Matrix-free room-temperature phosphorescence graphitic carbon nitride quantum dots based on halogen bond interactions for security applications

Yuelin Kong1 | Yu He1 | Yaping Wang2 | Gongwu Song1

1 Hubei Collaborative Innovation Center for Advanced Organic Chemical Materials, Ministry of Education Key Laboratory for the Synthesis and Application of Organic Functional Molecules, College of Chemistry and Chemical Engineering, Hubei University, Wuhan 430062, China
2 State Key Laboratory of Biocatalysis and Enzyme Engineering, School of Life Sciences, Hubei University, Wuhan 430062, China

Correspondence
Yue He, Hubei Collaborative Innovation Center for Advanced Organic Chemical Materials, Ministry of Education Key Laboratory for the Synthesis and Application of Organic Functional Molecules, College of Chemistry and Chemical Engineering, Hubei University, Wuhan 430062, China. Email: heyu@hubu.edu.cn

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Abstract
Herein, we first designed a one-step strategy to achieve matrix-free room temperature phosphorescence (RTP) materials by low temperature solid phase synthesis, using sodium citrate as carbon source and urea as nitrogen source. The NaF, NaCl, NaBr, and NaI were chosen as the inducer source of halogens to effective trigger the RTP emission from graphitic carbon nitride quantum dots (CNQDs) denoted F-CNQDs, Cl-CNQDs, Br-CNQDs and I-CNQDs. All the doped-CNQDs emitted bright fluorescence upon 365 nm irradiation. After the 365 nm UV lamp is off, only the Br-CNQDs showed green phosphorescence which can be observed by naked eye. The phosphorescence emission of Br-CNQDs is excitation-dependent from 510 to 610 nm with the average RTP lifetime of 70 milliseconds, which indicated Br-CNQDs could exhibit color-tunable phosphorescence. When 365 nm UV is off, Br-CNQDs emitted green afterglow for about 3 seconds observed naked eye, while the Br-CNQDs presented visible yellow afterglow for about 3 seconds with ceasing off 395 nm irradiation. The absolute phosphorescence quantum yield (PQY) of the Br-CNQDs powder reaches up to 3.64%. Thus, the color change and long lifetime of phosphorescence from the excitation-dependent feature could provide a security application for counterfeiting and information protection systems.

KEYWORDS
anti-counterfeiting, carbon nitride quantum dots, halogen, long emission wavelength, room temperature phosphorescence

1 | INTRODUCTION

Room temperature phosphorescence (RTP) materials are highly desirable due to their unique optical properties, such as long lifetime, large Stokes shift and high signal-to-noise ratio.1–3 These exceptional characteristics endow RTP materials with wide applications in the fields of information security, biological imaging and optical sensors.4–6 The most of efficient RTP materials are rare-earth-based inorganic compounds, organometallic...
compounds and pure organic compounds.\textsuperscript{[7–9]} However, the inorganic and organometallic RTP materials containing noble metals have the drawbacks of high cost and potential toxicity. The pure organic compounds suffer from the disadvantages of solvents required, low yields, or poor stabilities.\textsuperscript{[10]} Therefore, how to avoid the usage of toxic and expensive metals and simplify the preparation to a large extent should been taken into accounts for the development of new generation of RTP materials.

Carbon nitride quantum dots (CNQDs), a new kind of carbon based nanomaterials, are highly attractive due to promising applications including biomedicine, sensing and photocatalysts.\textsuperscript{[11–13]} To date, great achievements have been made to explore the fluorescence properties of CNQDs, while less attention has been paid on the RTP properties of CNQDs.\textsuperscript{[14]} Significant breakthroughs have been achieved to realize the RTP properties of carbon based nanomaterials by three strategies. (1) Different solid matrices such as polyvinyl alcohol, silica, filter paper and boric acid, have been employed to immobilize carbon dots to present RTP\textsuperscript{[15–18]} (2) Superior RTP performances of carbon dots have been demonstrated by crosslink-enhanced emission, aggregation-induced emission and self-protective effect, which suppresses the nonradiative transitions of triplet excitations.\textsuperscript{[19–21]} (3) The introduction of heteroatoms, such as phosphorus (P), nitrogen (N) and boron (B) into the carbon dots has also been proved to be feasible.\textsuperscript{[22–24]} Despite there are outstanding results from previous work, the research of carbon based nanomaterials, especially CNQDs, is still at its baby stage and has many problems, including the regulation of RTP emission lifetime and understanding of the emission mechanism of RTP should be further investigated.

