

# A COMPARATIVE STUDY OF CASSAVA PULP AND COMMERCIAL-GRADE CDS/LDPE COMPOSITE FILMS WITH APPLICATIONS IN PLANTS

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## Abstract

Carbon dots (CDs) synthesized from cassava pulp using a green hydrothermal method have gained attention for several applications. CDs in composite films were used for UV blocking due to their outstanding optical properties, which include high UV absorbance and photoluminescence. In this study, low-density polyethylene (LDPE) was mixed with 2 wt% CDs to form composite films via a solvent-casting technique. Physical, chemical, and optical characteristics were examined using a Zetasizer, FTIR spectrometer, UV-vis spectrophotometer, and UV transilluminator to compare cassava pulp carbon dots (CDs) with commercial-grade carbon dots (CDs-c). Subsequently, the optical properties of 2% CDs/LDPE composite films were compared with 2% CDs-c/LDPE composite films. UV blocking of the films was tested by measuring chlorophyll content and plant growth. The results suggested that CDs had optical characteristics comparable to CDs-c. The 2% CDs/LDPE composite films exhibit nearly identical UV absorbance to the 2% CDs-c/LDPE composite films. Yellow fluorescence of the two composite films is caused by the sensitivity of the carbon core structure along with the hydroxyl and carboxyl groups on the surfaces of CDs. Applications of composite films showed that plants protected from UVC by CDs/LDPE films had high chlorophyll content due to high UVC absorbance and greater light transmission. However, plants protected by CDs-c/LDPE film with low light transmission did not show affected chlorophyll. Although 2% CDs and CDs-c/LDPE films have high UVC absorbance, their UV-C blocking was limited.

**Keywords:** Green carbon dots; Cassava pulp; Low-density polyethylene; Composite film

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## Introduction

Carbon dots (CDs) derived from agricultural waste, plants, and other natural organic substances are zero-dimensional nanocarbon materials synthesized by green methods. These procedures include dry heating, hydrothermal, microwave-assisted, solvothermal, and ultrasonic approaches, using water or organic solvents (Chahal *et al.*, 2021; Jing *et al.*, 2023). They are also known as green carbon dots and have gained attention for several applications in bioimaging, chemical sensing, drug delivery, and photocatalysis (Chahal *et al.*, 2021; Jing *et al.*, 2023). Technical interest stems from their unique properties, including high UV absorbance, strong fluorescence, inertness, low toxicity, and high biocompatibility (Jing *et al.*, 2023; Liu *et al.*, 2020; Ozyurt *et al.*, 2023).

Currently, many researchers are developing composite films with CD content for use in food packaging and UV-blocking applications. This is due to their outstanding optical properties with high UV absorbance and photoluminescence (Taspika *et al.*, 2019; Zulfajri *et al.*, 2021). There are diverse types of polymers used as matrix-based materials to create composite films, for example, polyvinyl alcohol (PVA), polymethyl methacrylate (PMMA), polyethersulfone (PES), polystyrene (PS), and other polymers (Chen and Feng, 2015; Kwamman *et al.*, 2021; Zulfajri *et al.*, 2021). Recent research shows the essential details of the preparation steps, film properties, and potential applications (Chen and Feng, 2015; Kwamman *et al.*, 2021; Zulfajri *et al.*, 2021). These materials can improve packaging and provide UV protection technology.

Cassava waste, an agricultural residue after harvest, is a significant carbon source for producing CDs (Prakobboon *et al.*, 2019). The volume of cassava waste depends on the quantity of cassava produced in the country. In 2015, there were at least 7.3 million metric tonnes of cassava pulp waste in Thailand (Trakulvichean *et al.*, 2017). Most Thai post-harvest cassava wastes consist of stems and rhizomes that are usually burned in fields. This is estimated to be 66% of the total weight of cassava disposal (Prakobboon *et al.*, 2019). Such burning leads to the current air pollution problems. PM<sub>2.5</sub> particles are of great concern. They can harm human health and cause death resulting from respiratory diseases and cancer (Xing *et al.*, 2016). Thus, producing CDs instead of burning cassava waste can aid in mitigating environmental pollution and increase the value of this agro-waste.

