL aqueous solution containing 1.0 ×10^{-3} mmol $CuSO_4$ is added dropwise, provoking the formation of yellow color, corresponding to the reduction of the cation to the zero-valent metal and **CuNP-3** formation. The **CuNP-3** were kept in aqueous solution for characterization and used as the catalysts. Other CuNPs were synthesized in the same method.

2. The general procedure of the click reaction

All reactions were performed on a 0.50 mmol scale of triazole. The phenylacetylene (0.5 mmol), benzyl azide (0.6 mmol), **CuNPs-3** (0.05% mmol) and 2 mL of H_2O were taken into a round bottom flask equipped with stirrer. The resulting mixture was stirred for 24 h at 30 ℃. After cooling to room temperature, and extracted with acetic ether (3 × 10 mL). The combined organic phases were washed with brine (2 × 5mL), dried over anhydrous $MgSO_4$ and concentrated under reduced pressure. The residue was subjected to flash column chromatography with hexanes/EtOAc (5:1) as eluent to obtain the desired **3a** as light yellow solid (95.1% yield).

3. Catalysis of 4-nitrophenol reduction

An aqueous solution (2.5 mL) containing 4-nitrophenol (2.5 × 10^{-4} mmol) and $NaBH_4$ (2.5 × 10^{-2} mmol) is prepared in a standard quartz cuvette (3 mL, path length: 1 cm). The **CuNP-3** (5 mol%) is injected into this solution, and the reaction progress is detected by UV-vis. spectroscopic analysis every min at 25 ℃.

Acknowledgment

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Appendix A. Supplementary data

Supplementary data to this article can be found online at

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1. Graphene quantum dots stabilized copper nanoparticles has been synthesized in water
2. The as-synthesized CuNPs shows high catalytic activity in the "click" reaction
3. The as-synthesized CuNPs show high catalytic activity in the reduction of 4-nitrophenol

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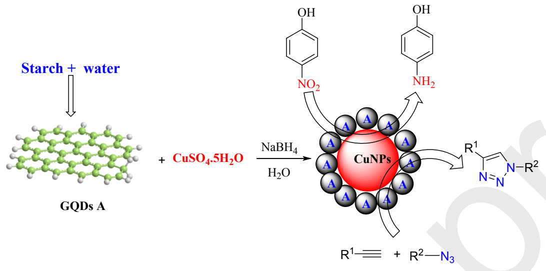
Here we report the synthesis of graphene quantum dots (GQDs) stabilized copper nanoparticles in water. The as-synthesized nanoparticles show high catalytic activity in the "click" reaction and reduction of 4-nitrophenol



Copper Nanoparticles

*Yu Huang, Weifeng Chen,Jialu Shen, You Wu, Yanlan Wang, and Xiang Liu**

Page No. 1– Page No.5

Synthesis of Graphene Quantum Dots Stabilized CuNPs and Their Applications in CuAAC Reaction and 4-Nitrophenol Reduction

Entry for the Table of Contents (Please choose one layout)

Layout 1:

Conflicts of interest

All the authors confirm that there are no conflicts to declare

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