Numerous of work demonstrated halogen bonding is a type of noncovalent interaction between a halogen atom and a negative site to encourage intersystem crossing from the excited singlet to triplet state of organic phosphorescence.\textsuperscript{[25]} For example, Yang et al. presented a novel strategy to enhance the quantum efficiency of ultra-long organic phosphorescence materials by modulating intramolecular halogen bonding via structural isomerism.\textsuperscript{[26]} Cai et al. employed a chemical strategy to improve phosphorescence efficiency with intermolecular $\pi$-type halogen bonding construction via isomerism.\textsuperscript{[27]} Recently, Geddes and colleagues designed a novel phosphorescent “heavy carbon” nanodot by effectively incorporating bromine into the nanostructure.\textsuperscript{[28]} Although this work was first to introduce internal heavy atom effect to fabricate phosphorescent carbon based nanomaterials, it had drawbacks in synthetic complexity and photostability.

Inspired with the effective internal heavy atom effect in the carbon dots, we designed a one-step strategy to achieve matrix-free RTP materials by low temperature solid phase synthesis, using sodium citrate as carbon source and urea as nitrogen source. The NaF, NaCl, NaBr, and NaI were chosen as the inducer source of halogens. The schematic illustration of the synthesis is shown in Scheme 1. From the Scheme 1A, the CNQDs emitted strong fluorescence under irradiation of UV lamp ($\lambda = 365$ nm) but no phosphorescence after the irradiation. When NaF, NaCl, NaBr, and NaI were doped in the CNQDs, respectively, we acquired doped CNQDs denoted F-CNQDs, Cl-CNQDs, Br-CNQDs, and I-CNQDs with different optical characteristics. All the doped-CNQDs emitted bright fluorescence upon 365 nm irradiation. After the 365 nm UV lamp is off, only the Br-CNQDs showed green phosphorescence which can be observed by naked eye. The phosphorescence emission of Br-CNQDs is excitation-dependent from 510 to 610 nm with the average RTP lifetime of 70 milliseconds, which indicated Br-CNQDs could exhibit color-tunable phosphorescence. In the Scheme 1B, when 365 nm UV is off, Br-CNQDs emitted green afterglow for about 3 seconds observed naked eye (Video 1 in Supporting Information), while the Br-CNQDs presented visible yellow afterglow for about 3 seconds with ceasing off 395 nm irradiation (Video 2 in Supporting Information). The absolute phosphorescence quantum yield (PQY) of the Br-CNQDs powder reaches up to 3.64%. The excellent phosphorescence performance of Br-CNQDs was demonstrated to apply for anti-counterfeiting and information protection.

2 EXPERIMENTAL SECTION

2.1 Reagents

Urea (CON$_2$H$_4$) was purchased from Aladdin Reagent Co., Ltd (Shanghai, China). Sodium citrate, sodium nitrate (NaNO$_3$), sodium chloride (NaCl) and sodium fluoride (NaF) were purchased from Bodi Chemical Co., Ltd (Tianjin, China). Sodium bromide (NaBr) was purchased from Macklin Reagent Co., Ltd (Shanghai, China). Potassium iodide (Kl) and lithium bromide (LiBr) were purchased from Macklin Reagent Co., Ltd (Shanghai, China). Potassium bromide (KBr) was purchased from Sinopharm Chemical Reagent Co., Ltd (Shanghai, China). All chemicals were of analytical grade and used without further purification.

2.2 Instruments

The morphology and crystal structure were observed on JEM-2010 FEF transmission electron microscope (JEOL, Japan) and Talos-200 FEI HR-TEM (Thermo Fisher Scientific, America). X-ray diffraction (XRD) pattern was
analyzed using a D8 Advance diffractometer (Bruker, Germany). Fourier-transform infrared (FT-IR) spectrum was characterized by a Spectrum one FT-IR spectrophotometer (Perkin Elmer, America). X-ray photoelectron spectroscopy (XPS) were performed with a VG Escalab 200 spectrometer using an aluminum anode (AlK\(\alpha\)) operating at 510 W with a background pressure of \(2 \times 10^{-9}\) mbar (Thermo Fisher, America). UV–vis absorption spectra were recorded on a Lambda 35 UV spectrometer (Perkin Elmer, America). Fluorescence measurements were acquired on a LS55 fluorescence spectrometer (Perkin Elmer, America). The absolute PL quantum yield (QY) was measured on a FLS980 photoluminescence spectrometer (Edinburgh, Britain). The fluorescence and phosphorescence lifetimes were measured on a FLS1000 photoluminescence spectrometer (Edinburgh, Britain).

