

## Comparing Three Methods For Producing Carbon Dots From Mangosteen Peel

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**Abstract:** Carbon dots are fluorescent nanoparticles that are around 10 nm in size. Carbon dots can be formed via pyrolysis, hydrothermal, and solvothermal procedures from raw materials such as mangosteen peels. Because it contains cyanidin and xanthone, which improve the intensity of carbon dot fluorescence, mangosteen peel waste can be utilized to make carbon dots. The presence of a urea passivation agent is expected to boost carbon dot luminescence intensity. The study aimed to develop carbon dots from mangosteen peel using three different methods: pyrolysis, hydrothermal, and solvothermal, and to assess their ability to produce luminous hues. Carbon dot yield was 21% by the solvothermal method, 5% by the hydrothermal method, and 2% by pyrolysis. All three methods produced blue carbon dot luminescence. The solvothermal method, hydrothermal procedure, and pyrolysis had the highest luminescence intensity. Adding urea as a passivation agent increased the luminescence of carbon dots. The solvothermal approach produced the highest carbon dot production and fluorescence intensity. The hydrothermal and solvothermal carbon dots made emissions at wavelengths of 413 nm and 454 nm, respectively, both corresponding to blue luminescence.

**Keywords:** Hydrothermal; Mangosteen Peel; Pyrolysis; Solvothermal; Urea.

### Introduction

A carbon dot (c-dot) is a nanocrystal with dimensions of  $>10$  nm [1]. C-dots comprise a  $sp^2/sp^3$  hybridized carbon framework with surface functional groups [2]. The fluorescent properties of c-dots make them often used in drug delivery, sensors, photocatalysts, and detecting harmful compounds in food [3]. The advantages of c-dots are low toxicity, good photostability and conductivity, soluble in water, and environmentally friendly [4].

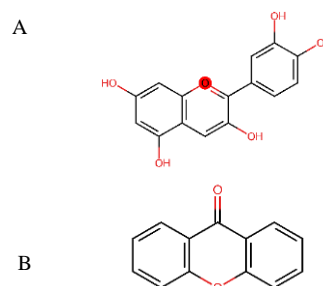
C-dots can be produced from natural carbon sources at low temperatures, allowing for more sustainable, less expensive, and more straightforward synthesis techniques [5]. In recent years, agricultural waste precursors have become popular because they are environmentally friendly and cheap, like mangosteen fruit peel waste [4].

Mangosteen peel contains anthocyanins, polyphenols, flavonoids, carbohydrates, calcium, phosphorus, proteins, lignin, hemicellulose, and cellulose. Polyphenolic compounds such as xanthenes and anthocyanins in the form of cyanidin are most widely contained in the mangosteen peel [6].

Both substances are hypothesized to be sources of functional groups that can enhance the presence of carbonyl and hydroxyl groups on the surface of the resultant carbon dot, increasing the fluorescence intensity (Figure 1). Judging from its structure, the compound has a conjugated double bond that can absorb light in the visible light area. Electronic conjugation and delocalization properties are caused by different ionization states and molecular rearrangements in the anthocyanin structure [7].

Passivation agents such as urea are also added to increase the luminescence produced [8-9]. Incorporating urea can increase the number of nitrogen atoms on the surface of a carbon dot. Functionalized nitrogen atoms on the surface can alter the density of the carbon dot's surface state, increasing fluorescence intensity [9]. Therefore, cyanidin and xanthone compounds and the addition of passivation are expected to increase the fluorescence intensity due to resonance within the structure or shift the fluorescence wavelength to a color other than blue.

Carbon dot synthesis can be done using several methods, such as hydrothermal, solvothermal, and pyrolysis. Previous research has been undertaken using pyrolysis to produce carbon dots from mangosteen peel [5]. However, the study has not been carried out to synthesize carbon dots using hydrothermal and solvothermal methods, and the results of carbon dot synthesis with mangosteen fruit peel from the three synthesis methods have not been compared. The method is classified as an environmentally friendly bottom-up synthesis with good photostability, conductivity, and fluorescence intensity [10].



