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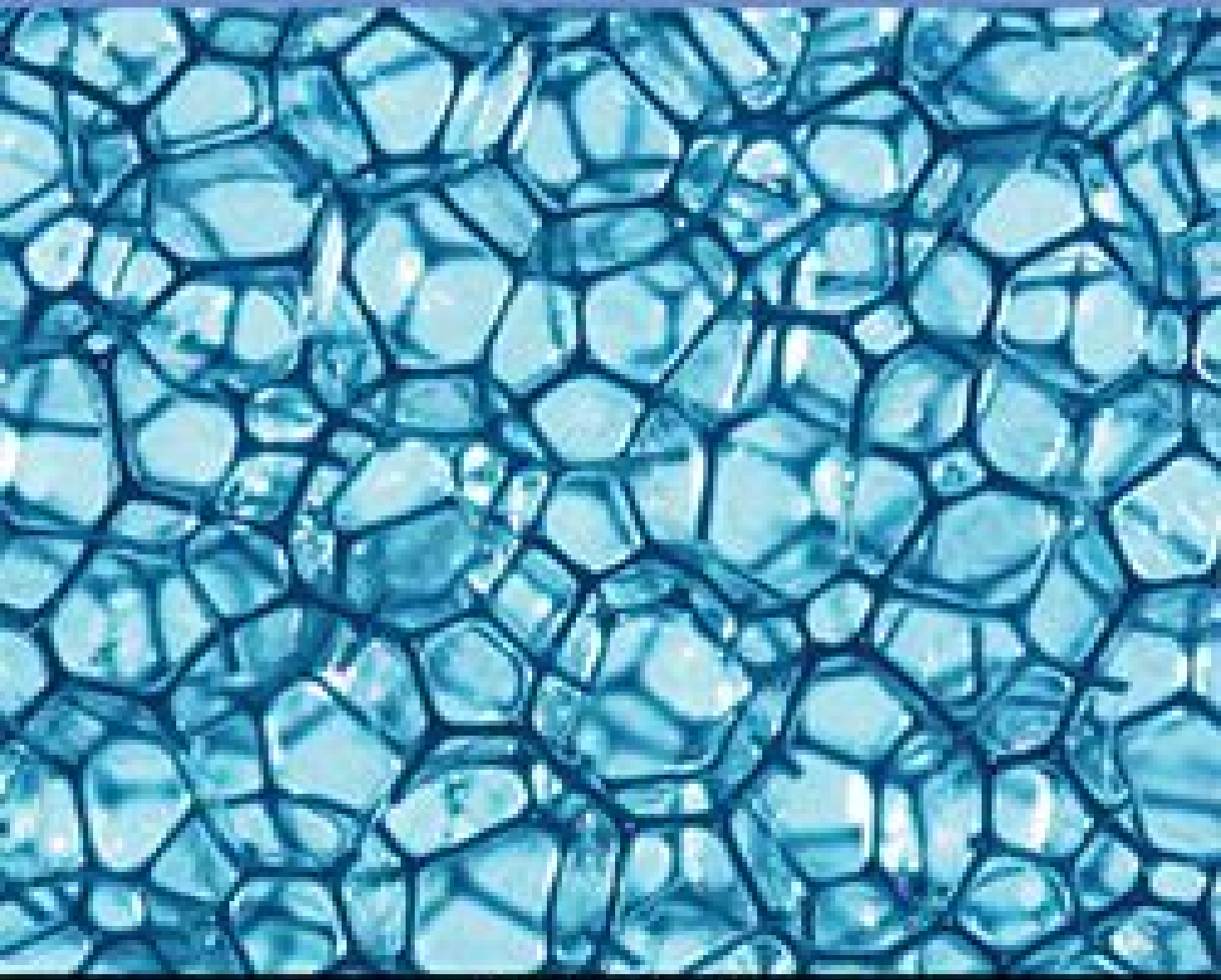

