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Poly(vinyl)alcohol film composited with carbon dots from water hyacinth stalks based on gamma irradiation for the UV blocking film

Tanagorn KWAMMAN[1,2], Threeraphat CHUTIMASAKUL[1], Panida SANGANGAM[3], Nattamon PUENGPOSOP[3], and Kanokorn WECHAKORN[3,*]

[1] *Thailand Institute of Nuclear Technology (Public Organization), Nakorn Nayok 26120, Thailand*
[2] *Mahidol University International College, Mahidol University, Nakhon Pathom 73170, Thailand*
[3] *Department of Chemistry, Faculty of Science and Technology, Rajamangala University of Technology Thanyaburi, Pathum Thani 12110, Thailand*

***Corresponding author e-mail:** kanokorn_w@rmutt.ac.th

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Abstract

UV-radiation in the range of 200 nm to 400 nm from sunlight can inhibit the photosynthesis process in plants, while visible light in the range of 400 nm to 700 nm promotes photosynthesis and plant growth. In this work, poly(vinyl)alcohol-carbon dots composite film (PVA-CDs film) was developed as a UV-blocking film due to the excellent optical properties of carbon dots and the film-forming properties of PVA. For the synthesis of carbon dots using gamma irradiation, water hyacinth stalk, an invasive aquatic plant, was used as a carbon source. The result showed that PVA-CDs composite film exhibited excellent UV absorption (200 nm to 400 nm, 91%) and fluorescence emission in the visible region. Furthermore, PVA-CDs composite film displayed a blue-to-red light emission of 57.2%, and the UV emission also significantly decreased.

1. Introduction

Sunlight spectrum consists of UV (200 nm to 400 nm, 6.6%), visible (400 nm to 700 nm, 44.7%), and infrared (700 nm to 1000 nm, 48.7%) regions [1]. However, plant growth is most sensitive to red (620 nm to 750 nm) and blue light (450 nm to 495 nm), which are directly involved in photosynthesis and protein production during plant development [2,3]. Moreover, UV regions from sunlight can inhibit the photosynthesis process and plant growth [2]. Therefore, UV blocking film for promoting agriculture is needed for the growth and quality of the plant. Generally, transparent polyester films used for greenhouse roofs have effectively filtered UV light.

The photoconversion technique has been applied to convert UV light to a specific wavelength *via* photoluminescence [4]. Kyprianidou-Leodidou *et al.* observed that polymer-embedded or coated TiO_2 and ZnO nanoparticles can enhance the photo- and thermal-stability, light scattering, and UV absorption. In 2016, Chahal *et al.* reported that the Ag nanoparticles/poly(vinyl)alcohol (PVA) film blocked UV light at 200 nm (98%), 300 nm (65%), and 400 nm (91%) [5]. Moreover, the $Cd_{(1-x)}Zn_{(x)}Se$ quantum dots/polyethylene (PE) film was used to convert UV light to blue and red light, promoting plant growth pumpkin by 25%, pepper by 30%, and tomato by 55% [6]. However, these composites required expensive raw materials and a prolonged time for the synthesis process.

The organic nanoparticles were reported as UV-blocking molecules. The colloidal lignin nanoparticles/PVA film showed good transparency and blocked 100% of the UVC (100 nm to 290 nm) and UVB (290 nm to 320 nm), and more than 50% of the UVA (320 nm to 400 nm) [5]. In 2018, the graphene synthesized from sugar beet leaves blocked 70% of UV light after mixing with PVA polymer [7]. Additionally, the conjugated aromatic compounds such as cinnamates, *p*-aminobenzoic acid, and oxybenzone can be filtrated the UV light *via* fluorescence emission [8].

Carbon dots (CDs) are novel nanoscale carbon materials with outstanding optical and electrical properties, promising to be excellent UV blocking materials [9]. The optical properties of CDs are able to be modified by morphology control, surface chemistry, and heteroatom doping [9-16]. The organic CDs strongly absorb the UV light (200 nm to 300 nm) and have excellent tunable fluorescence properties [9,11-16]. It was reported that the fluorescence quantum yield of CDs enhanced about 50% after embedding on the PVA matrix [13]. Furthermore, the PVA/CDs film still exhibited as a fluorescent sensor for tartrazine in food as low as 10 μM [17]. Importantly, Patil *et al.* reported that PVA/CDs film protected the grape color change from UV irradiation for 30 h for the lab-scale test [18].