This research aims to estimate a potential application of a CDs/LDPE composite film from cassava waste CDs produced from a green

hydrothermal method, combined with low-density polyethylene (LDPE). Its optical properties are compared with a CDs-c/LDPE composite film derived from commercial-grade carbon dots. No research has reported a comparative study of the properties of a composite film obtained from agricultural waste and commercial-grade carbon dots.

## Materials and Methods

### Materials

Two types of CDs were examined in this research, those made from agricultural waste and commercial-grade CDs. Cassava pulp was used as a carbon source for synthesizing CDs. It was received from a local cassava farm in Kanchanaburi, Thailand. Commercial-grade carbon dot (CDs-c) powder was supplied by Jiangsu XFNANO Materials Tech. Co., Ltd., China. Additionally, three chemicals were also used in the preparation of CDs and composite films. These are low-density polyethylene (PETLIN LD C150Y) purchased from PETRONAS Chemicals Group, Berhad, Malaysia. Xylene (Analytical Grade) was purchased from PanReac Quimica SLU, Spain. This solvent was used to dissolve and cast the LDPE film. Deionized water was used in all CD synthesis processes.

### Preparation of CDs

CDs were synthesized by a green hydrothermal approach. Initially, a cassava powder carbon source was prepared by drying in an oven at 60°C for 24 h, grinding with an electronic blender, and sieving through a 200-mesh screen. Subsequently, 4 g of dried cassava powder was mixed with 200 ml of deionized water and sonicated for 30 min. The mixture was poured into a Teflon container serving as the inner part of a hydrothermal reactor. This reactor was placed in an oven and heated at 200°C for 24 h (Watcharamongkol *et al.*, 2024). Filtration of the synthesized solution was performed using filter paper with 0.45- $\mu$ m pores and a membrane filter with 0.2- $\mu$ m pores. Last, the CD supernatant was dialyzed in deionized water for 48 h before freeze-drying (DW 1,0-60E, Heto Drywinner, Denmark) at -50°C to produce a light-brown dried powder (Watcharamongkol *et al.*, 2024).

### Preparation of Composite Films

All composite films were prepared using a solvent-casting method. First, LDPE, CDs, CDs-c, and xylene were used as precursors for film casting.

**Table 1. Mixture ratios of composite films**

| Formula  | Composition |           |          |             |
|----------|-------------|-----------|----------|-------------|
|          | CDs (g)     | CDs-c (g) | LDPE (g) | Xylene (ml) |
| LDPE     | -           | -         | 1        | 45          |
| 2% CDs   | 0.02        | -         | 0.98     | 45          |
| 2% CDs-c | -           | 0.02      | 0.98     | 45          |

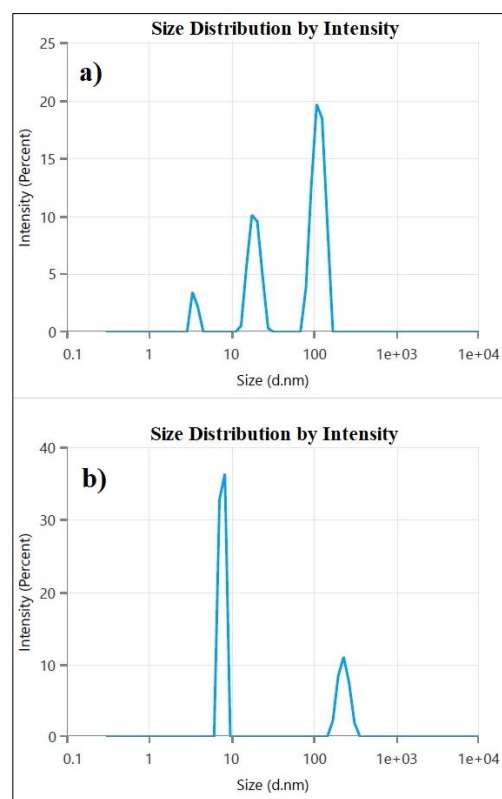
The mixture ratios of these composite films are defined in Table 1. There were three formulae, LDPE (blank film), 2% CDs, and 2% CDs-c. All film precursors were weighed as formulated. Subsequently, they were each dissolved in xylene and stirred at 400 rpm and 100°C for 3 h. Each solution was sonicated at 80°C for 30 min. Ten ml of each solution was pipetted into a glass Petri dish, placed in a vacuum oven, and dried at 90°C for 1 h. Subsequently, the produced powders were kept at room temperature for 24 h before removal from the Petri dish. The film thicknesses were approximately 0.02–0.03 mm.