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Abstract

Security ink using a carbon nanoparticle (C-dot)/PVA/chitosan-composite-based material has been successfully synthesized. The C-dot powder was prepared using a urea pyrolysis method. The precursors were synthesized using urea ($(NH_2)_2CO$, Mw = 60.07 g mol^{-1}) and citric acid ($C_6H_8O_7{\bullet}H_2O$, Mw = 210.14 g mol^{-1}) as the fuel and carbon sources, respectively. The C-dots were prepared by heating the precursor solution at 250 °C for 90 min. The security ink was fabricated using C-dots, polyvinyl alcohol (PVA, $(CH_2CH(OH))_n$, with Mw = ~20 000 g mol^{-1}) and chitosan as the dyes, resins and binders, respectively. The morphology and optical properties of the security ink were measured using SEM and EDX, a PL spectrometer and UV–vis spectroscopy. The viscosity properties of the security ink were measured using a viscometer. The characterization showed that the C-dots have a monodisperse particle size, a tetragonal structure and absorption spectra in the UV light region. It is shown that the PVA:chitosan concentration has a significant effect on the viscosity properties, so the viscosity is optimized for the security ink. In addition, the security ink was studied using a commercial printer, and the results show a good quality blue emission (450 nm) appearing under UV light exposure at 365 nm. The security ink C-dot/PVA/chitosan composite has potential applications in security, panel display, optoelectronic and optical devices on an industrial scale.

1. Introduction

Luminescent materials have semiconductor-like properties, which change in response to the stimulus of external radiation, such as that from UV light, thermal or nuclear sources. Luminescent materials have optical security features that are difficult to imitate and duplicate. Therefore, they are widely used as non-colour security inks for applications in optical data recording, storage and security [1]. Some photoluminescent materials have been widely used as fluorescent ink in organic dyes [2], conjugated polymers [3] and inorganic quantum nanodots [4]. Organic dye has low photostability and a small Stokes shift, while the conjugated polymers and inorganic quantum nanodots have a fluorescence characteristic that is easily tuneable with a narrow emission bandwidth and high photostability. In recent years, nanoparticles doped by lanthanide atoms and/or rare earth metal ions have been developed for application in anti-counterfeiting security ink. However, there are concerns regarding the long-term cytotoxicity risk and potential environmental pollution caused by lanthanide and rare-earth-ion-based security ink [5].

In recent research, our group succeeded in developing a new type of graphitic carbon-nanoparticle-based C-dot fluorescent material without using rare earth ions [6]. C-dots are the most stable allotropes of carbon, and their potential has developed due to their unique electronic and optical properties [7]. C-dots can be synthesized on a large scale using a nitrogen condensation process, including the use of melamine, urea and thiourea [8]. C-dots have been successfully developed for various applications, such as in solar absorber materials, bioimaging and sensors [9–11]. Compared with inorganic quantum nanoparticles, C-dots have many advantages such as their simple fabrication process, high quantum efficiency, good biocompatibility, low cost and cytotoxicity and high photostability [9].

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Table 1. The various PVA and chitosan mass concentrations in 100 ml of distilled water.

Sample	PVA (g)	Chitosan (g)
Non-blend	0	0
Blend 1	0.4	0.1
Blend 2	0.8	0.2
Blend 3	1.6	0.4
Blend 4	2.8	0.7