**Figure 1.** Structure of Cyanidin (A) and Xanthone (B)

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## Research Methods

### Sampling Techniques

Samples of mangosteen peel used were obtained from various varieties taken in wet and fresh state and then washed thoroughly.

### Sample Preparation

The cleaned and chopped mangosteen peel is dried in an oven at 150 °C for 3 hours. The dried peel is smoothed and filtered using a 100-mesh sieve.

### Determination of Mangosteen Peel Water Content by AOAC 2016

The porcelain cup was dried at 104 °C for 30 minutes and then chilled in a desiccator for 30 minutes before being weighed till it reached a steady weight. 5 g of fresh samples were placed in a saucer and dried at 104 °C for 5 hours before being chilled in a desiccator and weighed. The step is repeated three times until a fixed weight is obtained. The equation is used to determine the moisture content,

$$\text{Water content} = \frac{A-B}{A} \times 100\%$$

Information:

A = Weight of mangosteen peel before drying (g)

B = Weight of mangosteen peel after drying (g)

### Pyrolysis Carbon Dot Synthesis Method

Mangosteen peel powder weighed 15g, then powdered in a porcelain dish with Bunsen; urea 2.5 g is added as a passivation agent for samples that have become ash. The mixture is reheated in the kiln at 350 °C for 2 hours. Inside the desiccator, the porcelain cup is cooled to room temperature. The recovered ash was mixed with water before being sonicated for 10 minutes. The procedure is repeated in the same stages without adding urea and a temperature of 700 °C for 2 hours.

### Hydrothermal Carbon Dot Synthesis Method

Mangosteen peel powder weighed as much as 15 g mixed with 100 mL H<sub>2</sub>SO<sub>4</sub> 0.1 M for 30 minutes, then rinsed with water until neutral. The sample is then filtered and dried in the oven at 150 °C for 2 hours. Mangosteen peel powder is then added to 2.5 g of urea and mixed with distilled water 100 mL. The mixture is placed in a Teflon-lined autoclave and heated in a 200 °C oven for 8 hours. The autoclave is allowed to cool to ambient temperature. The same procedure is repeated at the same stage without adding urea.

### Solvothermal Carbon Dot Synthesis Method

Mangosteen peel powder weighed 15 g and then mixed in 100 mL ethanol. 2.5 g of urea is added to the mixture in a Teflon-lined autoclave. The autoclave is then heated in an oven for 5 hours at 180 °C. Allow the

autoclave to cool to the surrounding temperature. The same procedure is repeated at the same stage without adding urea.

### Carbon Dot Purification

Following the final step of the synthesis procedure, the carbon dots solution is centrifuged at 5000 rpm (38007 g) for 15 minutes to separate the supernatant from the sediment. The supernatant is re-filtered with filter paper before being filtered again with a 0.45 m PTFE syringe filter. The filter results are concentrated using a hot plate at a temperature that varies according to the solvent. Carbon dots with distilled water were heated at 130 °C, while carbon dots with solvent ethanol were heated at 80 °C to become solids. The yield result is calculated from the weight of the pure carbon dot solid and the initial weight of the sample.

### Carbon Dot Characterization

Carbon dot absorbance measurement aims to see the absorbance of carbon dots at a specific wavelength. UV-Vis spectroscopy was used to measure the absorbance of carbon dots at wavelengths ranging from 200 to 600 nm with a 5 nm resolution. Carbon dot absorbance measurement was carried out on mangosteen peel carbon dot samples.

Fluorescence spectroscopy was used to measure the intensity of carbon dot fluorescence in the wavelength range of 380–600 nm with a resolution of 5 nm. Carbon dot fluorescence intensity was measured on carbon dot samples of mangosteen peel. FTIR spectra were obtained by heating 0.01 g KBr, then pounded and mixed with 0.02 g carbon dot samples. The formed pellets are heated in the oven for 12 hours and then measured using FTIR.