#### Characterization of CDs and Composite Films

Particle sizes of the CDs were determined using a Zetasizer (Lab model, Malvern Panalytical, UK). CDs and composite films were subjected to functional group analysis under Fourier transform infrared (FTIR) spectrometry (Spectrum Two model, PerkinElmer Scientific, USA). The ATR mode was selected for observations in the 500–4,000  $\text{cm}^{-1}$  wavenumber range (Kwamman *et al.*, 2021). The UV-Vis spectrophotometer (UV5 Excellence Model, METTLER TOLEDO, USA) detected the UV absorbance of CDs and composite films in the 190–800 nm wavelength range. Transmittance in the same wavelength range was determined in a UV-absorbance experiment. The fluorescence of CDs and composite films was tested with a UV transilluminator (Lampada minUVIS, Desaga, Germany) at a 366 nm wavelength.

#### Application of Composite Films for Plants

Selected seedlings of Chinese cabbages (*Brassica rapa* L.) having a 5.0 mm stem length were transferred to 96 well-microplates with three replications. Each well received 150  $\mu\text{L}$  of 1/4 strength Hoagland's nutrient solution (Wang *et al.*, 2022), as a medium. The microplate was placed in a black plastic box with a film (LDPE, 2% CDs or 2% CDs-c) cover and then transferred to allow growth at 25 °C for 12 h under visible light with 6 h of UVC (3 h visible light + 6 h visible light and UVC + 3 h visible light) or 12 h of visible light with UVC (12 h visible light and UVC). The seedlings were evaluated after 2 and 4 days to determine stem and root lengths, fresh and dry weights, and chlorophyll contents. Seedling leaves were used to extract



**Figure 1. The particle size distribution of a) CDs, and b) CDs-c**

chlorophyll which was spectrophotometrically measured (Hiscox and Israelstam, 1979). Experimental data were evaluated using one-way ANOVA with Duncan's Multiple Range Test (DMRT) to investigate statistical differences in LDPE, 2% CDs, and 2% CDs-c films using R programming.

## Results and Discussion

#### Comparative properties of CDs and CDs-c

As shown in Figure 1, the particle size distribution, the z-average size, and the polydispersity index (PI) of CDs and CDs-c were evaluated using a Zetasizer. The CD results showed three peaks of different sizes, 3.566, 19.07, and 114 nm, with a z-average size of 96.01 nm.

Concurrently, CDs-c displayed two peaks, measuring 7.751 and 230.7 nm, with an overall z-average size of 0.1074 nm, indicating that the CD particle size was larger than the CDs-c. This is aligned with the PI of these particles. CDs with a 0.3124 PI value exhibited a higher degree of particle size non-uniformity than CDs-c, which had a 0.143 PI value (Danaei *et al.*, 2018).