The high quantum yield of CDs can be synthesized from biomass *via* pyrolysis, carbonizations, and hydrothermal methods [9,14,19-22]. However, these methods required high temperatures, toxic chemicals,

and organic solvents. With this regard, the radiation process is an alternative green synthesis method. The radiolysis of water by gamma irradiation generates active radicals (e^-, $HO^•$, $H^•$, H_2, and H_2O_2). They readily react with large biopolymer molecules in biomass such as lignin, cellulose, and hemicellulose to obtain smaller carbon molecules [23-25]. Moreover, the radiation process was proved to modify the physicochemical and optical properties of CDs [26-28]. However, a few previously literature reported the synthesis of CDs by the gamma irradiation process.

The water hyacinth (*Eichhornia crassipes*) is a problematic invasive aquatic plant that hinders the water flow and reduces the dissolved oxygen in Nakhon Nayok and Pathum Thani provinces. The conversion of water hyacinth to high-value compounds such as biochars [29], reinforcement fibers [30], biopolymers [31], and CDs [32] can be a promising solution. In this research, water hyacinth stalks were selected as the carbon source for CDs synthesis. The active radicals promote the top-down synthesis of CDs from water hyacinth *via* gamma radiolysis without heat and toxic chemicals. PVA-CDs composite film was applied for the UV blocking film.

2. Materials and Methods

2.1 Materials and Instruments

Water hyacinth stalks were collected from Pathumthani and Nakhon Nayok Province, Thailand. All chemicals were used as received without further purification. Poly(vinyl alcohol) (PVA, $M_W \approx 145{,}000$ kDa) was purchased from Sigma-Aldrich. Deionized water ($R > 18$ M·Ω·cm^{-1}) was used in all experiments. Gamma irradiation was obtained from cobalt-60, JS 8900 IR-155 (Nordion). UV-Vis absorption spectra were collected on a PerkinElmer Lambda 35 UV/Visible spectrometer. Fluorescence emission spectra were performed on Hitachi F-4600 fluorescence spectrophotometer. The zeta potential and particle size were measured on a Malvern zeta sizer Ultra. Fourier-transformed infrared spectroscopy (FTIR) spectra were recorded on Thermoscientific Nicolet is5/iD7 ATR in the range of 4,000 cm^{-1} to 500 cm^{-1}. The film thickness was determined on a Mitutoyo (JIS, JQA, No.103-137) micrometer.

2.2 Preparations of carbon dots (CDs)

The green powder of water hyacinth stalk was prepared by drying under sunlight for 3 days and in an oven at 80°C for 2 days, respectively. Then the material was ground by electronic blender for 2 times to 3 times until it appeared as powder. CDs were prepared from the powder of water hyacinth stalk by using gamma irradiation [32]. Briefly, the mixture of powder (0.5 g) and deionized water (25 mL) was sonicated under ultrasonic wave for 30 min and then pursue nitrogen gas for 30 min, respectively. The resulting mixture was irradiated by gamma-ray at the dose of 80 kGy. Firstly, the as-received yellow solution was filtrated by the filter paper and syringe filter 0.2 μm, respectively. Then, the filtrated solution was dialyzed in deionized water by dialysis bag (1 kDa) for two days and then freeze-dried to provide CDs as a yellow solid.

2.3 Preparation of PVA and PVA-CDs composite film

CDs (30 mg) were dissolved in deionized water (10 mL) with the ultrasonic treatment for 30 min. The mixture of poly(vinyl)alcohol (PVA, 1.5 g) in deionized water (20 mL) was heated with the vigorous stirring at 98°C for 2 h. After that, the CDs solution was added dropwise to the PVA solution with constant stirring at 98°C for 1 h to provide the homogenous solution. The resulting solution was poured onto a silicone mold and then dried in the oven at 60°C for two days to provide PVA-CDs composite film. The blank PVA film was prepared under the same condition. The mixture of poly(vinyl)alcohol (PVA, 1.5 g) in deionized water (30 mL) was heated with the vigorous stirring at 98°C for 2 h.