## Results and Discussion

### Comparison of Carbon Dot Yield

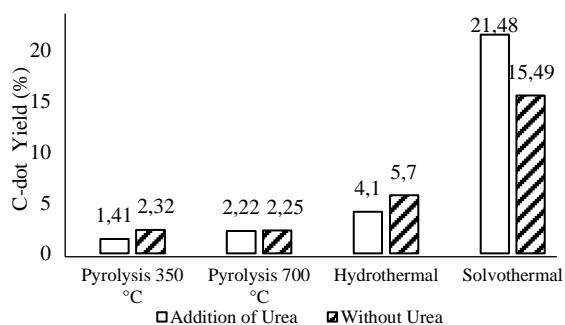
Carbon dot is synthesized from mangosteen peel containing cyanidin and xanthone compounds. Mangosteen peel has a moisture content of 62.53% (Table 1). These results differ from the literature with the moisture content of mangosteen peel at room temperature of 70.10% [11-12]. The difference in moisture content is influenced by air humidity, environmental conditions, fruit ripening process, and storage temperature [13].

Three synthesis methods result in different yields and characteristics of carbon dots (Figure 2). The lowest yield of the pyrolysis method is due to the pyrolysis process, which involves the stages of heating, dehydration, degradation, and carbonization at high temperatures. This stage is also known as thermal decomposition, which causes a less uniform size distribution. The presence of impurities such as minerals also inhibits the nucleation process and the carbon dot's growth, which is not optimal [8,14].

**Table 1.** Percentage of Water Content of Mangosteen Peel

Repetition	Water Content (%)
1	63.087
2	62.425
3	62.096
Average	62.536

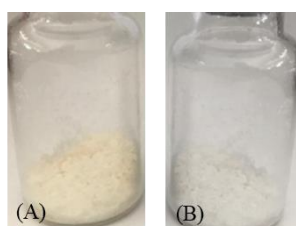
Hydrothermal and solvothermal methods have higher yields due to the process of hydrolysis of samples during preparation, resulting in smaller molecular sizes. Smaller molecular sizes are thought to increase the effectiveness of hydrothermal processes because all molecules can nucleate into carbon dots [15]. In addition, the synthesis process at high temperatures causes water and ethanol solvents to be in a supercritical fluid state. Supercritical fluids increase the viscosity of solutions, leading to more complete dissolved particles and better nucleation processes with particles of uniform size. This follows the literature stating that ethanol solvents can produce high yields and carbon dots with high emissions [16,17].



**Figure 2.** Comparison of mangosteen peel carbon dot yield

**Mangosteen Peel Carbon Dot Pyrolysis Method**

The result of the synthesis of carbon dot pyrolysis method is white, colorless in solution, and blue fluorescent under 366 nm UV lamp light (Figure 3). These results are similar to previous studies with carbon dots produced blue when illuminated by 365 nm UV lamps [5]. Similar results were also obtained on the precursors of watermelon peel waste, peanut shell waste, and lychee seed waste that produce carbon dots with high blue luminescence and solubility in water [14,18].



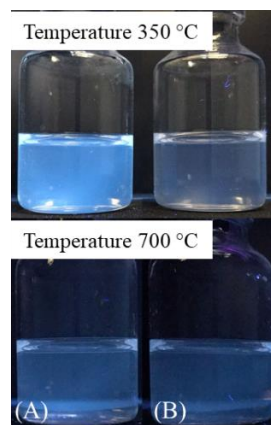
**Figure 3.** Solid of carbon dot from mangosteen fruit peel by pyrolysis method, (A) 350 °C, (B) 700 °C

Different synthesis temperatures in the pyrolysis method affect the carbon dot produced. Carbon dot with a synthesis temperature of 350 °C has a more intense blue