The functional groups on the CDs and CDs-c surfaces in Figure 2 were analyzed using FTIR spectrometry. Both CDs and CDs-c exhibited O-H stretching vibration peaks at 3,310  $\text{cm}^{-1}$  (CDs) and 3,417  $\text{cm}^{-1}$  (CDs-c),  $\text{sp}^3$  C-H stretching vibration peaks at 2,925  $\text{cm}^{-1}$  (CDs) and 2,960  $\text{cm}^{-1}$  (CDs-c), and C=O stretching vibration peaks at 1,639  $\text{cm}^{-1}$  (CDs) and 1,702  $\text{cm}^{-1}$  (CDs-c). Their surfaces consisted of hydroxyl and carboxyl groups, which correlated with the functional groups and the surface state to enhance the optical properties (Kang *et al.*, 2020; Nima *et al.*, 2020; Watcharamongkol *et al.*, 2024). Additionally, there were differences in some vibration peaks of CDs and CDs-c. For CDs, strong C-O stretching vibration peaks were observed at 1150, 1079, and 1019  $\text{cm}^{-1}$ . CDs-c showed a strong C=C stretching vibration peak at 1,661  $\text{cm}^{-1}$ , a C-H bending vibration peak at 1,449  $\text{cm}^{-1}$ , an O-H bending vibration peak at 1,367  $\text{cm}^{-1}$ , and C-N stretching vibration peaks at 1,162 and 1,037  $\text{cm}^{-1}$  (Chandra *et al.*, 2016; Watcharamongkol *et al.*, 2024).

The UV absorbance and fluorescence of CDs and CDs-c were observed under UV-Vis spectrophotometry and a UV transilluminator. These results are shown in Figures 3 and 4. Figure 3 presents the UV absorbance spectra of both CDs and CDs-c. Both showed strong absorbance in the UV region (200-400 nm wavelengths), especially in the UV-C range of 200-280 nm (Zhao *et al.*, 2022). The maximum absorbance peak of CDs was observed at a 325.4 nm wavelength, corresponding to the  $n \rightarrow \pi^*$  transition of C-O and C=O bonding. The CDs-c absorbance peak, however, was seen at a 242.6 nm wavelength, which is related to the  $\pi \rightarrow \pi^*$  transition of C=N and C=C bonding (Kang *et al.*, 2020; Nima *et al.*, 2020; Watcharamongkol *et al.*, 2024). The FTIR spectra in Figure 2 confirm the bonds on the surfaces of CDs and CDs-c. Figure 4 displays the bright blue fluorescent emissions of both CDs and CDs-c at a 366 nm wavelength. This is in agreement with the general fluorescence emission from the carbon sources of biomass that emit both blue and green light (Kanwal *et al.*, 2022). The fluorescent potential of both the CDs and CDs-c was similar. Furthermore, under white light, the CDs solution exhibited a light-yellow color while the CDs-c solution was light-brown.