### 2.3 Synthesis of CNQDs and RTP Br-CNQDs

The CNQDs were synthesized by a one-step solid phase reaction strategy.\(^{[29]}\) The RTP Br-CNQDs were synthesized by the strategy with modification. First, urea (0.1010 g), sodium citrate (0.0716 g) and NaBr (0.4283 g) were ground uniformly in an agate mortar. Next, the mixed powder was transferred to an autoclave, and heated at 180°C for 1 hour in the drying oven to obtain a yellowish brown solid.

### 2.4 Control experiments on the effect of reactants, anions and metal ions

The urea, sodium citrate, CNQDs, urea-NaBr, sodium citrate-NaBr, and NaBr were reacted separately. Halogen ions (F\(^-\), Cl\(^-\), Br\(^-\), I\(^-\)) and NO\(_3\)\(^-\) were chosen as doped main anions. Alkali ions (Li\(^+\), Na\(^+\), K\(^+\)) were chosen as doped main metal ions. The moles of the precursors and the method of synthesis was the same as Section 2.4.

### 2.5 Anti-counterfeiting applications

An alphabetic security code was simply designed by loading RTP Br-CNQDs into the letter patterns “P” and “WUHAN”. The hidden three security figures “067” were loaded on the three figure molds “888” by RTP Br-CNQDs, while the remaining parts of three figure molds “888” were full with non-RTP CNQDs as the interference. Similarly, the triangle in hexagram pattern was made by RTP Br-CNQDs while the rest was full with non-RTP CNQDs.

### 3 RESULTS AND DISCUSSION

Heavy atom effect (HAE) is employed as one of the most important ways to induce the phosphorescence of lumino-gens by improving the intersystem crossing (ISC) rate and
efficiently enhance spin-orbit coupling (SOC). Inspired by this, NaF, NaCl, NaBr and NaI were chosen as the inducer source of halogens for trigger the RTP of CNQDs via one-step low temperature solid phase synthesis, using sodium citrate as carbon source, urea as nitrogen source.

In order to understand the influence of reactants, halogen atoms and alkali ions on RTP CNQDs, a series of control experiments were carried out. The CNQDs, F-CNQDs, Cl-CNQDs, Br-CNQDs, and I-CNQDs were synthesized according to a previous report with modification. The Figure 1A shows that the CNQDs exhibited only fluorescence emission at 510 nm and no RTP emission with the excitation at 400 nm. When NaF, NaCl, NaBr, and NaI were doped in the CNQDs, respectively, only Br-CNQDs displayed both strong fluorescence emission at 470 nm and strong RTP emission at 580 nm with the excitation at 400 nm (Figure 1B). Compared with Br-CNQDs, F-CNQDs, Cl-CNQDs, and I-CNQDs exhibited weak RTP emission at 580 nm (Figure 1C), although they also exhibited strong broad fluorescence emission from 460 to 580 nm (Figure 1D). It revealed that halogen atoms (F, Cl, Br, and I) could induce the RTP of CNQDs via this one-step low temperature solid phase synthesis, which is probably due to the fact that the halogen atoms promote efficient spin-orbit coupling to increase the probabilities of the $S_0$ to $T_1$ transition and $S_1$ to $T_1$ intersystem crossing. The RTP enhanced ability of Br was much greater than that of other halogen atoms, which could be caused by that fluorine and chlorine have very weak SOC ability, ordinarily it could not induce efficiently room-temperature phosphorescence. The lack of strong phosphorescence in iodated nanodots could indicate that covalent attachment of iodine to the nanostructure is not as efficient as bromination to produce a phosphorescence signal.

To better understand the RTP and fluorescence characteristics, the RTP lifetimes and fluorescence lifetimes of different halogen doped CNQDs were measured. Under 400 nm excitation, the average fluorescence lifetimes of F-CNQDs, Cl-CNQDs, Br-CNQDs, I-CNQDs, and NO$_3$-CNQDs were, respectively, calculated to be 2.86, 3.54, 4.92, 2.82, and 3.63 nanoseconds (Figure S1). The average RTP lifetimes of F-CNQDs, Cl-CNQDs, Br-CNQDs, and I-CNQDs were measured under excitation wavelength at 400 nm and emission wavelength at 580 nm, the average RTP lifetimes of F-CNQDs, Cl-CNQDs, Br-CNQDs, and I-CNQDs were, respectively, 0.49, 0.38, 70, and 0.56 milliseconds by tri-exponential function (Figure 2). The RTP lifetime of Br-CNQDs is several hundred times higher than that
of F-CNQDs, Cl-CNQDs, and I-CNQDs. Not only the halogen atoms affected a lot but the alkali ions also favorable for prompting the efficiency of RTP. The Figure S2a displayed that RTP of CNQDs at 580 nm was greatly enhanced in the presence of sodium compared with other alkali bromide (LiBr and KBr). The fluorescence emission of CNQDs-LiBr, CNQDs-NaBr, and CNQDs-KBr were located at 480, 470, and 540 nm when exciting at 400 nm, respectively, (Figure S2b). The fluorescence lifetimes of CNQDs-LiBr, CNQDs-NaBr, and CNQDs-KBr were 6.43, 4.92, and 2.71 ns, respectively, (Figure S3). Br-CNQDs exhibited the best fluorescence and RTP characteristic. Therefore, NaBr was chosen as the inducer to trigger the RTP emission of CNQDs effectively through facile, green, one-step and cost-effective methodology.