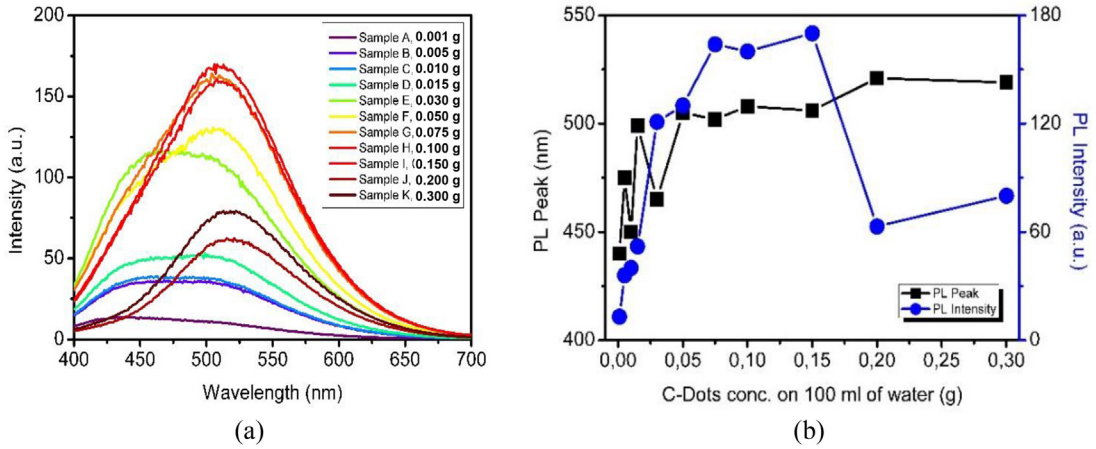

Figure 1. (a) The PL spectra and (b) PL properties of the C-dot solution for various concentrations under UV light exposure at 365 nm.

In this paper, we report the synthesis and characterization of a C-dot/PVA/chitosan-composite-based security ink prepared by the wet chemical method. We used PVA and chitosan as adhesives to control the oxidation and dissolution of the C-dot particles after printing. We varied the PVA/chitosan concentration synthesis to determine the composite viscosity optimization of the C-dot/PVA/chitosan-composite- based security ink. The morphology, viscosity and optical properties of the C-dot/PVA/chitosan-composite-based security ink were measured using SEM and EDX, a viscosity meter, a PL spectrometer, and UV–vis spectroscopy.

2. Experiment

The C-dot powder was synthesized using citric acid and urea as the carbon and fuel source, respectively. The precursor solution was prepared by mixing all the raw materials with a carbon/fuel (C/N) mass ratio of 3 g/3 g. The precursors were heated at 250 °C for 90 min using a commercial oven to obtain a black carbon powder [12]. The C-dot solution was prepared by dispersing the synthesized carbon powder with a varying mass from 0.01 g to 0.3 g in 20 ml of pure water. The C-dot solution was stirred using a magnetic stirrer until all the carbon powder had dispersed and become a clear brown solution.

The security ink was fabricated using the prepared C-dots, polyvinyl alcohol (PVA, Mw = ~20 000 g mol^{-1}) and chitosan (β-(1-4)-linked D-glucosamine and N-acetyl-D-glucosamine) to form a colloidal composite of C-dot/PVA/chitosan. The C-dot/PVA/chitosan composite synthesis has several steps: first, the chitosan powder is slowly dissolved in pure water with 2% acetic acid at room temperature until it becomes a homogeneous chitosan solution. Then, polyvinyl alcohol (PVA) and C-dots are mixed into the chitosan solution and stirred at 70 °C. To optimize the viscosity of the security ink, the PVA/chitosan concentration was varied from 0% to 2.8 % w/v, as shown in table 1. PVA and chitosan with a mass ratio of 4:1 were used to produce a composite polymer with the best hydrophobic properties [13]. In addition, the prepared security ink was printed onto several types of commercial paper to determine the nature of its interaction with the C-dot/PVA/chitosan composite.

The photoluminescence (PL) spectra (Cary Eclipse spectrofluorometer, Agilent, Australia) and the FT infrared spectra (FTIR, Bruker Optics, Ettlingen, Germany) were taken, and scanning electron microscopy (SEM, JEOL JCM-6000 Benchtop, Japan) was conducted to characterize the optical properties, chemical bonding and morphology of the prepared samples, respectively. The viscosity measurement of the optimized C-dot/PVA/chitosan-based security ink was conducted using a rotational viscometer TV-20 Tokimec at room temperature.