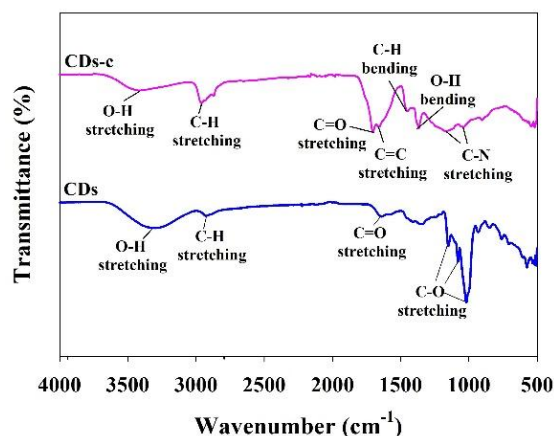


Figure 2. FTIR spectra of CDs and CDs-c

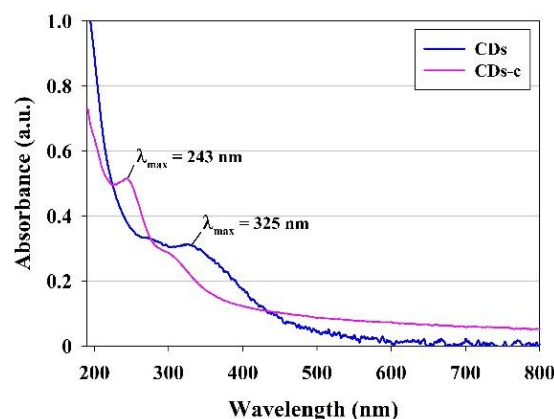


Figure 3. UV-Vis absorption spectra of CDs and CDs-c solutions

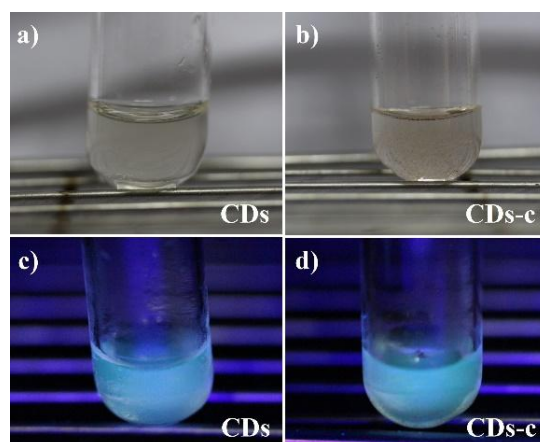


Figure 4. Fluorescent images of CDs and CDs-c under a-b) white, and c-d) UV light

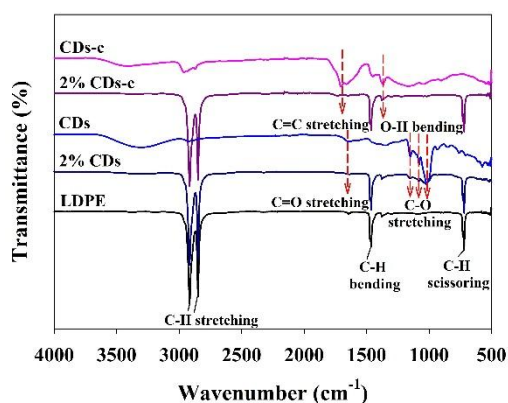


Figure 5. FTIR spectra of LDPE, 2% CDs/LDPE, and 2% CDs-c/LDPE films

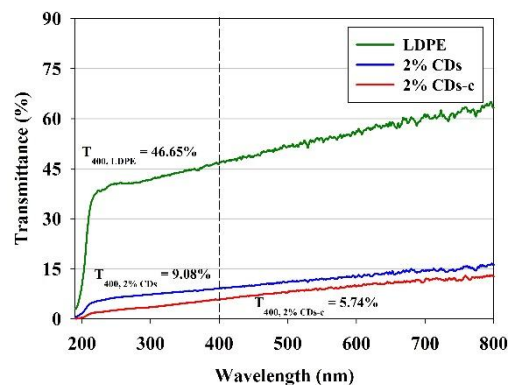


Figure 7. UV-Vis transmittance spectra of LDPE, 2% CDs/LDPE, and 2% CDs-c/LDPE films

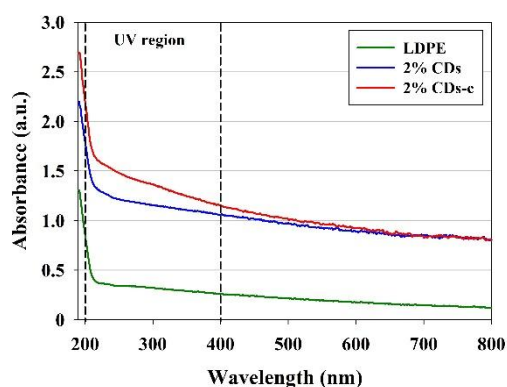


Figure 6. UV-Vis absorption spectra of LDPE, 2% CDs/LDPE, and 2% CDs-c/LDPE films

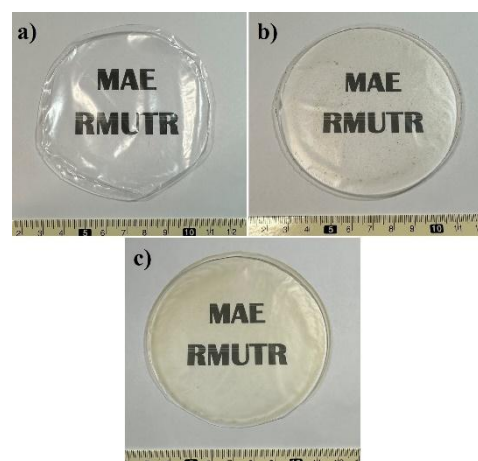


Figure 8. Digital images of a) LDPE, b) 2% CDs/LDPE, and c) 2% CDs-c/LDPE films under white light

