



# MODIFIED HOUSEHOLD PRESSURE COOKERS AS HYDROTHERMAL REACTOR FOR PRODUCTION OF CARBON DOTS FROM FISH SCALE WASTE

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## ABSTRACT

Recent advancements in the synthesis of carbon dots utilizing basic equipment are crucial for their large-scale production. This study explores the use of household pressure cookers to synthesize carbon dots from fish waste. A thermocouple and manometer are integrated into the cooker to regulate temperature and monitor pressure. Optimization of the equipment reveals that heating 2000 mL of distilled water to 100°C requires 20, 30, and 32 minutes with power inputs of 800 W, 700 W, and 600 W, respectively. The modified cooker achieves optimal pressure and temperature at 2.4 bars, 1.7 bars, and 1.5 bars, and 136°C, 123°C, and 122°C after 60 minutes of heating with the same power inputs. These results demonstrate that a household pressure cooker can effectively synthesize carbon dots. Experiments using dried Red Snapper fish spray powder, both unground and ground, at 800 W, 700 W, and 600 W, reach optimal pressure of 2.4 bar in 36, 49, and 58 minutes, and temperatures of 128°C, 131°C, and 136°C. Carbon dots are synthesized from fish scales as fine fibers with a 1:5 weight ratio of scales to distilled water, employing 600 W power, 2.4 bar pressure, 136°C temperature, 58 minutes of boiling, 5 minutes of centrifugation at 3500 rpm, and 45 minutes of ultrasound at 42 kHz. The formation of carbon dots is confirmed by an absorption peak at 405 nm in the UV-Vis spectrum, indicating high absorbance and bright blue fluorescence. Further investigation is required to optimize equipment using various fish scales and purification processes.

Keywords: Carbon dots; domestic pressure cooker; fluorescence; hydrothermal methods; pyrolysis.

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## INTRODUCTION

Fish scales are fisheries waste that has not been optimally utilized even though they contain many economically valuable organic compounds. Fish scales are rich in chitin as a natural biopolymer and collagen, which are sources of carbon, hydrogen, oxygen, and nitrogen [1-2]. Additionally, fish scales contain mineral content such as hydroxyapatite and calcium carbonate [3]. Fish scale waste is quite abundant in Indonesia and can have adverse effects on the environment, thus conversion into useful products such as nano-sized materials is necessary.

Fish scales, present a sustainable precursor for the synthesis of carbon dots <sup>[4]</sup>. The utilization of fish scales not only enhances the value of waste materials but also provides an environmentally friendly method for generating versatile carbon-based nanodots applicable in various fields.

Carbon Dots (CDs) are zero-dimensional nano materials with fluorescence capability, with carbon atoms as the core structure and heteroatoms as surface structures bound to carbon atoms <sup>[5-6]</sup>. CDs have many applications due to their low toxicity, high quantum yield, and ease of functionalization, making them potentially useful as biosensor materials, bioimaging materials, photocatalytic materials, and photothermal materials. Conversion of fish scales <sup>[1,7-8]</sup> into CDs materials continues to be developed using various methods such as hydrothermal methods <sup>[1,6,9-11]</sup>, immersion methods <sup>[12]</sup>, and sonochemistry methods <sup>[13]</sup>. Hydrothermal methods are the most commonly used bottom-up methods because they produce CDs with high quantum efficiency and non-toxicity, as well as lower production costs, although the production yield of CDs is lower than top-down methods <sup>[9]</sup>. Conventional hydrothermal synthesis of Carbon Dots requires high temperatures, high pressures, and long synthesis times, resulting in high energy consumption and expensive costs compared to other bottom-up methods <sup>[11]</sup>. Hydrothermal synthesis is generally carried out with specialized equipment serving as high-pressure hydrothermal reactors, typically stainless steel autoclave cylinders with a non-cheap Teflon coating <sup>[10]</sup>.