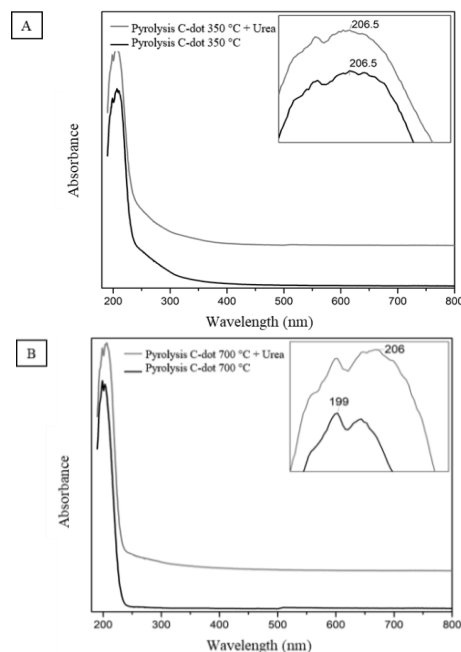
luminescence than the one at a temperature of 700 °C (Figure 4). The synthesis temperature of the carbon dot in the pyrolysis method should not be too low or too high.

Too low a temperature causes the material's carbonization not to occur ideally [14]. Too high a temperature causes the carbon source to become too oxidized, and the carbon dot's surface structure deteriorates, further leading to deterioration of the carbon dot's optical properties. The particle size of the carbon dot can shrink due to an increase in pyrolysis synthesis temperature with maximum emission of wavelength showing gradual blue shift [19].

Adding urea during the synthesis process also increased the intensity of bright blue luminescence under a 366 nm UV lamp at both temperature variations (Figure 4). These findings support the literature, which claims that adding passivation agents can enhance functional groups on the surface of carbon dots, increasing luminescence intensity [9,20].



**Figure 4.** Comparison of carbon dot luminescence by pyrolysis method with different synthesis temperatures under 366 nm UV lamp, (A) addition of urea (B) without urea



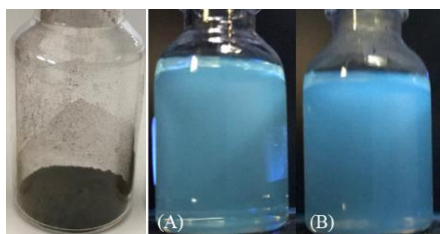
**Figure 5.** UV/Vis spectrum of carbon dot mangosteen peel by pyrolysis method. (A) 350°C (B) 700°C

These results are different from the literature, which states that the maximum absorbance of the carbon dot pyrolysis method is 350–550 nm [5] (Figure 5). Adding urea during the synthesis process does not significantly affect the resulting absorbance spectrum [5]. The absorbance spectrum of carbon dot in the lower range is thought to be due to the high synthesis process temperature. This is consistent with the literature, which suggests that too high a temperature reduces the CH<sub>2</sub> group of the carbon dots, hence lowering the conjugated  $\pi$  domain. The intensity of carbon dots luminescence decreases as the conjugated  $\pi$  domain is reduced [8,21].

### Mangosteen Peel Carbon Dot Hydrothermal Method

The hydrothermal method carbon dot results in black in solid form, brownish in solution state, and fluorescent blue color under 366 nm UV lamp. The luminescence of carbon dots with the addition of urea looks brighter when compared to without the addition of urea (Figure 6). These results are similar to the carbon dots from tobacco leaves, coffee grounds, and lemon peel with blue luminescence utilized in heavy metal detection. The blue luminescence is often found in carbon dots made from natural sources [14].

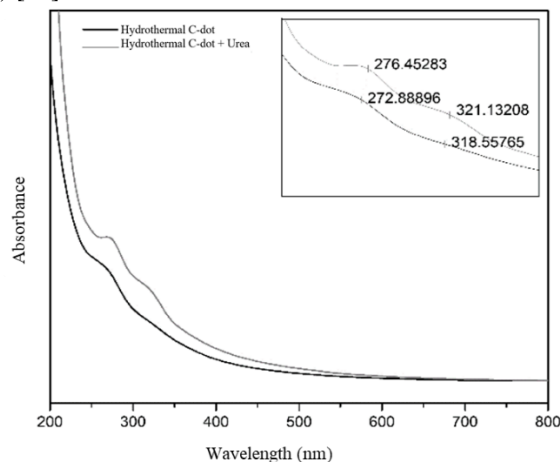
According to another study, adding urea can increase the fluorescence by adding nitrogen atoms to the carbon dot's surface [22]. The absorbance spectrum of the carbon dot without adding urea showed strong and small peaks at 272 and 318 nm and with urea at 276 and 321 nm. The  $\pi$ - $\pi^*$  electronic transition from C=C and the  $n$ - $\pi^*$  C=O transition are responsible for these firm and tiny peaks (Figure 7) [21].