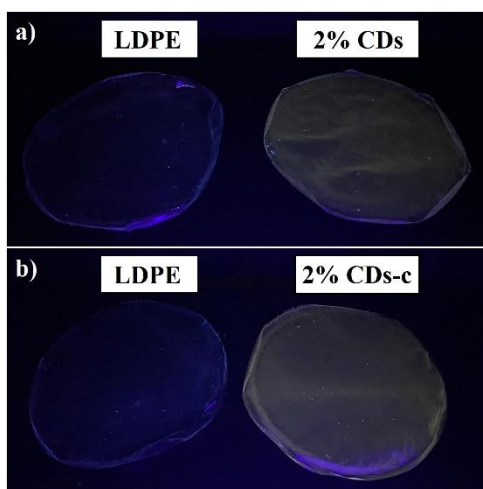
### Comparative Properties of Composite Films

The FTIR spectra of all sample films are shown in Figure 5. Strong C-H stretching, C-H bending, and C-H scissoring vibration peaks were observed at 2,916, 2,848, 1,463, and 719  $\text{cm}^{-1}$ , respectively, on the surface of the blank LDPE film. These vibration peaks were also detected in both 2% CDs/LDPE and 2% CDs-c/LDPE films, resulting from the 98 wt.% of LDPE within the composite films. On the surface of the 2% CDs/LDPE film, carboxyl groups were present exhibiting a C=O stretching vibration peak at 1,633  $\text{cm}^{-1}$  and C-O stretching vibration peaks at 1,152, 1,080, and 1,024  $\text{cm}^{-1}$ . The position of the C=C stretching vibration peak was at 1,646  $\text{cm}^{-1}$ , and the O-H bending vibration peak was at 1,377  $\text{cm}^{-1}$  on the surface of the 2% CDs-c/LDPE film. These results indicated that the functional groups of CDs

and CDs-C may play an important role in increasing the optical properties of both composite films.

Figure 6 exhibits the UV absorbance spectra of all specimen films. The UV absorbance of both the 2% CDs/LDPE and 2% CDs-c/LDPE films was significantly higher than that of the LDPE film. This is because CDs and CDs-c were present on the surfaces of the films, as shown in Figure 5. Moreover, the UV absorbance efficiency of 2% CDs/LDPE was nearly that of 2% CDs-c/LDPE film. Addition of CDs and CDs-c increased the UV absorbance in composite films, as explained in the discussion of UV absorbance of CDs and CDs-c, presented above.

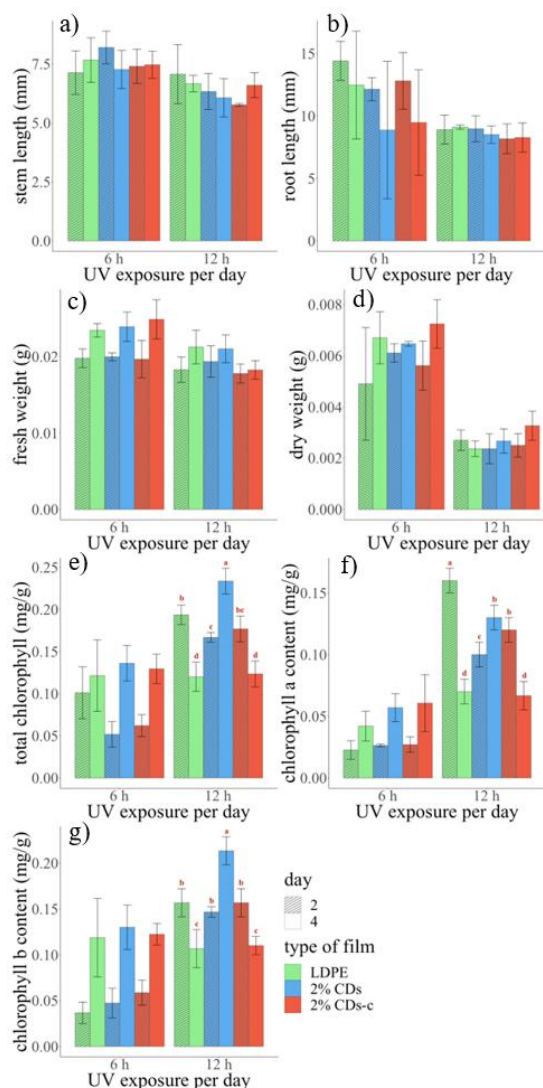
Figure 7 illustrates the UV transmittance of all films at a 400 nm wavelength ( $T_{400}$ ) (Zhao *et al.*, 2022). UV transmittance was 46.65% for the LDPE film, 9.08% for the 2% CDs/LDPE film, and 5.74%