In the limited resource conditions of developing countries, pressure cookers commonly used in households are utilized as cheaper hydrothermal equipment. The synthesis of CDs was achieved utilizing an domestic pressure cooker as a hydrothermal reactor using Citric acid (CA) and glucosamine (GA) served as the carbon sources <sup>[14]</sup>. The precursor was subjected to twenty consecutive cycles, each with a duration of 99 minutes, under the "high pressure" setting of the cooker. Also, domestic pressure cookers have been used to produce nanomaterials, as well as to produce silver nano particles using Gum kondagogu, silver nitrate (AgNO<sub>3</sub>), and ultra-pure water as raw materials <sup>[15]</sup>. Nonetheless, the specific temperature and pressure parameters employed in the aforementioned studies remain unspecified. For that, the domestic pressure cooker needs to be modified.

In this research, the enhanced domestic pressure cooker is equipped with a thermocouple and a pressure gauge, enabling the monitoring of temperature and pressure during the synthesis of carbon dots. By implementing these modifications, the aim is to furnish precise data regarding the experimental parameters, such as temperature and pressure, involved in the synthesis process of carbon dots. Subsequently, it is crucial to optimize performance parameters such as reactor pressure, reactor temperature, and heating duration to ascertain the appropriate synthesis conditions when utilizing this apparatus. This study seeks to enhance the efficiency of household pressure cookers for the synthesis of carbon dots derived from fish scale waste.

## METHODS

### MATERIALS PREPARATION

The primary material used for synthesizing CDs is sourced from the scales of the Red Snapper (*Lutjanus sp.*) fish, which are rich in collagen and chitin. These scales were obtained from a local market in Manado City <sup>[16]</sup>. Initially, the scales underwent a cleaning process that involved washing with tap water, followed by rinsing with distilled water, and were then air-dried under sunlight for two days <sup>[17]</sup>. Three distinct samples of Red Snapper fish scales were prepared, designated as Sample A, Sample B, and Sample C (Figure 1). Samples A and B consisted of dried fish scales, each weighing 200 grams, which were neither ground nor blended. These

were then combined with 2000 mL of distilled water, maintaining a fish scale to water mass ratio of 1:10<sup>[1,11]</sup>. The suspensions of Samples A and B maintained the same mass ratio of fish scales to distilled water; however, Sample A was subjected to pyrolysis using a pressure cooker at 800 W, whereas Sample B was pyrolyzed at 700 W. Sample C consisted of fish scales that were processed into fibers, with a total mass of 400 grams, and mixed with 2000 mL of distilled water, resulting in a fish scale to water mass ratio of 1:5<sup>[10]</sup>. The suspension of Sample C was pyrolyzed at a pressure cooker power of 600 W.



a. Sample A



b. Sample B

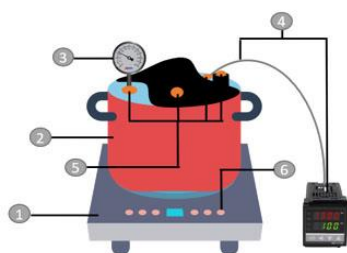


c. Sample C

**Figure 1.** Samples of Fish Scale

## PREPARATION OF HIDROTHERMAL REACTOR

The hydrothermal reactor utilized for synthesizing CDs in this study is a Unilert brand domestic pressure cooker, as referenced by Laber<sup>[14]</sup> and Kora<sup>[15]</sup>. This reactor has a capacity of 5.5 liters and is constructed from stainless steel. Modifications were implemented to the reactor to include instruments for monitoring pressure and temperature, as illustrated in Figure 2. The pressure gauge, produced by Tekiro, measures pressures from 0 to 2.5 bar. The temperature monitoring system consists of an Autonics TZN4S rex C-100 temperature controller, with a power rating of 10 watts and an electric voltage of 10 volts DC, supplemented by a type K thermocouple capable of detecting temperatures up to 1000°C. The reactor's heat source is a Memoo-brand induction stove (slim induction cooker) equipped with a timer and adjustable power settings. The domestic pressure cooker, due to its autoclave system, along with the induction stove and the pressure and temperature gauges, were acquired from a supermarket. After the modifications, 2000 mL of water were boiled for 60 minutes using varying stove power settings of 600 watts, 700 watts, and 800 watts. During the boiling process, the temperature and pressure of the water or steam within the reactor were recorded at 10-minute intervals. The purpose of boiling the water was to evaluate the reactor's performance in terms of temperature and pressure fluctuations over the duration of the boiling period.