3. Results and Discussion

3.1 Characteristic properties of CDs

CDs were prepared from water hyacinth stalks in a one-step gamma irradiation, regarding the previous literature [32]. The yellow CDs solution strongly absorbed the UV region (250 nm to 400 nm) with the maximum absorption at 270 nm as a result of $\pi \rightarrow \pi^*$ transition of C=C bonds [33] (Figure 1). Furthermore, the CDs solution displayed fluorescence emission in the visible region by the several excitation wavelengths at 320 nm to 500 nm and exhibited maximum fluorescence intensity at 439 nm with the excitation at 360 nm, corresponding to blue emission. The particle size and zeta potential of CDs in deionized water were determined by the dynamic light scattering (DLS) method (Figure 2). The average particle size of CDs was 236 ± 32 nm from the DLS spectrum. The zeta potential of CDs was -39.8 ± 4.6 mV, suggesting the negative surface charge because of hydroxyl groups on the surface, in accordance with FTIR data [32]. These results indicated that CDs could be applied in the polymer composite film due to their excellent optical properties, including UV absorption and strong visible-light emission, particle sizes in the nanometer, and water solubility.

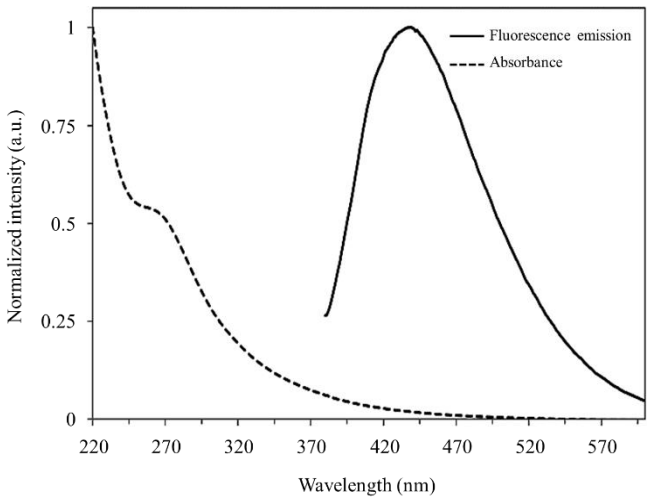

Figure 1. The normalized UV-Vis absorption and fluorescence emission spectra (λ_{ex} = 360 nm) of CDs (1.0 mg·mL^{-1}) in deionized water.

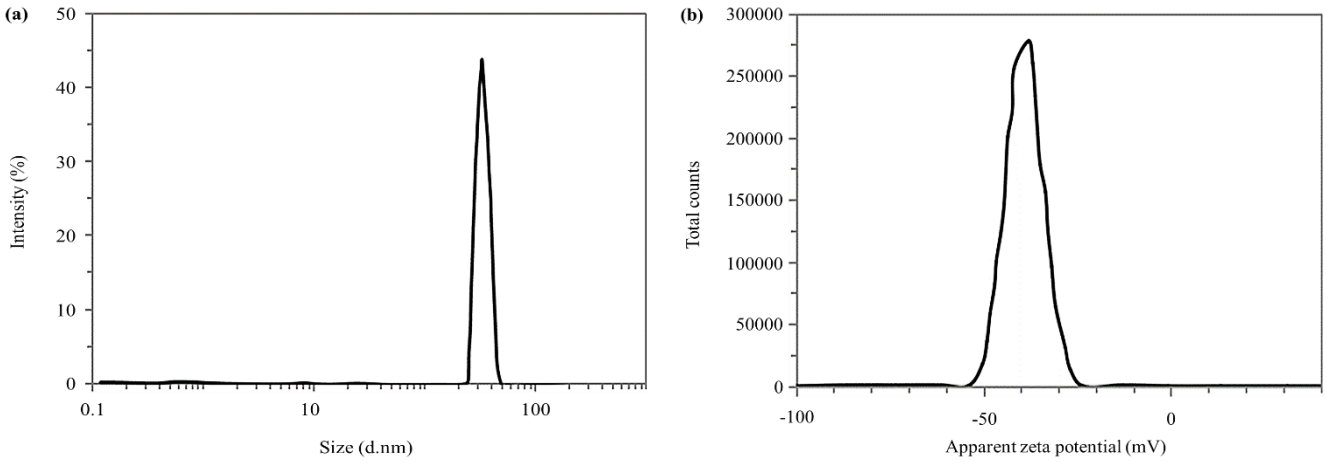

Figure 2. (a) DLS spectrum (size distribution by intensity), and (b) Zeta potential spectra of CDs in deionized water (1.0 mg·mL^{-1}).