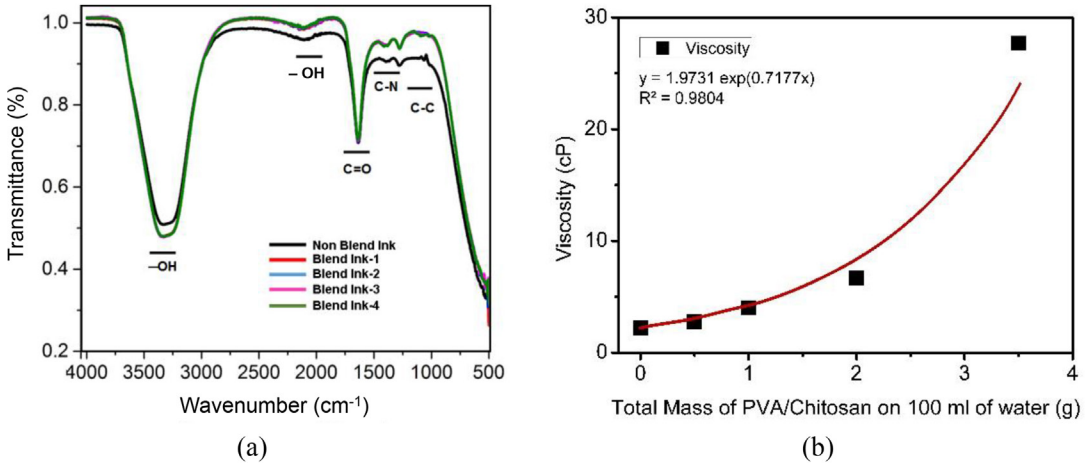

Figure 2. (a) The FTIR spectra of the ink sample solutions and (b) the viscosity of the C-dot/PVA/chitosan composite as security ink.

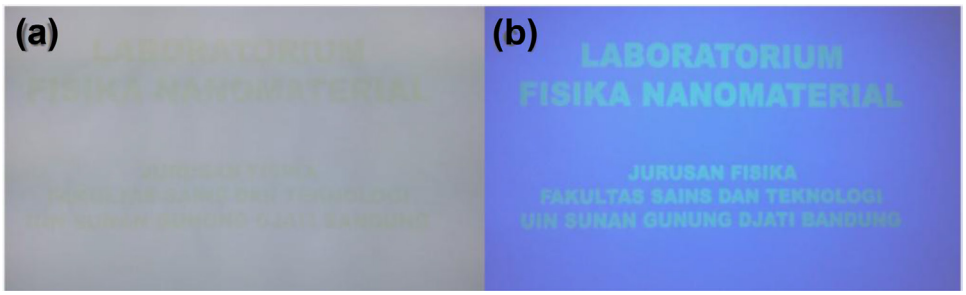

Figure 3. The printing results of the C-dot/PVA/chitosan-based security ink on A4 commercial paper: (a) under visible light and (b) under UV light exposure (365 nm).

3. Results and discussion

The C-dot solution was prepared by dispersing the synthesized carbon powder in pure water. Figure 1(a) shows the PL spectra of the C-dot solution for several concentrations between 0.01 g and 0.3 g in 20 ml of pure water. The PL spectra characterization shows that all the prepared samples of C-dot solution have a blue luminescence with a PL peak from 440 nm to 520 nm. Moreover, the blue luminescence properties of the prepared samples were optimized by the increasing the concentration of the C-dot solution, as shown in figure 1(b). At low concentrations, the C-dot solution has a wide range of PL emission peaks, and possibly two PL peaks at 400 nm and 500 nm. The existence of two PL peaks is caused by the interaction between the C-dots and chitosan with an excitation wavelength at 365 nm [13]. The PL properties showed that sample I, with a C-dot concentration at 0.75% w/v has the highest emission intensity. However, the PL intensity of the C-dot solution decreased for sample J (1% w/v) and sample K (1.5% w/v). The PL properties of the prepared samples show that the electronic structure of the C-dot particles remains unchanged, even when the C-dot concentration is enlarged (see figure 1(b)). However, the optimized PL intensity of the C-dot solution was probably caused by the photon–particle interactions between the C-dot particles and the excitation wavelength. The physical models for these phenomena are: (i) when the number of C-dot particles is low, the amount of C-dot particle emission is lower as well. (ii) When the number of C-dot particles increases, the number of C-dot emission photons is higher as well. However, (iii) when the amount of particles is high, some emitted photons are reabsorbed by the carbon particles, forming a nonradiative recombination, and causing the emission intensity to decrease (quenching effect recombination).