Legend: 1. Stove, 2. Presto, 3. Thermometer, 4. Pressure Gauge, 5. Presto Steam Cover Glue, 6. On/off button

a. Design of Reactor

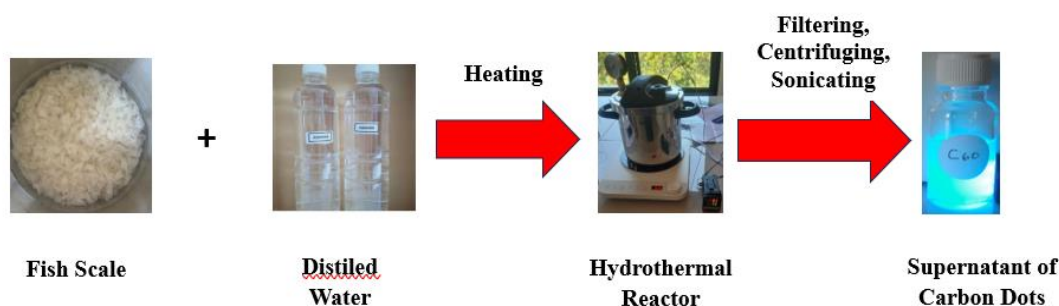


b. Modified Reactor

**Figure 2.** Reactor of Hydrothermal

## PROCEDURES OF FISH SCALE DERIVED CARBON DOTS FABRICATION

The synthesis of CDs was accomplished via a hydrothermal method, which involved boiling fish scales within a reactor. Figure 3 illustrates the schematic procedure for synthesizing CDs from fish scales. Specifically, 200 grams each of Sample A and Sample B, along with 400 grams of Sample C, were individually suspended in 2000 mL of distilled water. These suspensions were heated on a stove set to 800 watts, 700 watts, and 600 watts, respectively, until the reactor pressure reached 2.4 bar. At this point, the temperature was measured using a thermocouple, the heating was stopped, and the reactor was allowed to cool naturally to ambient temperature. The resulting liquid was filtered using Whatman filter paper number 42, which has a diameter of 13 centimeters and a pore size of 2.5 micrometers. To separate the supernatant from the sediment, each boiled fish scale suspension was centrifuged at 3500 revolutions per minute for 5 minutes. After centrifugation, the supernatant underwent a second filtration using Whatman paper number 42. In the final step, each supernatant was sonicated with a 42 kHz batch sonic device for 60 minutes. The supernatant obtained from ultrasonication was then stored in 20 mL vial bottles for the analysis of its optical properties.



**Figure 3.** Stages schematic of Carbon Dots synthesis from fish scale

## OPTICAL CHARACTERIZATION OF SUPERNATANT FLUID

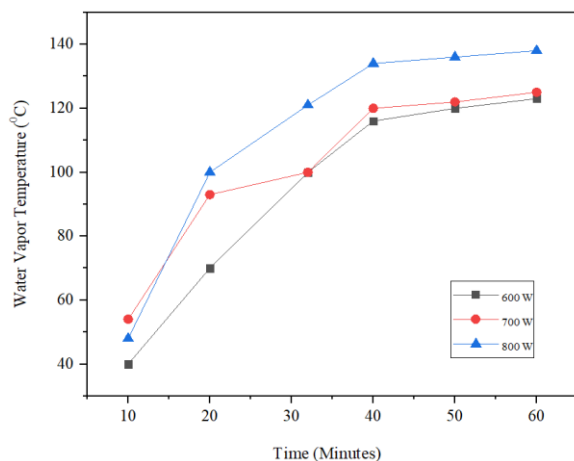
The optical properties of the supernatant liquid obtained from fish scale waste were examined through visual inspection in a controlled laboratory environment, using both natural sunlight and ultraviolet (UV) light to observe fluorescence phenomena <sup>[10, 12]</sup>. Furthermore, a semi-quantitative evaluation was performed using UV-Vis spectroscopy techniques <sup>[6]</sup>. Sunlight and violet light were employed to assess the color emission, as well as the scattering and emission of light from the supernatant. The semi-quantitative analysis was conducted at the Integrated

Laboratory of Sam Ratulangi University using a Shimadzu UV-Vis series 1800 spectrophotometer, which covers a wavelength range of 200-700 nm. This analysis aimed to elucidate the optical characteristics of the samples by analyzing the spectral absorbance patterns of each supernatant sample. The results of the UV-Vis characterization were processed using UV-Probe 2.42 software.