**Figure 9.** Fluorescent images of a) LDPE and 2% CDs/LDPE films, and b) LDPE and 2% CDs-c/LDPE films under UV light

for the 2% CDs-c/LDPE. The UV transmittance of both composite films was lower than that of the LDPE film, likely due to the UV-blocking properties of the CDs and CDs-c particles in the composite films, which are active in the UV range from 200-400 nm (Zhao *et al.*, 2022). These results are in accordance with the transparency of the sample films overlaying the black text in Figure 8. They showed high transparency under white light, although these composite films had a low value of UV transmittance. However, CD accumulation as small clusters on the 2% CDs/LDPE film can result in a lower UV-blocking capability compared to the 2% CDs-c/LDPE film, which had a more uniform particle distribution, as shown in Figure 8. The 2% CDs-c/LDPE film appeared more homogeneous, while the 2% CDs/LDPE film had poor particle distribution and small clusters from CD accumulation on its surface. Furthermore, Figure 9 presents the fluorescent emissions of all films at a 366 nm wavelength. Both composite films emitted a light-yellow fluorescence, while the blank film lacked fluorescence. CDs and CDs-c were still active under UV light, although they were mixed with LDPE to form composite films.

Figure 10 shows the protective effects of carbon dot films from UVC on plants. Under 12 h/day UVC, Chlorophyll a, b, and total chlorophyll levels in plants protected by 2% CDs/LDPE films were higher than under LDPE films. This result implies that CD particles in the composite films absorbed UVC, which can cause chlorophyll degradation in plants. At a high UVC level, CD films can protect plants from UVC



**Figure 10.** Plant response to UVC exposure for 6 and 12 h protected by LDPE, 2% CDs/LDPE, and 2% CDs-c/LDPE films determining a) stem length, b) root length, c) fresh weight, d) dry weight, e) total chlorophyll content, f) Chlorophyll a content, and g) Chlorophyll b content

degradation as indicated by the significant difference in chlorophyll contents between plants protected by CD film and control film. Although 2% CDs-c/LDPE films absorbed more UVC than CDs/LDPE films (Figure 6), Chlorophyll a and b as well as the total chlorophyll contents in plants protected by CDs-c/LDPE films were lower than CDs/LDPE films. This is likely due to the low light transmission of CDs-c/LDPE films, which affects plant biosynthesis of chlorophyll. Under 6 h/day UVC, there were no significant differences in

chlorophyll contents between plants protected by carbon dot films and the control. So, 6 h/day UVC in this experiment was not sufficient for chlorophyll destruction in these plants. Moreover, 12 h/day UVC damaged plant chlorophyll but it did not subsequently affect plant growth.

## Conclusions

Both CDs and CDs-c exhibited a strong blue fluorescence. The fluorescence of 2% CDs/LDPE and 2% CDs-c/LDPE composite films emit yellow light. However, CDs and CDs-c solutions had different UV absorbance spectra. The 2% CDs-c/LDPE composite films have slightly higher UV absorbance than the 2% CDs/LDPE composite films. Yellow fluorescence of these two composite films is induced by the carbon core structure's sensitivity, as well as the hydroxyl and carboxyl groups on CD surfaces. CDs/LDPE films decreased the effects of UVC on chlorophyll degradation with high light transmission, which promotes high chlorophyll contents. Although blocking UVC, the CDs-c/LDPE film limited light transmission, which influenced the low chlorophyll contents.

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