**Figure 6.** Solid carbon dot of mangosteen fruit peel by hydrothermal method; Comparison of hydrothermal method carbon dot luminescence under 366 nm UV lamp, (A) Without urea addition (B) Urea addition

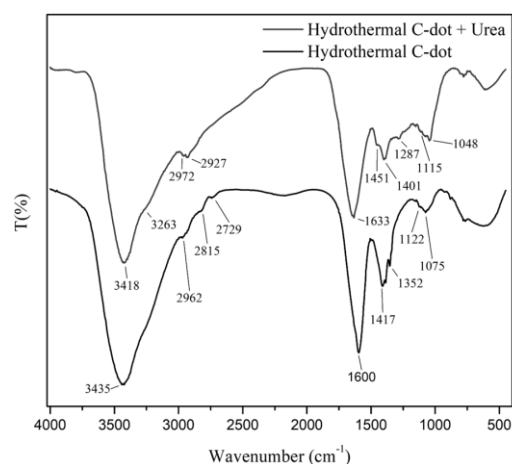
Based on the results of FTIR analysis (Figure 7), carbon dot mangosteen peel hydrothermal method with the addition of urea showed peak stretching OH (hydroxyl) 3418 cm<sup>-1</sup>, stretching N-H (amine) 3263 cm<sup>-1</sup>, extending OH (carboxylic acid) 2972 and 2917 cm<sup>-1</sup>, scissoring NH<sub>2</sub> (primary amine) and C=O (ketone) 1633 cm<sup>-1</sup>, stretching C=C 1451 cm<sup>-1</sup> related to the ring structure of the c-dot core, C-N pulling 1401 cm<sup>-1</sup>, and peaks appearing in fingerprint regions at 1287, 1115, and 1048 cm<sup>-1</sup> that can be derived from sp<sup>2</sup> and sp<sup>3</sup> vibrational stretching of C-H functional groups. Carbon dot without the addition of urea showed OH (hydroxyl) stretching peaks of 3435 cm<sup>-1</sup>, OH (carboxylic acid) stretching of 2962 cm<sup>-1</sup>, C=O (ketones) stretching of 1600 cm<sup>-1</sup>, C=C stretching of 1417 cm<sup>-1</sup> derived from the c-dot core structure, and peaks appearing

in fingerprint regions at 1352, 1122, 1075 cm<sup>-1</sup> (Figure 8) [23].



**Figure 7.** UV/Vis spectrum of carbon dot mangosteen peel by hydrothermal method

The difference between the two spectra lies in the stretching peaks of N-H (amines), NH<sub>2</sub> (primary amines), and C-N peaks due to the addition of urea, proving that carbon dots are successfully modified with nitrogen on the surface of their structure. Previous research has suggested that the C-N functional group is essential to producing photoluminescence [5]. The addition of nitrogen to the graphite structure of the c-dot core provides an emission energy trap, which boosts the electron pair's radiative recombination. Due to increased radiative recombination, the amount of photon emission also increases, which causes the intensity of photoluminescence to be higher [24].