3.2 Characterization of PVA-CDs composite film

Poly(vinyl)alcohol (PVA) was suitable as a polymer matrix due to the mechanical properties (high-strength and high-modulus) and biodegradable polymer [34,35]. For the fabrication of the polymer composite film with CDs (2% w/w), the CDs solution was added to the stirring PVA solution at 98°C. The blank PVA film was also produced under the same condition. The PVA-CDs composite film appeared a yellow transparent film and enhanced fluorescence intensity under UV light at 365 nm compared to the PVA film (Figure 3). The film thickness of PVA-CDs and PVA films was 133.3 ± 2.9 μm and 156.7 ± 10.4 μm, respectively.

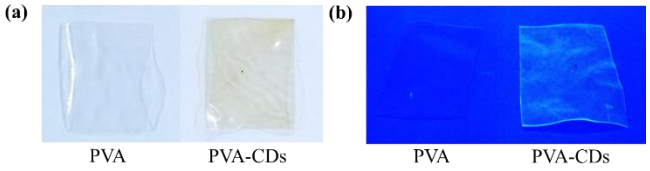

Figure 3. Photographs of PVA and PVA-CDs (2.0% w/w) composite films under (a) visible light, and (b) UV irradiation at 365 nm.

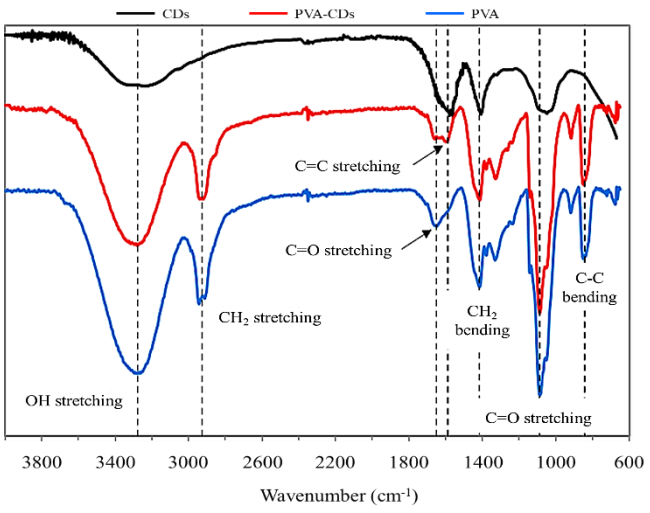

Figure 4. FTIR spectra of CDs, PVA and PVA-CDs composite films

FTIR spectra of CDs, PVA, and PVA-CDs composite films were also elucidated (Figure 4). FTIR spectrum of PVA film showed several absorption peaks such as 3275 cm^{-1} (O-H stretching of hydroxyl groups), 2937 cm^{-1} (CH_2 stretching of alkyl groups), 1651 cm^{-1} (C=O stretching of acetate group of PVA), 1415 cm^{-1} (CH_2 bending), 1091 cm^{-1} (C-O stretching), and 841 cm^{-1} (C-C stretching of characteristic skeletal PVA) [36,37]. FTIR spectra of PVA-CDs composite film displayed intense absorption peaks including 3289 cm^{-1} (O-H stretching of hydroxyl groups), 2920 cm^{-1} (CH_2 stretching of alkyl groups), 1652 cm^{-1} (C=O stretching of acetate group of PVA), 1589 cm^{-1} (C=C stretching of CDs, 1420 cm^{-1} (C-O stretching), 1111 cm^{-1} (C-O stretching), and 841 cm^{-1} (C-C stretching of characteristic skeletal PVA). The FTIR spectrum of CDs exhibited an intense peak at 1570 cm^{-1}, which was attributed to the C=C stretching of sp^2 carbons according to PVA-CDs composite film. The O-H stretching of PVA-CDs film appeared a slight shift at 3286 cm^{-1} compared to the PVA film (3275 cm^{-1}), suggesting that intermolecular interaction of PVA matrix and CDs was observed *via* hydrogen bonding according to the previous literature [38].