To determine the existing functional groups (chemical bonding) of the security inks, the C-dot/PVA/chitosan-prepared samples were characterized using FTIR, as shown in figure 2(a). The FTIR spectra results indicate the existence of several chemical bonds between the C-dots and the polymers, and also show their interaction, such as C–C at 950–1030 cm^{-1} and C–N at 1400–1420 cm^{-1}. In addition, the functional groups of –OH (water combination band) appear at 3250–3420 cm^{-1} and 1655 cm^{-1}; these results show the surface interaction between the C-dots, polymers and solvents [13]. This indicates that the C-dot particles have the ability to disperse in water, due to their dominant hydrophilic nature [14]. The chemical bond between the PVA and chitosan was expected to form a composite polymer with the C-dots. Furthermore, when the prepared security ink was printed on a

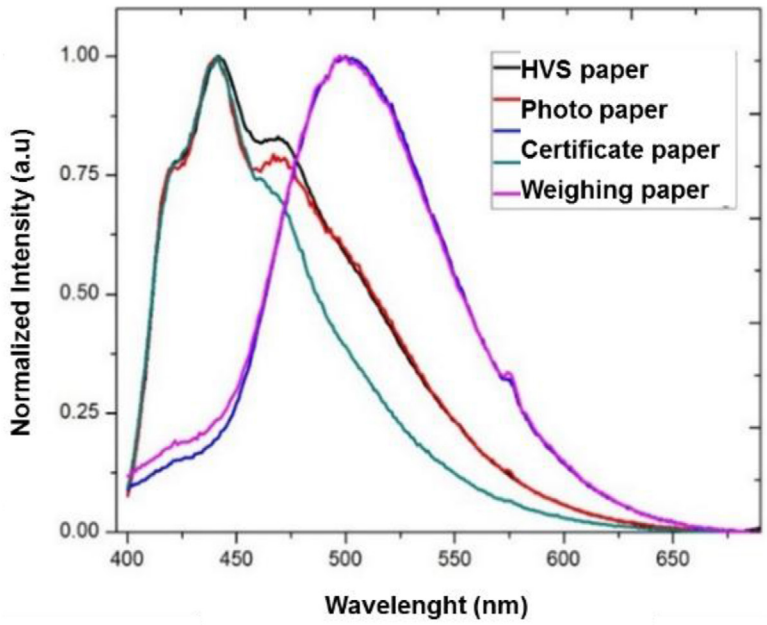

Figure 4. The PL spectra of C-dot/PVA/chitosan printed on various types of paper under UV light exposure at 365 nm.

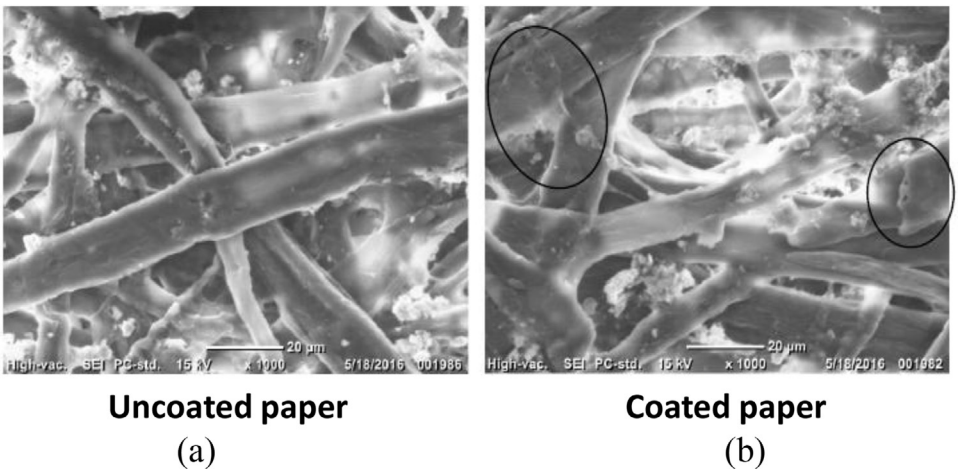

Figure 5. SEM images of the A4 commercial paper (a) uncoated and (b) coated with the C-dot/PVA/chitosan-composite-based security ink.