In addition, the control experiments with different reactants were also conducted and the results are displayed in Figure 3A. No RTP was observed when urea or sodium citrate was used alone as a precursor with or without the addition of NaBr. The strong RTP material denoted Br-CNQDs could be acquired only when urea, sodium citrate and NaBr were reacted together by low temperature solid phase synthesis. To obtain the optimal synthetic conditions, the influences of reaction time, reaction temperature and the ratio of reactants were investigated (Figures S4 and S5). The spectroscopic properties of Br-CNQDs were further characterized. The UV-Vis absorption curve of Br-CNQDs (Figure 3B) presented a strong absorption peak at 340 nm due to the n−π* transition of C=N/C=O bonds. The fluorescence emission (Figure 3C) and the phosphorescence emission (Figure 3D) indicated that Br-CNQDs were excitation-dependent. The Figure 3C shows that the fluorescence peaks shifted from 440 to 510 nm by tuning the excitation wavelength from 280 to 440 nm. The Figure 3D displays the phosphorescence peaks shifted from 510 to 610 nm by tuning the excitation wavelength from 280 to 440 nm. The absolute PQY of the Br-CNQDs powder was measured to be 3.64% when the excitation wavelength and emission wavelength were located at 400 and 580 nm, respectively.

To further understand the structure and composition of Br-CNQDs, Transmission electron microscope (TEM), Fourier transform-infrared spectroscopy (FTIR), X-ray diffraction (XRD) and X-ray photoelectron spectroscopy (XPS) of CNQDs and Br-CNQDs were carried out. The size and morphology of the Br-CNQDs were examined by the TEM and HRTEM. The Figure 4A displays that the morphology of Br-CNQDs is spherical and evenly distributed. The lattice spacing of Br-CNQDs obtained by HRTEM was approximately 0.29 nm, which is a typical of carbon nitride (inset in Figure 4A). The Figure 4B shows the diameters of Br-CNQDs were mainly distributed in the
**FIGURE 3**  
A, Fluorescence and phosphorescence emission spectra of CNQDs; B, Phosphorescence emission spectra of CNQDs-NaBr, urea, sodium citrate, CNQDs, urea-NaBr, sodium citrate-NaBr, and NaBr; B, UV-Vis absorption spectra of Br-CNQDs; C, FL spectra of Br-CNQDs at various excitation wavelengths; D, Ph spectra of Br-CNQDs at various excitation wavelengths

**FIGURE 4**  
A, TEM and HRTEM images of Br-CNQDs; B, Size distribution; C, The XRD spectra of CNQDs, Br-CNQDs, and NaBr; The XPS spectrum of Br-CNQDs: C 1s (D), N 1s (E), and Br 3d (F)
narrow range of 2.49±0.12 nm, which is consistent with that of CNQDs in Figure S6a and S6b. The XRD pattern was used to further study the crystal structure of CNQDs, Br-CNQDs, and NaBr. As shown in Figure 4C, Br-CNQDs had the same peak (002) and peak (100) with CNQDs, corresponded to the characteristic interlayer packing of the aromatic system and the in-plane structural packing motif of the tri-s-triazine unit, respectively. [32] In addition, Br-CNQDs had the same sharp peak at 29.88° 2θ with the NaBr. Thus, bromine atom was successfully introduced into the CNQDs.