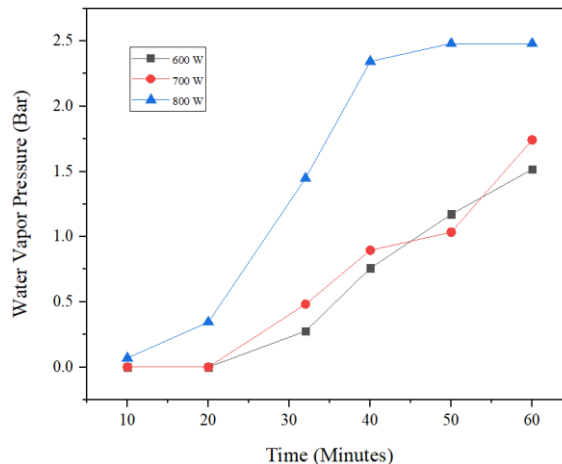
## RESULTS AND DISCUSSION

### PERFORMANCES OF THE HYDROTHERMAL REACTOR

The optimization of performance was concentrated on the patterns of temperature and pressure parameters concerning heating duration, as demonstrated in Figures 4 and 5. Figure 4 illustrates the variations in steam temperature within the reactor. The steam temperature achieved 100°C at different intervals contingent on the stove power level. Specifically, at 600 W, 700 W, and 800 W, the water temperature reached 100°C in 32 minutes, 30 minutes, and 20 minutes, respectively. These results indicate that higher stove power facilitates a more rapid attainment of 100°C due to an augmented heat supply. The steam temperature increased significantly from the 10th to the 40th minute, but after the 41st minute, the rate of temperature increases decelerated. The optimal temperature achieved at the 60-minute mark was 122°C for 600 W, 123°C for 700 W, and 136°C for 800 W. Based on the time required to reach 100°C, the reactor's operational duration will be extended with lower stove power.



**Figure 4.** Steam Temperature in the Reactor vs Boiling Time



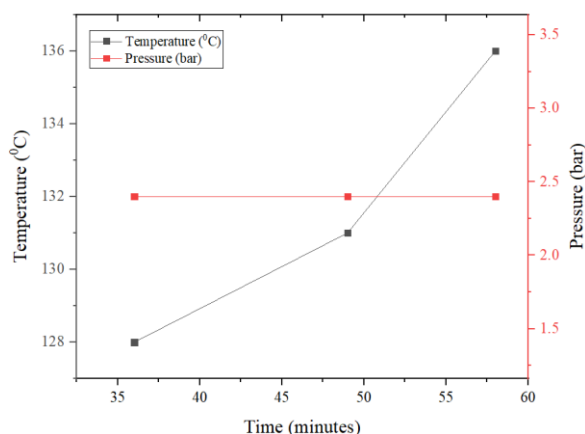
**Figure 5.** Steam Pressure vs Boiling Time

Figure 5 depicts the variations in steam pressure within the reactor over a 60-minute heating interval. At the conclusion of this period, the optimal pressures achieved were 1.5 bar for a 600 W stove, 1.7 bar for a 700 W stove, and 2.4 bar for an 800 W stove. The pressure exhibited a pronounced increase from the 10th to the 40th minute, subsequently approaching stabilization near the maximum value indicated by the pressure gauge when utilizing an 800 W stove. The modified reactor's performance, constrained by the pressure gauge's upper limit of 2.5 bar, typically operates for less than 60 minutes at an 800 W stove power setting. The pressure-time graph for stove powers of 600 W and 700 W reveals a steep ascent up to the 60th minute, suggesting that continued heating beyond this duration would result in further pressure escalation towards the gauge's maximum operational threshold. The duration of reactor operation for heating distilled water is contingent upon the stove's power, with lower power settings leading to extended operational periods.