The absorption spectra showed that the PVA-CDs composite film remarkably absorbed the UV light with the maximum absorption peak at 272 nm due to the $\pi \rightarrow \pi^*$ transition of CDs. The observed absorption spectra of PVA-CDs composite film were similar to the CDs solution. The PVA film slightly absorbed in the UV region (Figure 5(a)). For the transmittance spectra, PVA-CDs composite film effectively transmitted 91% of visible light compared to 9% of UV light, whereas a PVA film strongly transmitted in UV-visible wavelength (Figure 5(b)). The PVA-CDs composite film showed fluorescence emission in the UV-Visible region (300 nm to 700 nm) by excitation wavelength at 240 nm to 500 nm (Figure 6(a)). In contrast, the blank PVA film only displayed characteristic fluorescence emission peaks in the UV region with the excitation range of 240 nm to 320 nm (Figure 6(b)). These results indicated that the PVA polymer matrix displayed fluorescence peaks in the UV region of the fluorescence spectra for the PVA-CDs composite film, corresponding to the previous literature [39]. Moreover, the maximum fluorescence emission wavelength of the PVA-CDs composite film was slightly red-shift compared with the solution of CDs because of the PVA film environment [13]. Additionally, the intermolecular interaction of CDs and the PVA matrix affected the fluorescence behavior of PVA-CDs composite film [40].

The spectral composition of PVA-CDs and PVA films were calculated from integrated fluorescence intensity spectra in several color ranges, including violet (380 nm to 450 nm), blue (450 nm to 495 nm), green (495 nm to 570 nm), yellow (570 nm to 590 nm), orange (590 nm to 650 nm), and red (650 nm to 700 nm). The spectral composition by the excitation at 360 nm showed that the blue-to-red spectral composition of PVA-CDs and PVA films was 57.2% and 35.7%, respectively (Figure 6). Therefore, the 21.5% increase in blue-to-red spectral of PVA-CDs composite film compared to the PVA film. The results indicated that the PVA-CDs composite film might be applied in the agriculture polymer film based on light conversion by UV absorption and fluorescence emission in visible light, increasing photosynthesis and plant growth [41].

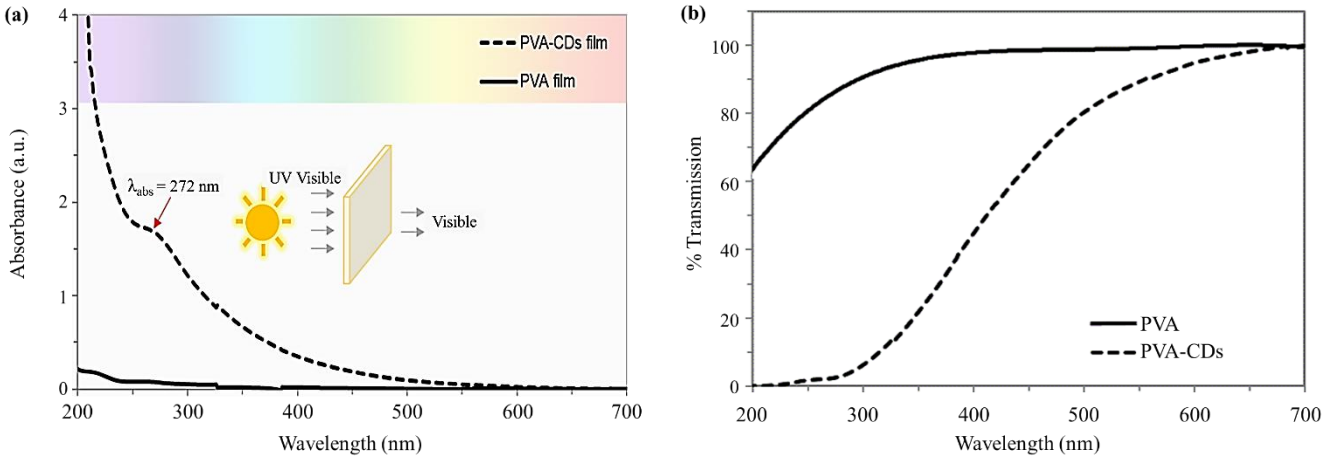

Figure 5. (a) UV-Vis absorption, and (b) transmittance spectra of PVA and PVA-CDs composite film.