**Figure 8.** FTIR spectrum of carbon dot mangosteen peel by hydrothermal method

### Mangosteen Peel Carbon Dot Solvothermal Method

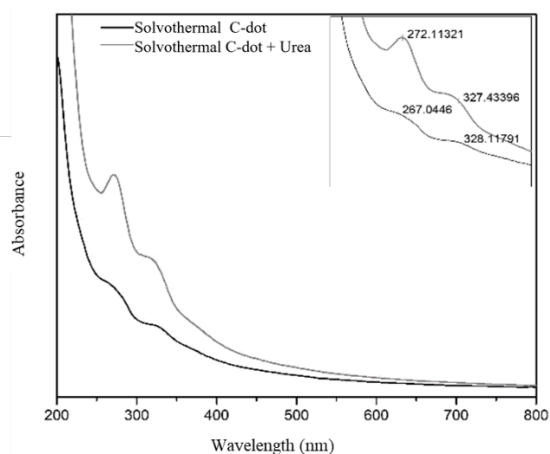
The results of the solvothermal method carbon dots are black in solid form, brownish when dissolved, and glow blue when illuminated by a 366 nm UV lamp. The luminescence of carbon dots with the addition of urea looks brighter when compared to without the addition of urea (Figure 9). The brighter luminescence is due to the addition of nitrogen atoms

sourced from urea, thus improving the surface state of the carbon dots [9].



**Figure 9.** Solid carbon dot of mangosteen peel by solvothermal method; Comparison of solvothermal method of carbon dot luminescence under 366 nm UV lamp, (A) without urea addition (B) with urea addition

The absorbance spectrum of the carbon dot without adding urea showed strong and small peaks at 267 and 328 nm and with urea at 272 and 327 nm. These solid and small peaks are due to the  $\pi$ - $\pi^*$  electronic transition of C=C and the  $n$ - $\pi^*$  C=O transition (Figure 10) [21]. When compared to other precursors, such as tulsi leaves, bamboo waste, rice husks, and food waste, they have similar absorbance peaks at a wavelength of 250-320 nm [24-26].

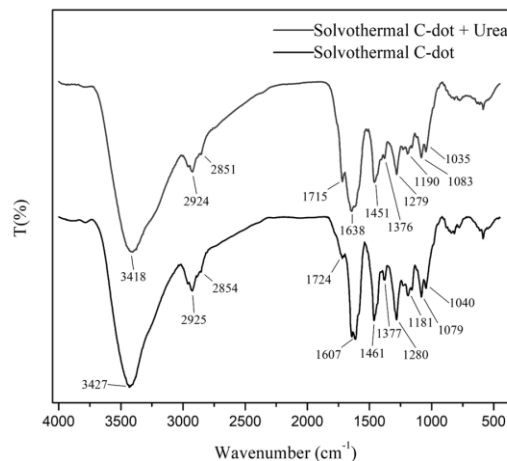


**Figure 10.** UV/Vis spectrum of carbon dot mangosteen peel solvothermal method.

Based on the results of FTIR analysis, carbon dot mangosteen peel solvothermal method with the addition of urea shows the peak stretching OH (hydroxyl) and NH (amine)  $3418\text{ cm}^{-1}$ , extending OH (carboxylic acid)  $2924\text{ cm}^{-1}$ , stretching C=O (ketones)  $1715$  and  $1638\text{ cm}^{-1}$ , wavenumber  $1415\text{ cm}^{-1}$  represents stretching OH (alcohol) as well as stretching C=C derived from ethanol solvent and c-dot core structure, C-N stretching of  $1376\text{ cm}^{-1}$ , and peaks  $1279$ ,  $1190$ ,  $1083$ , and  $1035\text{ cm}^{-1}$  represent CH structures with  $sp^2$  and  $sp^3$  configurations [27].

The results of the solvothermal method c-dot spectrum without the addition of urea showed OH (hydroxyl) and NH (amine) stretching peaks of  $3427\text{ cm}^{-1}$ , OH (carboxylic acid) stretching of  $2925\text{ cm}^{-1}$ , C=O (ketones) pulling of  $1724$  and  $1607\text{ cm}^{-1}$ , OH (alcohol) stretching and C=C stretching at  $1461\text{ cm}^{-1}$ , C-N stretching of  $1377\text{ cm}^{-1}$ , and some peaks that appeared in the fingerprint region, such as  $1280$ ,  $1181$ ,  $1079$ , and  $1040\text{ cm}^{-1}$  representing the CH of  $sp^2$  and  $sp^3$  configurations (Figure 11).