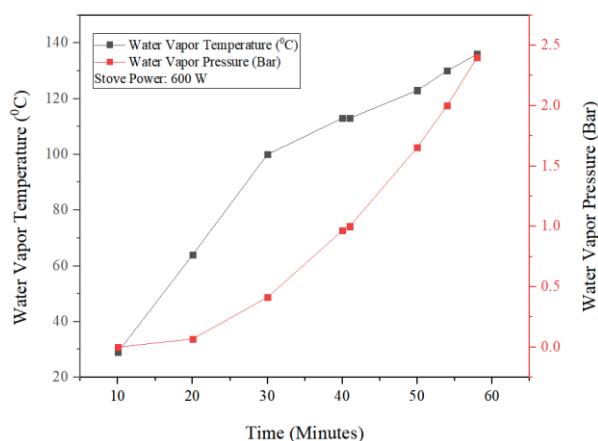
Figures 4 and 5 illustrate a divergence in the trends of temperature and pressure increases for stove powers of 600 W and 700 W during the period between the 40th and 60th minutes. The temperature trend begins to plateau, whereas the pressure trend continues to rise. The heat from the stove is primarily used for the evaporation of water rather than for increasing the water's temperature. The rapid increase in pressure indicates a growing number of water vapor molecules within the reactor. This contrasts with the scenario involving an 800 W stove, where both temperature and pressure trends reach equilibrium as the water vapor approaches saturation, consistent with Gay-Lussac's law. According to the ideal gas law <sup>[18]</sup> in a 5.5 L reactor heated for 60 minutes with an 800 W source, the estimated quantity of water vapor molecules is 0.39 mol, or approximately  $2.34 \times 10^{23}$  molecules, assuming the complete evaporation of 2 L of distilled water. The examination of the boiling point of water, the temperature of steam, and the optimal steam pressure in relation to the maximum reading of the pressure gauge demonstrates that the modified reactor, derived from a household pressure cooker, operates effectively for the heating and evaporation of distilled water. These results suggest that this reactor is appropriate for experimental procedures in the synthesis of carbon dots using fish scales as the precursor material.

### FABRICATION OF CARBON DOTS FROM FISH SCALE WASTE

Figure 6 presents the variations in reactor temperature and the optimal heating duration when fish scales are boiled at a pressure of 2.4 bar, using stove power levels of 600 W, 700 W, and 800 W. Sample A, processed with an 800 W stove, reached a pressure of 2.4 bar in 36 minutes at an optimal temperature of 128°C. Sample B, using a 700 W stove, achieved the same pressure in 49 minutes with an optimal temperature of 131°C. Sample C, utilizing a 600 W stove, required 58 minutes to reach 2.4 bar at a temperature of 136°C. These results indicate that using a 600 W stove for hydrothermal treatment results in the longest boiling time and the highest optimal temperature while maintaining the reactor pressure at 2.4 bar. Figure 7 further illustrates the trends in temperature and pressure increase within the hydrothermal reactor. A temperature of 100°C is reached at the 30th minute with a steam pressure of 0.413 bar, and it takes 28 minutes for the steam pressure to reach 2.4 bar. Hydrothermal treatment for 28 minutes at temperatures exceeding 100°C facilitates the penetration of hot water, dissolving minerals in fish scales, such as collagen, peptides, chitin, and other organic minerals, potentially leading to carbonization <sup>[6]</sup>.