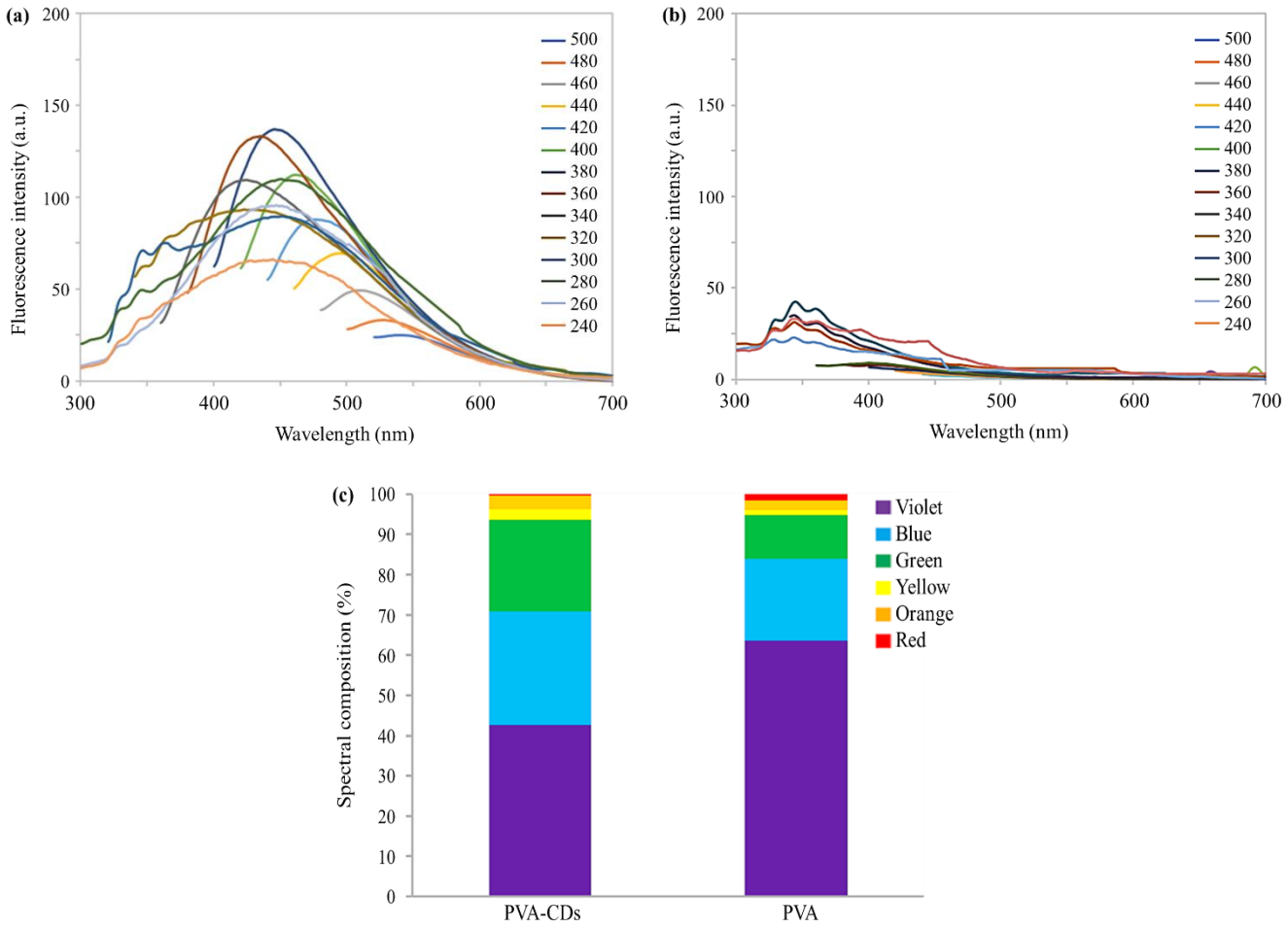

Figure 6. Fluorescence emission spectra of (a) PVA-CDs composite film (b) PVA film, and (c) spectral composition of PVA-CDs and PVA films by excitation at 360 nm.

4. Conclusions

In summary, poly(vinyl)alcohol-carbon dots composite film was successfully developed for UV blocking film. Gamma radiolysis was used for the green synthesis of carbon dots from water hyacinth stalks. The poly(vinyl)alcohol- carbon dots composite film exhibited a yellow transparent film and strong UV absorption. The composite film showed high transmittance in the visible region (400 nm to 700 nm, 91%) compared to that of the UV region (200 nm to 400 nm, 9%). The composite film displayed fluorescence emissions in the visible region by different excitation wavelengths, increasing the blue-to-red spectral composition by 21.5% compared with the PVA film. The intermolecular interaction between the hydroxyl group of the PVA matrix and the carboxyl group of carbon dots was hydrogen bonding, resulting in the slightly red-shift fluorescence emission.

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