The surface functional groups of CNQDs and Br-CNQDs were observed by FTIR spectroscopy. In Figure S6c, the broad absorption peak at 3459 cm⁻¹ is assigned to stretching vibrations of N-H and O-H, the peaks at 1708 and 1420 cm⁻¹ were attributed to the bending vibration of C–NH and stretching vibrations of CN heterocycles, respectively. Also, the peak at 1591 cm⁻¹ was related to C=O stretching vibrations. Compared to CNQDs, the Br-CNQDs had an additional absorption peak at 500–700 cm⁻¹ attributed to the vibration of C–Br. Using XPS measurements to determine the surface elemental composition of Br-CNQDs. The full range spectrum showed four peaks at 285.0, 399.2, 532.3, and 68.5 eV, which were ascribed to C 1s, N 1s, O 1s and Br 3d, respectively (Figure S6d). The C 1s spectrum (Figure 4D) consisted of three peaks, corresponding to C–C/C=C (283.9 eV), C-O/C–N (285.0 eV) and C=O (287.1 eV), respectively. The N 1s spectrum (Figure 4E) had two main peaks, attributing to C=N (398.4 eV) and N-H (400.9 eV), respectively. As for the O 1s spectrum (Figure S6e), the two peaks centered at 531.0 and 532.2 eV were assigned to C=O and C–O, respectively. The Br 3d spectrum (Figure 4F) had two main peaks, attributing to C–Br (69.5 eV) and Br⁻ (68.4 eV), respectively. The above results prove that we have successfully synthesized Br-CNQDs.

Although multicolor fluorescence materials were received much attention, rare RTP materials with tunable color have been explored so far. Thanks to the long lifetime of RTP Br-CNQDs, their potential application in anticounterfeit technology and information encryption was exploited (Figure 5). The RTP Br-CNQDs was filled in the mold of capital letter “P,” which means phosphorescence. When 365 nm UV was off, the capital letter “P” emitted green afterglow for about 3 seconds observed naked eye, while it presented visible yellow afterglow for about 3 seconds with ceasing off 395 nm irradiation (Figure 5A). Then, the molds of capital letter “WUHAN” were loading by Br-CNQDs. Under a hand-held UV lamp (λ_ex = 365 nm or λ_ex = 395 nm), bright green or yellow “WUHAN” was obtained after switch off the lighting source (Figure 5B). According to the model shown in Figure 5C, the hidden three security figures “067” are loaded on the three figure molds “888” by RTP Br-CNQDs, while the remaining parts of three figure molds “888” were full with non-RTP CNQDs as the interference. Once the UV lamp was turned off, the green or yellow color number “067” was visible owing to the delayed RTP of the Br-CNQDs. Upon 365 nm
irradiation, the hexagram pattern displays bright blue fluorescence. After the excitation was ceasing off, the green or yellow RTP triangles were present, the fluorescence of CNQDs from angle part of hexagram is indistinguishable (Figure 5D). Thus, the color change and long lifetime of phosphorescence from the excitation-dependent feature could provide a security application for counterfeiting and information protection systems.

4 | CONCLUSIONS

In summary, the matrix-free RTP Br-CNQDs based on halogen bond interactions were first synthesized via one-step low temperature solid phase method, inspired with the effective internal heavy atom effect in the carbon dots. The synthesis method has the advantages of simple process and short time-consuming. When NaF, NaCl, NaBr, and NaI were doped in the CNQDs, respectively, we acquired doped CNQDs denoted F-CNQDs, Cl-CNQDs, Br-CNQDs, and I-CNQDs with different optical characteristics. All the doped-CNQDs emitted bright fluorescence upon 365 or 395 nm irradiation. After the UV lamp turned off, only the Br-CNQDs showed green or yellow RTP which can be observed by naked eye. The Br-CNQDs exhibited excitation-dependent emission from 510 to 610 nm with the average RTP lifetime of 70 milliseconds, which indicated Br-CNQDs could exhibit color-tunable phosphorescence. The color change and long lifetime of phosphorescence feature of Br-CNQDs could provide a security application for counterfeiting and information protection systems.

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CONFLICT OF INTEREST

The authors declare no conflict of interest.

DATA AVAILABILITY STATEMENT

Research data are not shared.

ORCID

Yu He https://orcid.org/0000-0002-8068-5825

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AUTHOR BIOGRAPHIES

Yuelin Kong obtained a BS in chemistry education from China Three Gorges University, China. She is currently studying for her MS degree at Hubei University.
Yu He is an associate professor in College of Chemistry and Chemical Engineering at Hubei University. Her current interests include analytical chemistry and environmental chemistry.

Yaping Wang received her MS degree at Hubei University. Now, she is working in State Key Laboratory of Biocatalysis and Enzyme Engineering, School of Life Sciences, Hubei University.

Gongwu Song is a professor in College of Chemistry and Chemical Engineering at Hubei University. His current interests mainly focus on analytical chemistry and environmental chemistry.

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