**Figure 6.** Optimal Temperature and Boiling Time for Fish Scale Samples



**Figure 7.** Temperature and Pressure Variations during Fish Scale Boiling with a 600 W Stove



The visual representation of the boiling outcomes for each sample is illustrated in Figure 8. The data reveal a noticeable color transformation in the liquid pre- and post-boiling. Initially, the liquid is transparent, akin to distilled water, but it becomes slightly turbid following the boiling process. Notably, the liquid from Sample C displays a more intense yellowish-brown hue compared to those from Samples A and B. Prior research has demonstrated that CDs synthesized from fish scales via the hydrothermal method yield a brownish suspension, as shown in Figure 9. Consequently, the suspension from Sample C (Figure 8c) is more consistent with empirical observations than those from Sample A (Figure 8a) and Sample B (Figure 8b) [1,6,11]. The intensity of the yellowish-brown coloration is associated with the formation of CDs. The observed color change in the boiled water from Red Snapper fish scales mixed with distilled water is due to the physical and chemical constituents of natural fish scales (collagen, hydroxyapatite, lipids) forming nano-scale fluorescent particles that dissolve in water. During the boiling of Red Snapper fish scales, pyrolysis occurs, resulting in the dehydration of molecules within the fish scales and the subsequent formation of CDs [12]. The boiling duration and temperature within the autoclave reactor significantly affect the pyrolysis process of fish scale material into CDs, alongside pressure parameters [10]. In addition to temperature and boiling time, the primary factor contributing to the color variation in Sample C's suspension compared to Samples A and B is the mass composition of fish scales [10,14]. Sample C contains a greater mass of fish scales than Samples A and B, leading to a higher organic content. The greater the organic content, the more raw material is available for CDs formation, resulting in a higher yield of CDs [6].



a. Sample A (800 W; 2,4 bar; 128°C; 36 minutes)



b. Sample B (700 W; 2,4 bar; 131°C; 49 minutes)



c. Sampel C (600 W; 2,4 bar; 136°C; 58 minutes)



2 g fish scale powder, 20 mL MilliQ water, autoclave, 200°C, 24 hours  
**Figure 9.** Carbon Dots Suspension

**Figure 8.** Boiling Result Suspension

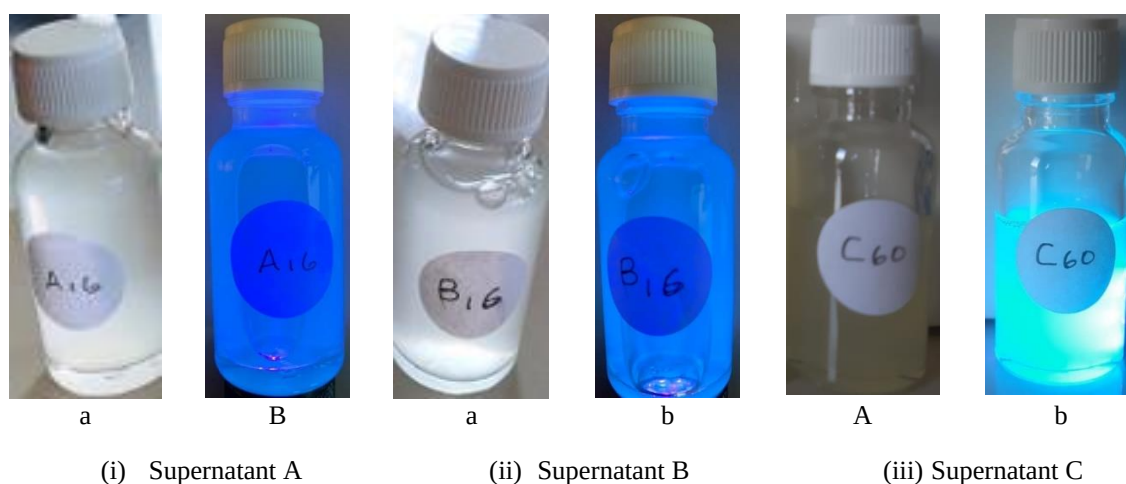
## OPTICAL PROPERTIES OF CARBON DOTS

Figure 10 depicts the coloration of the supernatant from sample A (Figure 10 (i)), sample B (Figure 10 (ii)), and sample C (Figure 10 (iii)) under both sunlight (a) and UV light (b) exposure. In sunlight, supernatants A and B appear as translucent, cloudy solutions, whereas supernatant C exhibits a light yellowish-brown color, indicating the presence of carbon dots as identified by Yao [12]. Under UV light, supernatants A and B demonstrate the Tyndall effect, scattering the light and producing a bluish-purple hue akin to the UV source. The results demonstrate that supernatants A and B did not display fluorescence effects due to the absence of carbon dot particles. The supernatant C obtained from the thermal processing of Red Snapper fish scales exhibits circular dichroism properties by absorbing high-energy violet light and subsequently emitting blue light at longer wavelengths. This blue luminescence occurs spontaneously following the absorption of violet light during the process of photoexcitation. The extent to which the nanoparticles within the supernatant absorb violet light correlates with an increased absorbance value, thereby enhancing photoexcitation and the resultant emission

of blue light. This luminescent emission is transient and ceases once the UV lamp is turned off.