The difference from adding urea in the synthesis process has no significant effect on the results of FTIR spectrum analysis. Both spectra feature C-N strain peaks that contribute to the intensity of carbon dot photoluminescence [5]. Amine functional groups are found in treatment without adding urea due to the content of mangosteen peel, which is more soluble in semi-polar solvents, such as cyanidin and xanthone [28].



**Figure 11.** FTIR spectrum carbon dot mangosteen peel solvothermal method.

### Solvent Effect on Mangosteen Peel Carbon Dot

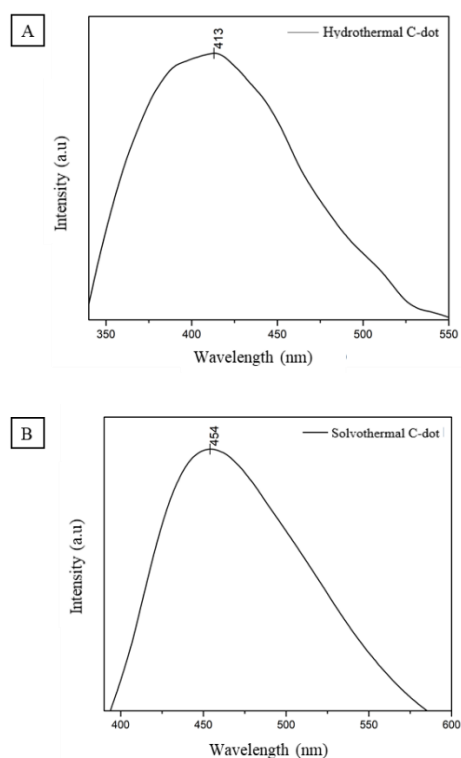
The carbon emission spectrum of mangosteen peel carbon dot hydrothermal and solvothermal methods is shown in Figure 12. Peak fluorescent emissions are obtained with emission wavelengths of 340–600 nm. The highest emission spectra of hydrothermal and solvothermal methods are 413 nm and 454 nm, respectively. The emission spectrum obtained corresponds to the luminescence of hydrothermal and solvothermal results in blue [5,22]. The results obtained were similar to the emission of mangosteen peel carbon dot that had been done previously by adding a passivation agent in the form of urea of 456 nm [5].

Similar research with calcination methods and precursors of mangosteen flesh produced blue luminescence with emissions of 330 nm [29]. When compared, the results of hydrothermal and solvothermal methods have larger wavelengths. However, when compared with the fluorescent peak of carbon dot from palm kernel shell with the same process, the same peak was obtained at 400 nm with excitation at 360 nm [24].

The photoluminescence mechanism of carbon dots involves radiative electronic transitions at  $\pi$  orbitals [5,30]. The carbon dot's emission energy is related to the particle size of the carbon dot formed. In the solvothermal carbon dot method, the emission obtained has a greater wavelength than the hydrothermal method.

The wavelength shift pointing to the right is known as the bathochromic shift. The bathochromic shift is induced by radiation from excitons trapped on

the surface of the c-dot as a result of increased surface oxidation [31]. The solvothermal c-dot bathochromic shift is influenced by the effect of the solvent used during synthesis. Ethanol, which is semi-polar, causes an increase in the interaction of the chromophore group with the solvent, resulting in a shift to a larger wavelength [32].



**Figure 12.** Carbon dot emission spectrum (A) hydrothermal (B) solvothermal

## Conclusion

All three methods successfully synthesized carbon dots from mangosteen peel waste. The solvothermal method produces carbon dots with the highest yield and intensity compared to hydrothermal and pyrolysis methods. Carbon dot mangosteen peel in hydrothermal and solvothermal methods has a better intensity than pyrolysis visually observed. The carbon emissions dot of the mangosteen peel is blue according to the color of its luminescence. Modification with the urea passivation agent succeeded in visually increasing the carbon dot fluorescence of mangosteen peel, but there is no significant difference from the resulting FTIR spectrum.

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