substrate, such as paper, the PVA/chitosan behaved as a thin layer of composite polymer with strong hydrophobicity. The hydrophobic nature of the PVA/chitosan composite was expected to prevent the re-dissolution or the oxidation process of the C-dot particles. The viscosity of the C-dot/PVA/chitosan composite was measured for various PVA/chitosan concentrations, as shown in figure 2(b). The viscosity results show that the addition of PVA and chitosan increases the viscosity of the security ink exponentially. The increasing viscosity of the C-dot/PVA/chitosan composite might have been caused by the increasing amount of PVA/chitosan bonding crosslinks.

The solution of ink blend 1 was chosen as the security ink for the printing test using a commercial printer (HP Deskjet 1010). The printing stages consisted of: (i) injection of the ink into an empty cartridge, (ii) automatic printing on several types of paper, and (iii) the drying process at room temperature. Figure 3 shows the printing result of the security ink on an A4 paper sample. The initial observation proved that the security ink had successfully been printed on the paper, and exhibited blue emission under UV light exposure (365 nm). To understand the interaction between the security ink and the paper, as well as its influence on the optical properties, the PL spectra were measured for various types of commercial paper. Figure 4 shows the PL spectra of the manually printed prepared security ink on various types of commercial paper. In general, the PL spectra showed that the printed security ink exhibited a blue emission with a PL peak from 410–530 nm under UV light exposure (365 nm). However, the PL spectra showed that the emission properties of the printed results are highly dependent on the interaction of the security ink with the commercial paper. The shift in the properties of the PL was possible, since the paper may

have contained a material that undergoes photoluminescence, such as melamine and others. Therefore, further in-depth study on the interaction between carbon-nanoparticle-based security inks and paper needs to be done.

Figure 5 shows an SEM image of A4 commercial paper both uncoated and coated by the C-dot/ PVA/chitosan-composite-based security ink using a commercial printer. The observation results show that the uncoated and coated commercial paper have the same morphological characteristics. On closer inspection, the coated paper had a thin layer of polymer composite on the paper fibres; in addition, the porosity of the coated paper was lower, since the composite polymer had filled in the pores. Therefore, it is concluded that the C-dot/PVA/chitosan-based security ink can be manually or automatically printed, and that the security ink composites strongly interact with the morphology of the paper.

4. Conclusions

Security ink using a carbon nanoparticle C-dot/PVA/chitosan-composite-based material has been successfully synthesized. The C-dot powder was prepared using a urea pyrolysis method. The precursors were synthesized using urea ($(NH_2)_2CO$, Mw = 60.07 g mol^{-1}) and citric acid ($C_6H_8O_7{\bullet}H_2O$, Mw = 210.14 g mol^{-1}) as the fuel and carbon sources, respectively. The C-dots were prepared by heating the precursor solution at 250 °C for 90 min. The security ink was fabricated using C-dots, polyvinyl alcohol (PVA, $(CH_2CH(OH))_n$, with Mw = ~20 000 g mol^{-1}) and chitosan as the dyes, resins and binders, respectively. The characterization showed that the C-dots have a monodisperse particle size, a tetragonal structure, and absorption spectra in the UV light region. The viscosity properties show that the PVA:chitosan concentration has a significant effect which optimizes the viscosity of the security ink. In addition, the security ink was studied using a commercial printer, and the printing results show a good quality blue emission (450 nm) appearing under UV light exposure at 365 nm. The C-dot/PVA/chitosan-composite-based security ink has potential applications in security, panel displays, optoelectronic and optical devices on an industrial scale.

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