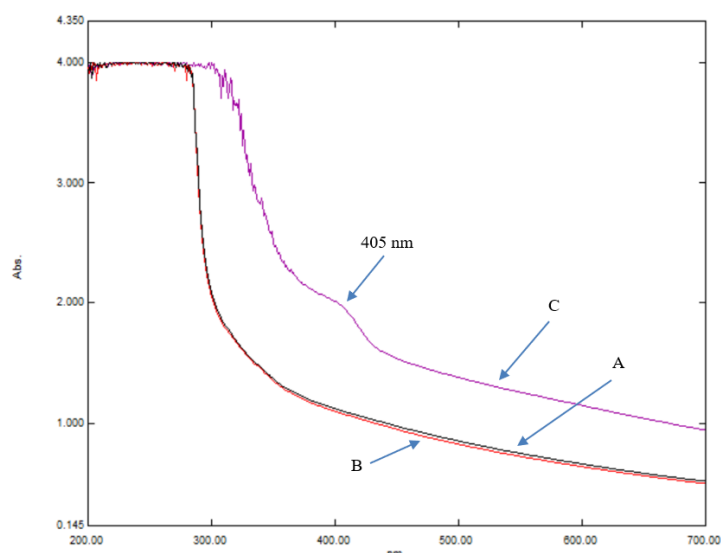
The synthesis of CDs through the hydrothermal method utilizing a modified pressure cooker reactor can be semi-quantitatively assessed using UV-Vis spectroscopy, which elucidates their spectral absorbance characteristics. As illustrated in Figure 11, the UV-Vis spectra of samples A and B display patterns reminiscent of fish scales, with ultraviolet light absorption occurring between 200 and 287 nm<sup>[16]</sup>. The resemblance in spectral patterns between samples A and B implies that they share similar optical properties. Importantly, the supernatants of these samples lack CDs, as evidenced by the absence of spontaneous emission and absorption peaks in the visible spectrum. In contrast, the UV-Vis spectrum of sample C exhibits an absorption peak at 405 nm, indicative of CDs synthesis, consistent with findings by Zhang et al., where an absorption peak was noted at 320 nm, as depicted in Figure 12a<sup>[4]</sup>. The luminescence phenomenon and the distinct absorption peak identified in the carbon-containing supernatant in this study are consistent with the findings reported by Athinaran, as depicted in Figure 12b. in 2016<sup>[10]</sup>, Laber synthesized CDs using citric acid as the primary raw material<sup>[14]</sup>. This process, conducted with a domestic pressure cooker, revealed that the absorption peak of the CDs is situated between wavelengths of 300-400 nm, and 600 nm. The detection of an absorption peak in the UV-Vis spectrum, as reported in previous research, indicates the presence of carbon nanoparticles in the supernatant C.

The 405 nm absorption peak is associated with blue light emission upon exposure to ultraviolet light, as demonstrated in Figure 10. This peak is related to photoexcitation followed by fluorescence, characterized by significant emission intensity<sup>[16]</sup>. The appearance of absorption peaks in the UV-Vis spectra signifies electron excitation from the highest occupied molecular orbital (HOMO) to the lowest unoccupied molecular orbital (LUMO), followed by electron transitions that can be  $\pi \rightarrow \pi^*$  or  $n \rightarrow \pi^*$ <sup>[11]</sup>. This confirms the presence of CDs particles in the supernatant of sample C.

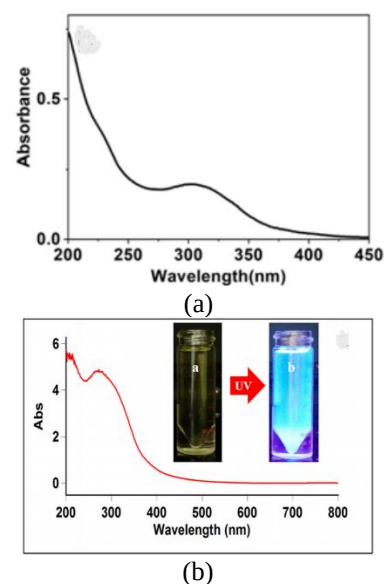


**Figure 10.** Supernatants A, B, and C: a) under sunlight irradiation; b) under UV light irradiation





**Figure 11.** UV-Vis Spectral Analysis of Supernatant Sample A, Sample B, and Sample C



**Figure 12.** UV-Vis Spectral of the Fish scales derived CDs <sup>[4,10]</sup>

The findings of this study demonstrate that the synthesis of CDs is affected by various parameters, including the stove's power, the boiling duration, the pressure inside the pot during boiling, the optimal boiling temperature, and the variations in the duration of ultrasonic treatment. Ultrasonic waves at a frequency of 42 kHz contribute to the carbonization process, aiding in the transformation of fish scales into CDs. These waves produce mechanical effects akin to pyrolysis during thermal treatment, thereby accelerating reaction speed, mass transfer, and solvent penetration into the inner layers of Red Snapper fish scales <sup>[16]</sup>. It is recommended to use fish scales that have been blended into a slightly fine and fibrous texture as the raw material. The synthesis of CDs was successfully accomplished using a modified pressure cooker equipped with pressure gauges and thermocouples, employing a 600 W electric stove, a boiling time of 58 minutes, an optimal steam temperature of 136°C, a steam pressure of 2.4 bar, and ultrasonic treatment lasting 45 minutes. However, to assess the reliability of this equipment, it is imperative to conduct a comprehensive analysis of fish scale samples collected from a wide array of fish species.

Utilizing a household pressure cooker to synthesize carbon dots from fish scale waste offers a promising avenue for cost-effective, large-scale production. Nonetheless, scaling up production presents several challenges, particularly in the purification process of carbon dots <sup>[19]</sup>. Achieving efficient purification of a substantial quantity of carbon dots swiftly and economically, while minimizing the production of contaminated water during post-processing, remains a significant hurdle for environmentally sustainable production. Moreover, the long-term stability and safety of carbon dots in complex environments necessitate thorough validation to meet practical application standards. Although most studies indicate that carbon dots are non-toxic within certain concentration ranges, their long-term behavior and metabolic fate in intricate biological environments are not yet fully understood, raising concerns about their clinical applicability. Future research should prioritize these areas to establish safety benchmarks. Additionally, the by-products and residues generated may pose risks to biocompatibility, necessitating further refinement of the cleaning and purification processes, particularly in large-scale production.

## CONCLUSION

A modified household pressure cooker, augmented with pressure gauges and thermocouples, was employed as a hydrothermal reactor for the synthesis of CDs. The reactor operated optimally for a duration of 60 minutes, achieving steam pressures of 1.52 bar on a 600 W stove, 1.74 bar on a 700 W stove, and 2.4 bar on an 800 W stove. The time required to reach the boiling point of water at 100°C was 20 minutes for an 800 W stove, 30 minutes for a 700 W stove, and 32 minutes for a 600 W stove. The 600 W stove demonstrated the most efficient boiling time and temperature at a working pressure of 2.4 bar compared to the 700 W and 800 W stoves. CDs were successfully synthesized from Red Snapper fish scales using the following parameters: 400 grams of finely fibrous fish scales, 2000 mL of distilled water, a 600 W stove, a boiling time of 58 minutes, an optimal boiling temperature of 136°C, a pressure of 2.4 bar, centrifugation for 5 minutes at 3500 rpm, and ultrasonic treatment at 42 kHz for 45 minutes. The transformation of fish scales into CDs is confirmed by an absorption peak in the UV-Vis spectrum at 405 nm, indicating blue light emission when the CDs supernatant is exposed to ultraviolet light. In future research endeavors, it is imperative to evaluate the reliability and efficacy of household pressure cookers in the synthesis of CDs using fish scale waste derived from various fish species and purification processes.

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